INTERNATIONAL UNION OF CRYSTALLOGRAPHY

# IUCr Newsletter

Volume 29 · Issue 3

Collected archival edition
Original article text and available illustrations

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LETTER FROM THE PRESIDENT

# My reflections after the Prague Congress



The newly elected IUCr President, Hanna Dabkowska, addresses the Prague Congress participants from the garden of her home in Canada.

Hanna Dąbkowska

It is a great honour to be able to write to you all on the first page of the *IUCr Newsletter*. It introduces me as the IUCr President to the crystallographic community and allows me to make some personal comments about the unusual times in which we are living.

Prague was my eighth IUCr Congress, after Warsaw, Seattle, Florence, Osaka, Madrid, Montreal and Hyderabad. I enjoyed all of them as much for the warm, friendly atmosphere as for the excellent science.

2020 was a challenging year. The Conference Chair, Professor Radomír (Radek) Kužel, and his team were well ahead with preparations for the Prague Congress. Then, with the pandemic declared at the beginning of 2020, all plans started to change. It took a lot of time and discussions at IUCr Executive Committee (EC) level to decide that the 25th Congress and General Assembly (GA) should be postponed. We were anticipating that by 2021 life would go back to normal ... .

In the middle of June 2021 when the COVID-19 delta variant started to propagate around the world and the chance for in-person travel became even more limited, Radek and his team decided to have the Congress and GA in the hybrid mode. The hybrid mode is the most challenging and demanding but we can all confirm that the Congress was planned and executed flawlessly. So were all three GA meetings.

Normally, the EC meets with Commissions and Committee Chairs, Journals and Other Scientific Body Representatives during the 10 days before the Congress. Jet lag has often been an issue but we all knew how to deal with it. This year, similar meetings (very efficiently organized by Alex Ashcroft on Zoom) were happening nearly every day in June. Because of the geographical distribution of all involved, we were starting very early in the morning (for the Americas) or ending very late (for Asia). It was worth it as the meetings were very informative and positive, and Sven Lidin made sure that all the questions were asked (and answered) within the time limits.

The EC met on Zoom again a few times in August, just before the Congress, to prepare for the GA and EC (virtual!) elections. We had two excellent candidates for Vice-President and ten very good ones for three EC slots. Again because of the different time zones the final results were only known after the Congress Closing Ceremony. All the results were posted immediately on the IUCr website.

The important Old & New EC meeting, which traditionally wraps up the Congress, has been postponed until the end of September (after the deadline for this Letter).

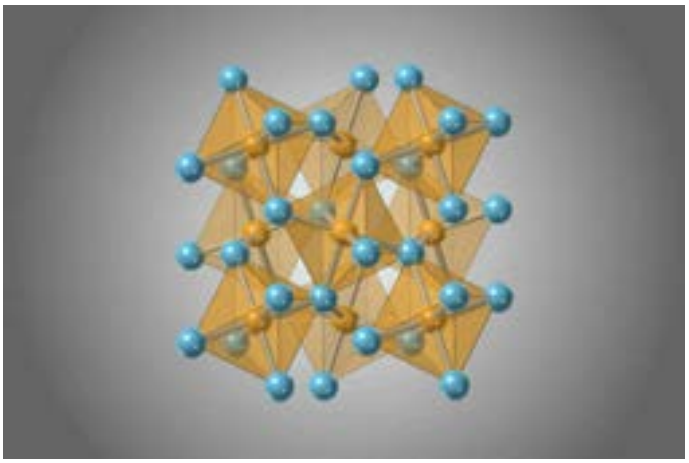
In 2021 we learned that – with a strong community and devoted leaders – one can execute a successful, scientifically sound virtual or hybrid meeting. We also learned that friendship and collaboration created during many previous conferences, schools and congresses is stronger than the pandemic. But we also confirmed that personal contacts cannot be replaced by technology. To be able to create the world that we enjoy, we all need to make the effort to overcome the obstacles. This time the obstacle is COVID-19 – so we have to fight for equal access to vaccines for everyone in the world and with the vaccine hesitancy in some communities.

It is very important that we prepare for financial challenges in the coming years. This means that money has to be put aside for attending meetings. For ourselves and for the future generation of crystallographers and structural scientists. The IUCr – being a not-for-profit organization – uses the income generated by its 10 journals and *International Tables* to support young scientists around the world so the IUCr becomes their organization too.

Now we should look forward to new opportunities preparing for the post-pandemic world. Please publish in IUCr journals as you – and only you – are responsible for the steady growth of scientific knowledge and the impact factor (so overrated but still meaningful). Update your entry in the *World Directory of Crystallographers* (WDC), consider joining the IUCr Associates Programme and start preparing for the next meetings in your area and for the 26th IUCr Congress in Melbourne.

EDITORIAL

# Editorial



Aurostibite (drawn using *CrystalMaker*).

Mike Glazer

So, the IUCr Congress in Prague has passed successfully. I have to take my hat off to Radek Kužel and his team, working under the most difficult of circumstances, caused by … you know what. I don't know how they managed it, but I think it is likely that hybrid meetings like this will probably become the standard in the future. It went off so smoothly and professionally. I was able to tune into several talks from France, where I was staying at the time. Marvellous! One result is that we have a new President, Hanna Dabkowska, and Vice-President, Santiago García-Granda. Congratulations to them both.

I am sorry to say that it has been a lousy year for crystallographers this year, as so many have gone. In this issue, we have obituaries for Carroll Johnson, John Reid, Hans Boysen, John Squire, John Spence and Tibor Koritsanszky. And then, very recently, we have heard of the death of Jack Dunitz, a well-known figure to all crystallographers. I hope we shall have an obituary for him in the next issue.

Here, we have several articles on the great J. D. Bernal, who died 50 years ago. These articles were recently printed in the British Crystallographic Association's *Crystallography News*, and I am pleased that John Finney, the Editor, has allowed us to reprint them here. Bernal was one of those towering figures in crystallography, a man of incredible intellect and influence. Every crystallographer should know about him. There is an excellent book on his life by Andrew Brown entitled *J. D. Bernal: The Sage of Science*. It is well worth a read, especially if you are interested in the history of our subject. Bernal was not only a key figure in crystallography, he was someone with many other interests. He was controversial for his views on Marxism, with the result that he was better known in the Soviet Union than here in the West.

This reminds me of a story. Many years ago, I was having a coffee at Birmingham (UK) airport when I noticed at the next table a man reading Andrew Brown's book. So, I leant over to him and said to him that I had known Bernal myself. It appeared that he was writing a review for one of our newspapers, *The Daily Telegraph* (https://www.telegraph.co.uk/culture/books/3649528/The-unknown-polymathic-crystallographer.html). He said to me, "You know, Bernal was such an evil man!" Of course, I swiftly tried to put him right, but I don't think to much avail.

I recall meeting Bernal a few times at Birkbeck College, London. The first time was when the late Howard Flack and I were students in 1966 in the laboratory of Kathleen Lonsdale and used to attend evening classes on crystallography at Birkbeck. At one point, Bernal came in to address us. He was by this time very ill, having had a stroke. I remember that he was in a wheelchair and had a massive loudspeaker through which he could try to talk. It was sad to see him in this state, and the poor man must have felt

very frustrated. He was at that time President of the IUCr, but, because of illness, he was unable to attend the Moscow IUCr Congress that year; instead, Kathleen Lonsdale stood in for him as acting President.

So do get a copy of Brown's book if you can. It is well written and fully explores all the intriguing aspects of the life of Bernal. One minor correction I would make. Brown mentions that Bernal invented the phrase "weapons of mass destruction." However, this first appeared in a book called *We* (Мы) by Yevgeny Zamyatin in 1921, which described a dystopian world. I am reasonably sure that, given Bernal's interest in Russia, he must have been aware of this.

One of Bernal's most famous students was Dorothy (Crowfoot) Hodgkin, who had so many images of her published, including on postage stamps. Magdolna and Istvan Hargittai tell us about a couple of less well-known pictures of her. Just recently, a short BBC video about the life of Dorothy Hodgkin has appeared at https://www.bbc.co.uk/ideas/videos/the-exceptional-life-of-dorothy-crowfoot-hodgkin/p09x02lh?playlist=made-in-partnership-with-the-royal-society.

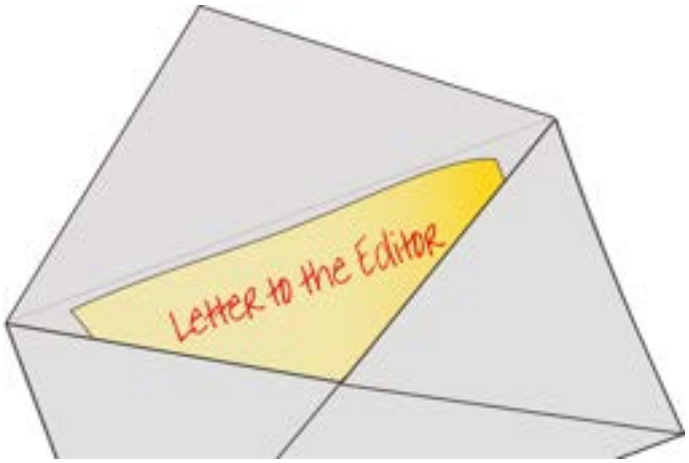
Another major article in this issue is on the 100 years history of ferroelectricity, written by Nicola Spaldin and Ram Seshadri. Ferroelectricity is a phenomenon exhibited by certain crystalline materials in which an electric polarization can be switched by an external electric field, much like the way magnetisation can be switched by an applied magnetic field in ferromagnets. Nicola tells me that she had a lot of fun researching this topic.

Leopoldo Suescun also tells us about teaching symmetry in the Latin American countries. This has been mainly within the framework of the IUCr Commission on Mathematical and Theoretical Crystallography. Crystallography has been growing fast in this region, where they have their own crystallographic association, LACA.

Finally, many of you will remember Michael Woolfson, who died in 2019. Now, the Royal Society has made available his biographical memoir written by Keith Wilson and Eleanor Dodson.

LETTER TO THE EDITOR

# A letter of thanks to the Prague IUCr Congress Organizers




Mariusz Jaskolski

Professor Mariusz Jaskolski

Department of Crystallography

Faculty of Chemistry

A. Mickiewicz University

Poznan

POLAND

22 August 2021

I think we all owe a note of appreciation to our Czech colleagues who organized the 25th IUCr Congress in Prague (and in cyberspace).

Not only had they to organize a hybrid Congress for the first time in the history of the IUCr; worse than that: they had a “normal” Congress almost ready in mid-2020, then had to go through a wave of hellish uncertainty, to finally have to face a total crash of the 2020 meeting. They withstood the blow bravely and immediately started putting together the 2021 Congress in a mixed mode, which by itself would be a sufficiently tall challenge. We should also remember that at some point on this road Czechia was the worst hit nation in Europe.

In the end we’ve had a very successful Congress and a totally new experience. Finally, after a long period of abstinence we could meet our friends, even if only virtually (I was myself a virtual participant).

Looking back upon this disastrous period of almost two years of the COVID-19 pandemic, I think we should be grateful to our overseers that the 2020/21 Congress was put in the hands of our Czech colleagues. With the typical level-headed Czech view of things, calm perseverance and even a touch of humour, they were able to accomplish a task that with a more pessimistic attitude would seem to be totally impossible.

I am sure I will express the feeling of many participants, real and virtual, and indeed of our community, if I send a Big Thank You to Radek, Pavlina, Ivana and all our Czech Friends who have accomplished the impossible!

IUCR CONGRESS

# A very different General Assembly




IUCr Executive Secretary Alex Ashcroft (left, at home) and outgoing IUCr President Sven Lidin (in Prague) at the third General Assembly session.

Alex Ashcroft

The 25th Congress and General Assembly of the IUCr was a voyage into the unknown for all of us. In the entire history of the IUCr, there had never previously been a hybrid meeting, or a quadrennium, for that matter. The 1989 General Assembly is listed as “Extraordinary”, as it was held in addition to the scheduled meetings. While the Prague Congress was scheduled (albeit for a year earlier), this meeting was also “extraordinary” in a very different way. The organisation of the Congress was done by Radek Kužel and his team, and I thought they did an excellent job under extraordinarily difficult circumstances. My only involvement was in the General Assembly: three short sessions with fewer than 100 delegates, in comparison with a full week with a whole order of magnitude more participants. The level of commitment and attention to detail from Radek and his team, over a much longer period than usual – preparing for a meeting in 2020, then one in 2021, with the uncertainty as to whether it could be physical, virtual-only or hybrid – was remarkable.

Preparation for the General Assembly (as with the Congress) was made more complicated as many of us didn’t know until about a week before it was due to happen whether we would be able to attend in person or not. Outgoing IUCr President Sven Lidin only received permission to travel from his University at around the same time that it became clear that Czech immigration would not allow UK citizens to enter the country, which made it impossible for me to travel. Many national committee delegates faced the same uncertainties, while Radek had to make guesses about room sizes, catering and who would be able to present in person.

With delegates from many countries unable to travel, and a statutory requirement for secret balloting, a new voting system had to be used. I had taken advice from the International Science Council, and from other International Scientific Unions, who had suggested the “Simply Voting” software, and I was very grateful to members of the Executive Committee (EC), Chester staff and my family who participated in various test votes while I got used to the interface, we satisfied ourselves that it would be secure and easy to use, and we worked out what the limitations of the software would be. From discussions with the EC, it was clear that the priority was that the voting procedure we used would conform to the IUCr’s rules and that the same people would be elected as would have been elected had all the delegates been in a room together and following the usual processes. It was also crucial that everyone who was entitled to vote should have the opportunity to do so, irrespective of the time difference between Prague and their location. The downside of this was that it stretched the voting period out over a number of days rather than hours, which caused inconvenience to the delegates and prolonged suspense for the candidates. I

want to take this opportunity to thank all the delegates for continuing to participate through their weekend and, in many cases, travelling days, and to thank the candidates for their patience and good grace throughout the process. I would also like to thank the Tellers: Kari Rissanen from Finland and Eduardo Granado from Brazil, for their help with the process and ensuring that the procedures we were using were fair.

It was a very strange experience participating in the General Assembly sessions from home. It would have been lovely to have had the chance to finally meet some of the many people I have had email contact with over the last four years, and to catch up with EC and Finance Committee members, and some of the people I enjoyed meeting in Hyderabad. Despite the lack of physical contact, there was still a very strong feeling of community during the sessions. I could communicate with outgoing General Secretary Luc Van Meervelt *via* the Congress software chat channel, I had a WhatsApp chat with incoming Vice-President Santiago García-Granda and outgoing IUCr President Sven Lidin on my phone, which was very useful for sharing information, and I received numerous extremely helpful emails during the General Assembly sessions from delegates who brought things to my attention that I could pass on to Sven *via* Santiago. With roll-call and test votes on the first two sessions and the official balloting in the third, it was a busy time for the delegates and the organisers. The part of Chester where I live suffered a brief power cut less than 10 minutes into the second General Assembly session that caused a brief spike in my stress levels until it was clear that my Mac and broadband router had both re-booted without problems!




The new Executive Committee against a background of Calgary, Canada, which will host the 27th IUCr Congress in 2026.

I was very relieved that the voting went smoothly, and that the delegates were very tolerant of both the intrusion into their time that voting required and of my pestering emails reminding them to vote. I am looking forward to working with the new President, Hanna Dabkowska; the new Vice-President, Santiago; the new General Secretary and Treasurer, Bo Brummerstedt Iversen; and the newly elected EC members: Thomas Proffen, Angela Altomare and Manfred Weiss. It would have been nice to shake the hands of the outgoing EC members, with whom it has been such a pleasure to work over the past four years, though. I hope I will get to see them all again in Melbourne, but to Luc, Radek, Marvin Hackert and Wulf Depmeier (as well as the continuing EC members) – thank you for making my job so enjoyable over the past four years. The 25th General Assembly was very different, and very stimulating, but I hope the 26th will be more traditional.

IUCR CONGRESS

# Reports on the 25th IUCr Congress




When the Czech and Slovak Crystallographic Association (CSCA) won its bid, at the 23rd IUCr General Assembly in Montreal in 2014, to hold the 25th anniversary IUCr Congress and General Assembly in Prague in 2020, it had no conception of the huge challenges that lay ahead. Nevertheless, the organizers triumphed and produced an excellent hybrid event in 2021 that is being hailed as the beginning of a new era. A round-up of the Congress by the Chair is published below, followed by a number of reports from Plenary Lecture and Session Chairs.

## A hybrid Congress in a time of pandemic, August 2021

Radomír Kužel, IUCr25 Chair

After all, the Congress was realized basically as planned in the last few months as a hybrid event and in a very enjoyable atmosphere for both on-site and online participants, as attested by the emails received after the congress (see below). The possibility for online presentation was a very positive aspect because for invited speakers this can be quite simple and much cheaper. Only a few approached speakers declined the invitation. There were more than 2500 pre-registered people and the total number of final registrations reached nearly 1700. Because of travel restrictions, less than one third of those registered were able to travel to Prague; most of these were from Poland and Germany although others arrived from France, Spain, Sweden, Denmark, Finland, Croatia, Italy, USA and other countries.



## Programme

The high-quality programme consisted of 3 plenary lectures, 5 lectures connected with prizes, 35 keynote lectures, 103 standard sessions, 7 special sessions, a Software Fayre – always 9 sessions in parallel, 55 poster sessions and several commercial presentations. In total 624 lectures and over 550 posters in different forms were presented at the Congress.

Significantly, two sessions were devoted to the structural biology of coronaviruses and to new methods to fight the pandemic. In biocrystallography, the highest number of abstracts were submitted to the sessions on structural biology of enzymes and bioinformatics. The new W. H. and W. L. Bragg Prize Lectures were also related to biocrystallography – Structure-guided design of next-generation malaria vaccine (Jean-Philippe Julien, Canada) and Targeting COVID-19 Viral Enzymes in an Evolving Landscape of Publishing and Peer Review (James Fraser, USA) – as well as the plenary lecture by David Eisenberg: The structural biology of pathogenic amyloid fibrils, which are associated with many degenerative diseases such as Alzheimer's.




The Ewald Prize Lecture and one plenary lecture were related to crystallographic data and databases, the Ewald Prize having been awarded to Olga Kennard (UK) for the development of the Cambridge Structural Database, and the Plenary Lecture by Helen Berman (USA) describing the history of the growth of the Protein Data Bank. Several sessions and the pre-Congress school were devoted to electron crystallography (structural studies by electron diffraction). The school, which had nearly 200 registered participants, was mostly online but the last day was in the hybrid format. In this branch, the Gjønnes Medals were awarded to Sven Hovmöller (Sweden) and Ute Kolb (Germany). Another significant topic of the Congress was quantum crystallography. For both of the above topics, a relatively large number of experts were able to attend in person. Among materials, those attracting the most attention were materials for energy conversion and storage often studied *in situ* (also the subject of Clare Grey's plenary lecture: *In situ* and *ex situ* studies of battery materials with magnetic resonance and diffraction methods), nanomaterials and magnetic materials. The highest number of abstracts were in the sessions "Complex structure of minerals and inorganic compounds" and "Phase transitions in complex materials". There was also much interest in the session on disordered materials. Despite there being little publicity about the Congress among chemical crystallographers, the highest number of registered people was from this area – materials important for pharmaceutical industry, metal–organic frameworks (MOFs) and catalysis. Two sessions were dedicated to the application of crystallography in art, and many sessions to individual techniques such as neutron scattering, small-angle scattering, synchrotron radiation, XAFS *etc*.

All the congress materials – recordings of all lectures, posters and other recordings (meetings, concerts) are available for viewing by registered participants till the end of November 2021 (www.iucr25.org, iucr.gcon.me).

All responses of local and remote participants show that the congress was successful in most aspects and better than expected considering that a number of people were sceptical about the hybrid format. Perhaps, it can be taken as an example for future hybrid congresses. Credit should be given to all people involved in the organization. For the good impression of online participants, probably the greatest credit goes to T.R.I., their gCon presentation system and their very professional approach. We were also very satisfied with the Conftool registration system.

The relatively long absence of real events was reflected in a positive acceptance by the onsite participants, the work of the staff of the Prague Congress Center and the catering company, Zátiší. All

were doing their best for the success of the conference. Of course, the organizers should be mentioned first: colleagues from the Faculty of Mathematics and Physics - Milan Dopita and other colleagues and students; people from the Institute of Organic Chemistry and Biochemistry CAS (Pavlína Řezáčová, chair of the Local Organizing Committee); Faculty of Science, University of South Bohemia in České Budějovice (Ivana Kutá Smatanová, chair of the International Programme Committee); from the Institute of Biotechnology (CAS); and a very important group, Auletris (headed by Martin Haloun).

In addition to the scientific programme, a social programme was organized – boat trips on the Vltava river, the conference dinner with a Brand New Band concert in National House and a train trip to Pilsen brewery. For more see https://www.iucr25.org.

Different conference statistics are available at https://iucr25.org/hybrid-conference-statistics/

## Feedback

*Dear Radek Well now! The organization of the Congress was fantastic and everything including the remote presentations and interactions were perfect. I congratulate you and your team in achieving the seemingly impossible. This Congress will surely go down in everyone's memory as a very special success. I am lost in admiration for what you have been able to do. Kind regards Mike*

*Dear Radek, Pavlina, Ivana, and all Czech Friends, What you have done with the 2020/21 IUCr Congress organization is beyond description. I am writing to express my highest appreciation and congratulations to you and your teams: you have overcome the impossible and organized an amazingly successful IUCr Congress. The organization was flawless but also the scientific content was magnificent. From my point of view (a very active on-line participant) everything worked perfectly; and the quality of the virtual environment was breathtaking. A quantum leap in the mastery of IUCr congress organization. With my all best wishes, Mariusz*

*Dear Radek, Please allow me to heartily congratulate you and also your team for the organization of the wonderful IUCr Congress. You did an excellent job and organized a great conference. As an on-line attendee I could fully enjoy the meeting. It is a huge advantage that the lectures will be available on-line for three more months. As you said in your closing remark, it is the start of a new era. I believe that the in-person meetings are very important but from now on hybrid conferences and archiving the conference events are recognized and will be followed. I both liked the lecturer and the chair functions of the gCon platform. The poster session was the best I have ever experienced. All best wishes, Petra*

*Dear Radek, the congress is almost at the end. Since I will not be able to do this in person, let me send you my biggest and warmest congratulations for what you have achieved, it has been a splendid congress with an excellent program and a perfect organization. You and your colleagues have been fantastic. Thank you so much for allowing us to gather in this novel mode, but with really common and joint spirits. Alessia*

*Dear Radek and Ivana, I had to leave the Congress early to join my family for a vacation. I just wanted to let you know that I think you have done a fantastic job organizing the Congress, especially under these difficult circumstances. Everything was great at all levels of organization, and I think the scientific quality of the meeting was very high. I have heard similar opinions from many other people. I think the Congress was definitely a success. Thank you for all your work! With best wishes, Marcin*

*Dear Radek, Ivana, Pavlina, and Martin, Congratulations on successfully hosting such a wonderful congress. None could have managed the COVID situation better than you did. The content was top quality, and the opportunities to connect with colleagues were many. I spent the whole morning today working through continuing research-related email conversations started during the congress. I'll still look forward to watching recordings of some presentations that I missed. Your hybrid meeting platform will be a benchmark for future congresses. Thank you for so many years of effort to strengthen the union and to make Prague 2020 a great experience. I wish you a speedy recovery from the long weeks of fatigue! Branton*

*Dear Radek, I would like to thank you and all the organizers for an unforgettable congress. Every IUCr congress stays in my mind as an inspiring and engaging experience from a scientific point of view. This time I would say that, looking at you and at your team, I learned from your example how it is possible to face unpredictable events and challenge them by exploring new paths without losing your smile and kindness. As a chair of a session I would like to acknowledge your support in all the phases of the difficult task of assembling the program and the support of the technical team during the session. This IUCr congress was a remarkable event for me as a scientist and more important as a human being. I enjoyed every moment! Grazie di cuore. Consiglia*

*Dear Radek, It was an amazing Congress and the job you and your teams (all of them) did is unprecedented! We all enjoyed the Prague Congress even though we were not there. You proved that it is possible to "do" a big, hybrid meeting, many others before you tried and failed. You, "The Prague", showed a new way. Frankly I prefer old way - but this IS the way of the future. Hanna*

## Plenary Lecture 1: The data universe of structural biology

## Speaker: Helen Berman

Chair: John Helliwell




There are just three Plenary Lectures at an IUCr Congress. At IUCr25 one of these was delivered by Dr Helen Berman on the Protein Data Bank (PDB). We highlight this slide of Helen which so neatly captures all the decades of the PDB including the diversity of the curated structures, technology developments and the very active community involvement in the PDB.

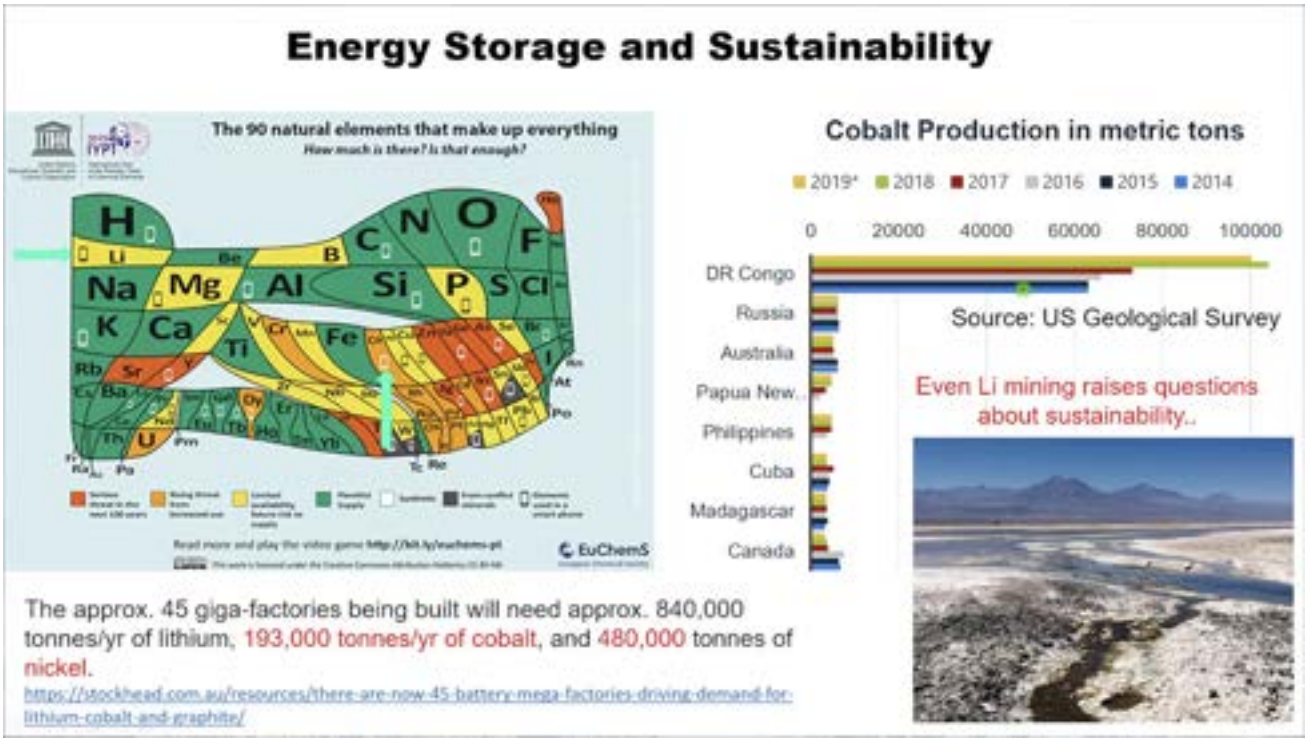
Dr Helen Berman delivered one of the three Plenary Lectures. Helen's career commenced at the University of Pittsburgh, USA, where she received her PhD under the supervision of George Jeffrey in 1969. Helen moved to the Institute for Cancer Research in Philadelphia, where she began her studies of nucleic acid complexes with carcinogens and drugs. She continued her research in nucleic acid interactions at Rutgers University, where her interests in computing and databases led to her work as Director of the Nucleic Acid Database and as Director of the Research Collaboratory for Structural Bioinformatics (RCSB) Protein Data Bank (PDB). Helen was there at the inception of the PDB in 1971 at the Cold Spring Harbor Conference on Quantitative Biology Symposium on the "Structure and Function of Proteins at the Three-Dimensional Level", which she described in her Lecture, and onwards to the

present day. Under her leadership the scientific and educational mission of the PDB has greatly expanded. This included working with film producers in Hollywood on programmes to explain medical themes such as HIV. Helen has been recognized with many awards, notably the 2006 Buerger Award from the ACA and the 2012 Carl Brändén Award from the Protein Society. As Chair of Helen Berman's Plenary Lecture at IUCr25 I concluded the Plenary session by offering a vote of thanks to Helen and all involved in making the PDB what it is today, a fantastic resource.

## Plenary Lecture 2: *In situ* and *ex situ* studies of battery materials with magnetic resonance and diffraction methods

### Speaker: Clare Grey

Chair: Jürgen Senker

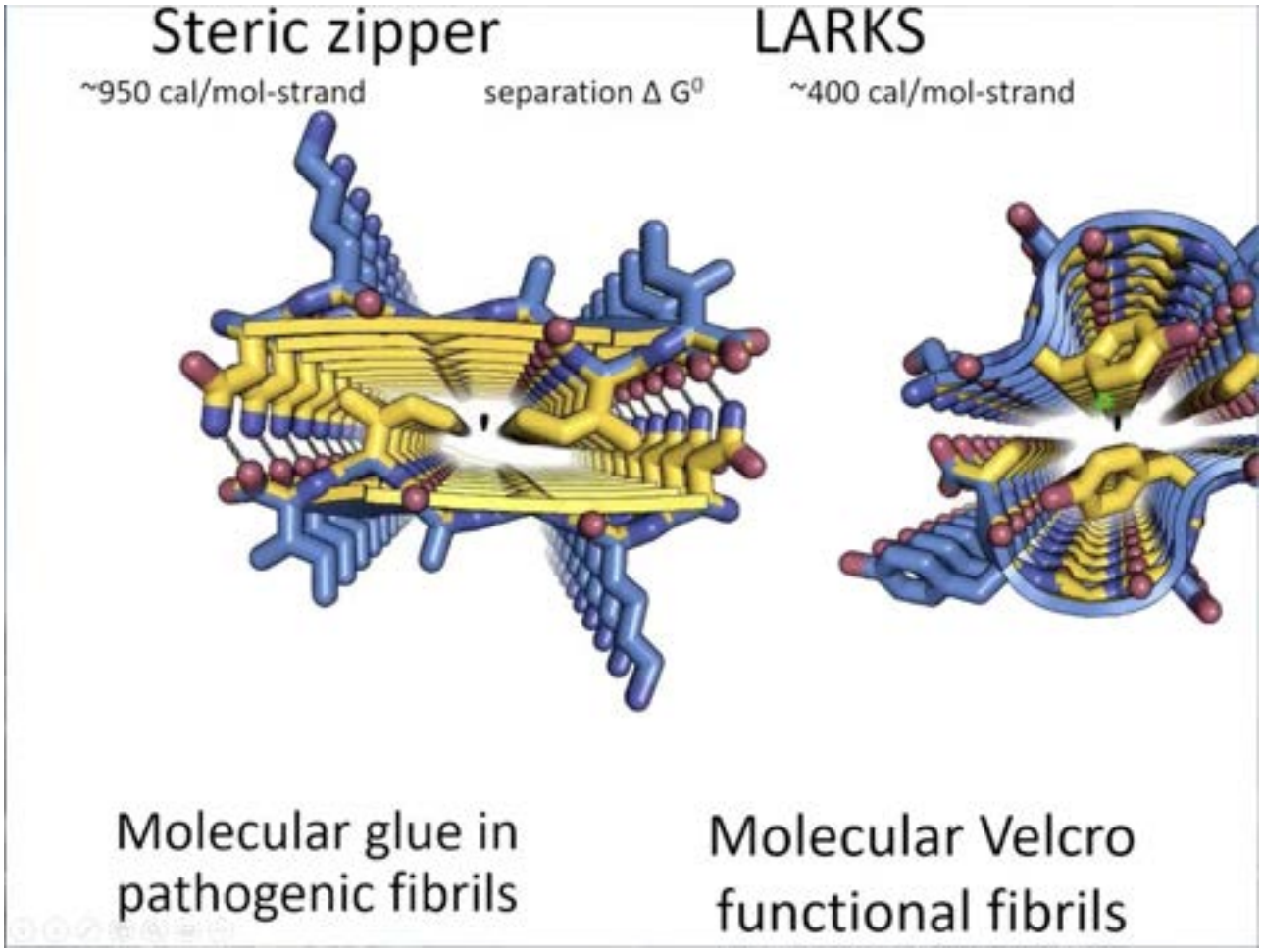



Clare Grey started her academic career in Oxford, UK, in the group of Tony Cheetham. After receiving her PhD in 1991, she first joined the Veeman group in Nijmegen as Royal Society Post-doctoral Fellow and then DuPont as a visiting scientist. From 1994 onwards, she worked as Assistant, Associate and Full Professor in Chemistry at the State University of New York. Since 2009 she is Geoffrey Moorhouse Gibson Professor in Materials Chemistry at the University of Cambridge and Adjunct Professor at Stony Brook. Clare is one of the leading experts on *in-situ* and *operando* studies of modern battery materials with diffraction and solid-state NMR spectroscopic methods. Clare presented a thrilling and lively lecture, summarizing the current state of the art while simultaneously highlighting ongoing challenges. By focusing on lithium-ion batteries, she could stress the impact such studies have on our daily life. With illustrative examples of her research, she pointed out pros and cons of various cathode and anode materials, explained the conundrum of stable $LiCoO_2$-type MMM phases using high nickel content and demonstrated how to follow charging and discharging cycles of batteries *in situ*. In particular, her studies offer unique insight into the complexity of chemical reactions at the electrodes, including the continuous growth, dissolution and segregation phenomena of intermediate solid phases. By covering also materials for sodium-ion batteries, she introduced the controversial discussion about next-generation batteries, desired to be able to replace the less abundant lithium.

## Plenary Lecture 3: The structural biology of pathogenic amyloid fibrils

## Speaker: David Eisenberg

Chair: Lars Redecke

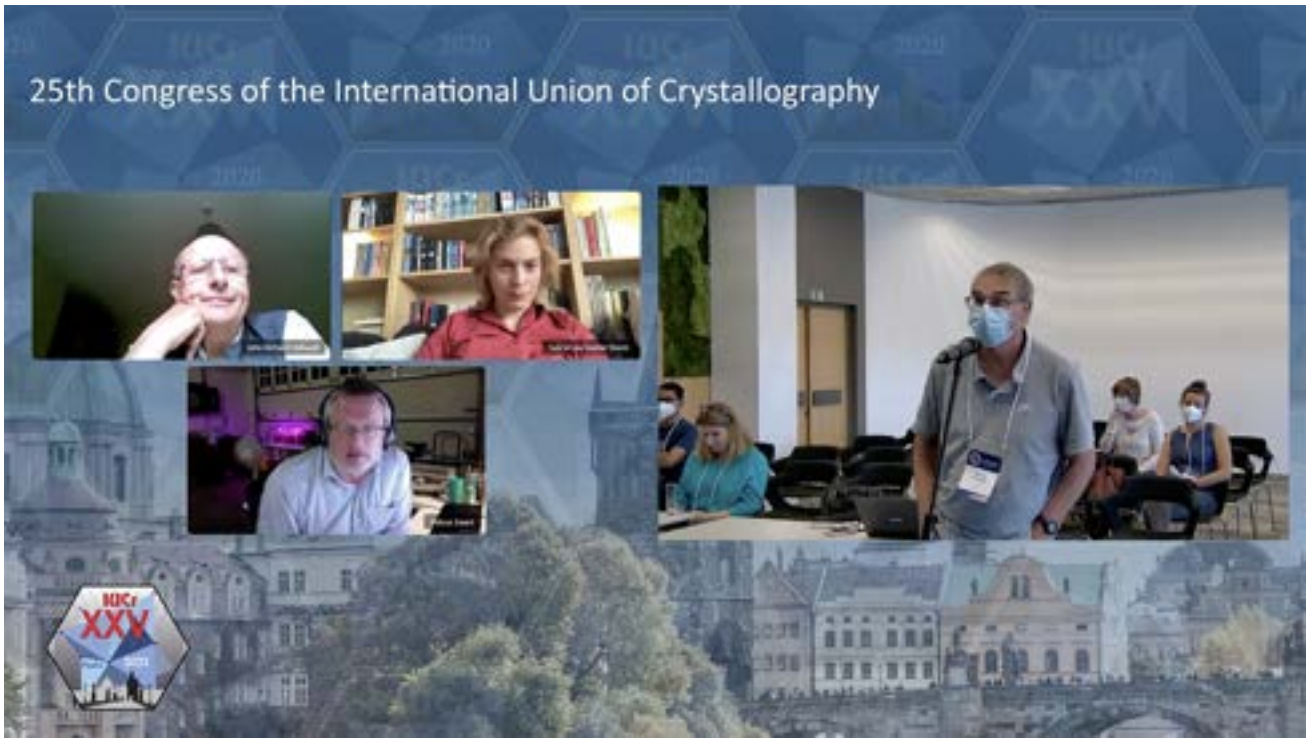



David Eisenberg (UCLA, USA) presented the current knowledge of the structure and stability of pathogenic and functional amyloids, complemented by promising results in structure-based aggregation inhibitor design. Amyloid fibrils are assumed to be the causation of several diseases, including many of the most prevalent degenerative disorders. More than 80 small segment structures from 20 amyloid-forming proteins revealed common features of pathogenic amyloid structures: two or more protofilaments are twisted around each other, each protofilament is a stack of identical bent protein arches from beta-sheets, representing 2D structures that interact *via* energetically highly stabilized homotypic steric zippers, while highly polarized backbone H-bonds connect the layers. Although polymorphs of identical sequences are common, preferred polymorphs are usually isolated from diseased brains, since a specific polymorph might cause the disease, or is formed by the disease condition. Atomic level insights into fibril structures enabled the design of peptidic inhibitors that bind the growing fibril as a cap, preventing the addition of new strands, as confirmed by using seed-containing brain extracts and biosensor cells producing fluorescence-labelled tau protein. But labile amyloid-like fibrils are also formed by a large number of non-pathogenic proteins containing low-complexity amyloid-like reversible kinked segments (LARKS). LARKS may represent adhesive segments in functional fibrils, which are less stable compared to pathogenic fibrils, and mostly monomorphic. However, mutation can transform functional amyloids into pathogenic amyloids.

## MS-7: High throughput vs. careful planning: How to get the best data?

Chairs: John Helliwell and Selina Storm

In times of highly brilliant beamlines and fast detectors, data collections only take seconds – but what is the way to get the best data? Aina Cohen presented SSRL's efforts to enable more specialized experiments such as experiments at room temperature, controlled dehydration experiments or light-triggered experiments remotely, making specialized experiments possible in times of crises. Danny Axford, senior beamline scientist at Diamond's beamline I24, exploits their brilliant microfocus beam in multiple crystal approaches and serial crystallography, pushing the limits of fast data collections and highest quality data. Peter Zwart from LBNL demonstrated that the use of Gaussian process regression in high-throughput infrared spectromicroscopy reduces the amount of measurements and significantly speeds up the measurements. Marcus Müller (Dectris, Switzerland) first remembered his colleague Andreas Förster, who was a highly valued member of the community and who had sadly passed away in May 2021. Marcus then presented the best strategies of collecting data with hybrid photon counting detectors, ranging from fine slicing to strategies for phasing strategies at room temperature. In the next talk, Amy Thompson (University of Queensland, Australia) introduced her approach of automatic analysis for elastically flexible crystals called CX-ASAP, reducing the processing time significantly. Martin Malý from the Czech Technical University then reported a new approach to assess the optimal resolution cut-off with *PAIREF*.




Screenshot taken during MS-7, contributed by Samar Hasnain, which encapsulates the unique nature of the Congress. The four people in action are taking part from different places; only one is in the live, socially distanced, audience.

Can we answer the question whether the best data are obtained *via* high throughput or careful planning? As the late and great Michael Rossmann indicated, it all depends on the challenge you are facing. For his pioneering work with highly radiation sensitive virus crystals there is but one option: "shoot first and ask questions later" (Rossmann & Erickson, 1983). Since that time, the development of incredible detector and beamline capabilities has expanded the possibility to the researcher to 'just open the shutter'. Furthermore, the huge amount of data that can be obtained within a short time creates new challenges, which require new solutions such as those presented in this session.

How to get the best data depends on what your objectives for your measurements are, which should certainly be carefully planned. This is a similar overall conclusion to the individual conclusions of the speakers in this session spanning a quite vast array of topics.

## Reference

Rossmann, M. G. & Erickson, J. W. (1983). *J. Appl. Cryst.* **16**, 629–636.

## MS-8: Social media and new frontiers for spreading crystallographic information

Chairs: Andrea Ienco and Helen Maynard-Casely

The aim of this microsymposium was to hear how social media and other tools are being used to communicate crystallographic research. After the massive efforts of the International Year of Crystallography in 2014, how has the science communication landscape for crystallographic ideas changed? The session had two invited speakers, Delphine Chenevier (European Synchrotron Radiation Facility, France) and Rhiannon Morris (Walter and Eliza Hall Institute, Australia). Delphine highlighted the importance of a strategy for approaching social media, and how important it is for science to show its human face – The 'Humans of ESRF' project is a fantastic example of that.[1] Rhiannon, who has a large following across social media where she talks about her research and structural biology at large, emphasised that there are many ways to use social media, and broke it down into four types of use illustrating this with numerous examples.[2]

Following on from our invited speakers, we had four contributed talks. Ilaria Gimondi (Cambridge Crystallographic Data Centre, UK) presented on how they are using social media to promote the (now one million) structures in the Cambridge Structural Database to a wide range of users (from kids to researchers [3]) – including their successful hashtags #FeatureStructureFriday and #TopTipsTuesdays (also those attending #IUCr25 check out the #CSDLeaderboard). Sneh Patel (University of Auckland, New Zealand) talked us through how memes can be really flexible to convey complex science ideas and foster cohesion in many different groups.[4] Elizabeth H. Driscoll (University of Birmingham, UK) presented the awesome tactile Battery Jenga project and discussed how to make social media and science outreach accessible to all.[5] Paolo Mazzeo (University of Parma, Italy) illustrated how important social media has been as a tool for the Young Crystallographers of Italy group, especially in COVID times, including a series of interviews with crystallographers on YouTube.[6] He also introduced a very exciting new comic-book project.

The session was enjoyed by 30 viewers online, as well as a capacity crowd at the conference centre in Prague. The wide range of topics and perspectives shared by the speakers, communication experts as well as researchers engaged in science communication, very much showed how important sessions like this are for sharing ideas to make crystallography as relevant as we can.

## References

[1] https://humans.esrf.fr

[2] You can follow Rhiannon on Twitter @Scientist_Rhi and on Instagram @scientist_rhi

[3] https://www.ccdc.cam.ac.uk/Community/educationalresources/; https://www.ccdc.cam.ac.uk/Community/educationalresources/activities-for-kids/

[4] Inorganic Memes for C2v Teens https://www.facebook.com/inorgmemes/; X-ray Crystallography maymays https://www.facebook.com/crystallographicmemes/

[5] Driscoll, E. H., Hayward, E. C., Patchett, R., Anderson, P. A. & Slater, P. R. (2020). *J. Chem. Educ.* **97**, 2231–2237; Lithium Shuffle https://www.rsc.org/news-events/community/2020/mar/lithium-shuffle/ (accessed January 2021)

[6] Young Crystallographers of Italy YouTube Channel https://www.youtube.com/channel/UCroIyv9r_oYEXwbSApAI_9g



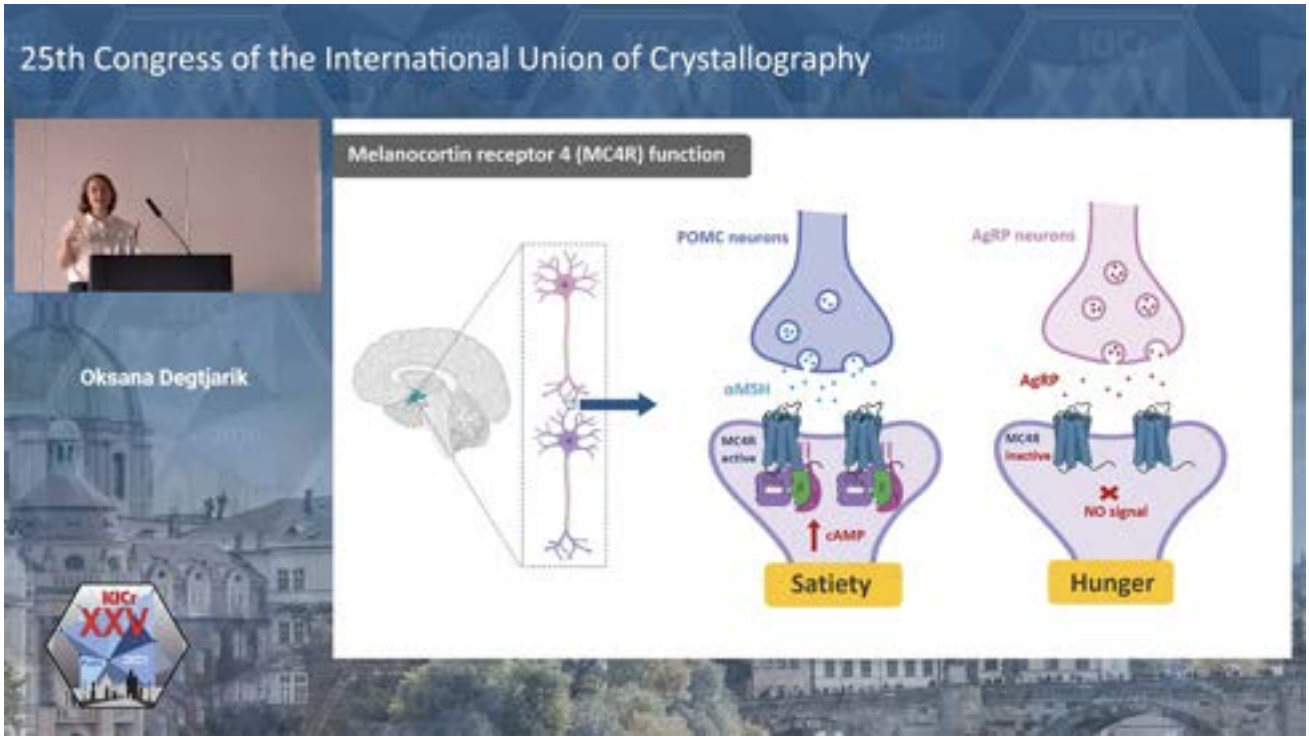

Left: a crystallographic meme. Centre: Battery Jenga and the Lithium Shuffle. Right: the comic-book project.

## MS-74: Structural biology of receptors, signaling and membrane proteins

Chairs: Susan Buchanan and Michael Parker

We were honored to co-chair a microsymposium on transmembrane signaling, a very active area of structural biology research that, despite years of studies and a few Nobel Prizes along the way, still has many mysteries to explore and puzzles to solve. We were fortunate to attract leading speakers from four continents which presented its own challenge with a virtual meeting. Although the session was said to start at 10.20 am in Prague, for the co-chairs it was starting at 6.20 pm for one of us and 4.20 am for the other!

The session was led off by Isabel Moraes (National Physical Laboratory, UK) who described her structural work on the archaerhodopsin-3 transporter with crystal structures resolved to 1.1 Å resolution, quite an achievement for a membrane protein. The high resolution highlighted how minimal displacements of charged and polar groups within the low dielectric environment of the membrane can induce changes in the chromophore conformation. Our next speaker was Robert Keenan (University of Chicago, USA) who described his cryoEM work on a ~360 kDa ribosome-associated complex comprising the core Sec61 channel and five accessory factors which revealed a large assembly at the ribosome exit tunnel organized around a central membrane cavity. Oksana Degtjarik (Weizmann Institute of Science, Israel) gave a fascinating talk on the cryoEM structure of the melanocortin receptor 4, a G protein-coupled receptor that plays a key role in regulation of food consumption and energy expenditure, bound to an agonist recently approved for the treatment of obesity. Michael Healy (University of Queensland, Australia), presented crystal structures of components of the Commander complex, an ancient and conserved regulator of intracellular trafficking. Bronte Johnstone (University of Melbourne, Australia) described the crystal structures of a bicomponent pore-forming toxin from a bacterium that lives in the midgut of mosquitoes. The session was rounded off with a talk from William Bourguet (Center for Structural Biology – Montpellier, France), who described his studies on the retinoid X receptor/pregnane X receptor heterodimer, which plays an essential role in controlling xenobiotic responses, which revealed its binding promiscuity and ability to accommodate bipartite ligands. We thank all the speakers for very stimulating talks which highlighted the full gamut of structural tools that can be deployed to answer key biological and medical questions.




## MS-96: Crystallography schools to promote interdisciplinarity in science

Chairs: Alice Brink and Serena Chiara Tarantino

This microsymposium described a vibrant and lively crystallographic community, actively and tirelessly engaged in a plethora of educational and outreach initiatives. The intention is to support the growth of researchers and provide tools to allow crystallographers to meet the demands of interdisciplinary challenges within the future scientific research fields and to strengthen the unifying role of crystallography. Our speakers came from diverse backgrounds across the globe and presented many initiatives, which promotes interdisciplinarity.



Group photo of MS-96 chairs and speakers taken in the Congress Virtual Foyer. Marielle Agbahoungbata and Dubravka Sisak do not appear in the photo.

D. G. Lamas (Argentina) described several education and outreach activities carried out throughout Latin America in the past year. He highlighted how the intrinsic interdisciplinary character of these activities enriched all participants. M. Y. Agbahoungbata (Benin) described the X-TechLab, a regional crystallographic research and training platform, the result of the interaction between the Lightsources for Africa, the Americas, Asia, Middle East and Pacific (*LAAAMP*) and the Sèmè City hub, one of Benin Government's flagship projects, which aims to create a world-class knowledge and innovation centre in Africa. D. Sisak (Switzerland) gave a talk on how the use of social media, industry–academia collaborations, and online interaction tools in both real and virtual schools can contribute to build and maintain crystallographic communities. L. Suescun (Uruguay) gave a personal 10-year perspective on teaching in crystallography schools in Latin America and shared some of the strategies he developed to improve understanding of symmetry in a class of students coming from different backgrounds. L. Dawe (Canada) described their methods to develop high impact crystallography skills through local undergraduate curricula and the presentation of regional workshops and schools, with particular reference to experiences in the ACA region. S. Ward (UK) highlighted efforts made by CCDC to cultivate more interdisciplinarity in science and engage scientists across research areas and ages through their involvement in a variety of schools, workshops and science festivals globally and by the creation of more virtual resources.

IUCR CONGRESS

# My Congress experience



Leopoldo Suescun settles down to watch the Opening Ceremony from his home in Uruguay.

Juanma García-Ruiz, Leopoldo Suescun, Louise N. Dawe


## Juanma García-Ruiz

## Instituto Andaluz de Ciencias de la Tierra, Granada, Spain


Radek (Kužel), Ivana (Kutá Smatanová) and Pavlína (Řezáčová) make an awesome trio. The IUCr Congress that they organized along with their team in circumstances as complex as those imposed by the pandemic was an extraordinary success. From my point of view, it has been a model congress in various aspects. The organization on-site was lovely, with many young people ready to help you, a reasonably wide social program with music playing a central role, and outstanding catering for everyday coffee and lunch breaks. I still remember the taste of the spinach cream, the goulash and the Mexican (kind of Azteca) soup provided during the normal days of the Congress.

I guess everybody will agree that the Conftool platform used for registration and abstract submission, reviews, *etc*. was excellent. The rooms for microsymposia and keynote and plenary lectures were well equipped. Two technicians in each room made sure that audio and video quality was under control, while young people in the rooms made sure that the audience used the microphones so that everybody in the world could participate.

But it has also been a model as a virtual congress. My experience as chair and as a lecturer on-site confirms it. I arrived late to my first session as chair on-site. I was nervous about not managing the situation because the keynote speaker was already waiting for me in her Institute. However, the technicians in the room explained to me in one minute (textual) how to use the platform. And it worked perfectly. I think that the next IUCr Congress in Melbourne and other conferences and workshops could learn very much from the excellent platform used for the hybrid presentations – gCon developed by T.R.I. In the near future, I guess that hybrid conferences, allowing virtual and live presentations, will be the norm. This is why the Prague Congress will be a landmark and a mirror where future organizers will look for information and methodology.

We know that big challenges offer big opportunities. Radek, Ivana and Pavlína took this big one and imagined new solutions for IUCr2020/2021 and the future. And yes, this will be the Congress where for the first time, we all started our presentations with "Good morning, good evening, good night, wherever you are in the world."

**Leopoldo Suescun**

**Universidad de la República, Montevideo, Uruguay**

- "I will set the alarm clock for 03:45," I said to my wife on Saturday 14 August.

- "I will need to shift my timetable to adjust to the Congress for the next week."

- "Don't be noisy. I'm not attending" was her laconic answer.

That was the beginning of an intense week of workshops, lectures, short talks, chats and online encounters with friends and colleagues from all around the world.

The hybrid IUCr Congress was a great event I enjoyed almost as much as if I had been there.

I must confess I was not very ready. Being at home, travelling daily to the office, with normal everyday activities, and no 24-hour period of travelling, hadn’t given me the usual impetus to dive into the details of the extensive program, so I only had a few scheduled events in my calendar (opening ceremony, my students’ posters and my microsymposium), those I could not miss. Having barely examined the program, I had explored the gCon platform even less in order to see how things were going to work. Surprisingly, a few hours into Saturday afternoon (my time) the straightforward use of the platform was clear and my enthusiasm was high.

I greatly enjoyed and made profit from the flexibility of the platform. The chat space was good to look for colleagues and drop some lines, and also receive greetings and questions.

The virtual foyer looked awkward in the beginning, but turned out to be very useful for wandering around, finding colleagues by chance and enjoying an informal videoconference or a planned meeting. The Questions tab in the talk windows was also a great way to interact. Further proof that I was there. Last, but not least, switching from one session to another in a couple of clicks truly enabled me to see a lot more of the Congress scientific program than I usually do.

If I had to give a suggestion for improvement for future hybrid events, I would change the person-to-person video conference capability in the poster sessions. First, it was rather short, second it felt awkward to enter a virtual room with someone you probably don't know and start asking questions. Maybe if this feature was integrated into the virtual foyer, where you could see not only the posters without necessarily approaching the presenter but also the presenters and the circles of people asking/discussing with him/her, in a more public/less personal setup it would be easier to interact and also an experience closer to the in-person one.

Regarding the scientific talks, I am looking forward to watching all those I missed, so I can't choose the one I liked the best yet. However, there is a short list of colleagues I have already contacted after their very inspiring talks. If I had to choose a couple that I enjoyed a lot, those were the Keynote Lecture by Harold Stokes and the Ewald Prize Lecture honoring Olga Kennard given by Suzanna Ward.

Besides all the very interesting talks I attended, the microsymposium that inspired me the most was MS-8 "Social media and new frontiers for spreading crystallographic ideas" co-chaired by Helen Maynard-Casely and Andrea Ienco. As a social media user and follower of Helen on Twitter, I found the session to be even more entertaining and informative than expected. It was perfect to have it on Sunday because it charged my batteries for the great science to come. This kind of microsymposium should be repeated at future congresses, as it helps us find better ways to communicate our science to the public and also to our students. Following this session, I decided to include a meme at the end of every class in my Crystallography course next year. Maybe I'll share them. Follow me (@leopoldosuescun) :-).

As I’m writing this, about a week after the closing ceremony ended, I'm still enjoying many of the talks I missed but also trying to evaluate the event.

I missed the many *na zdraví* I would have enjoyed, the many hugs and handshakes and the many shared mate sips. But the science and the social parts were still there, so I have not missed the fruitful exchange

of ideas and contact with the community. For all the good of this I thank Radek and Ivana who became my friends in Hyderabad, and Pavlina and Hašek, whom I hope I will be able to thank personally in Melbourne.

### Louise N. Dawe

### Wilfrid Laurier University, Waterloo, ON, Canada

I was a virtual participant in the 25th General Assembly and Congress of the IUCr. I chaired the Canadian delegation to the General Assembly; co-chaired the joint bid from the Canadian National Committee for Crystallography, the US National Committee for Crystallography and the American Crystallographic Association to host IUCr2026 in Calgary, Canada; and I gave a lecture in the session on “Crystallography schools to promote interdisciplinarity in science”.

Some of the things that I missed as a virtual participant were joining colleagues over meals, trying local food and drink, and exploring a new part of the world. Surprisingly, what I did not miss was new science and ideas! The virtual platform was excellent. It was simple to navigate and convenient to move from one session to another, without disturbing anyone in the room. Further, the availability of the recordings after the Congress meant that I was able to review lectures that I could not attend in real time and that I could re-watch lectures that I especially enjoyed (for example, the lectures in the session on “Science meets art: Crystallography and cultural heritage”!).

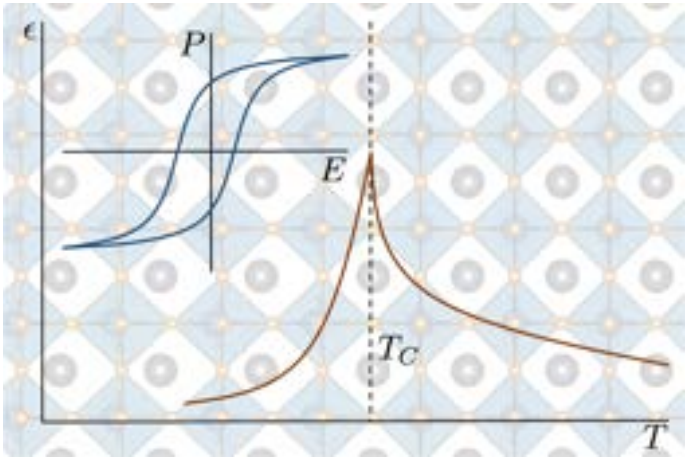
The virtual chat/mail functions enabled me to privately write to presenters as soon as their lecture was finished to ask questions and arrange for individual virtual discussions. It was also perfect for sending little notes to the vendors, and others in the virtual exhibit, even during times when the booths were not staffed.

The quality of the video and audio from the General Assembly was clear, and while I was disappointed not to be present to congratulate our new Executive Committee members and President of the Union, I was still happy to be able to participate in real time with the General Assembly proceedings.

A special thank you to Radomír Kužel and all members of the local organizing committee. It is hard to conceive of the enormity of planning for the Congress and General Assembly amidst a global pandemic. But this team truly broke ground with respect to inclusive meeting practices for our members and created an event that will be a model for the future.

FEATURE ARTICLE

# History of ferroelectrics – a crystallography perspective




Nicola A. Spaldin, Ram Seshadri

"*Underlying the whole treatment is the assumption that the physical properties of a solid are closely related to its structure, and that the first step to understanding the physical properties is to understand the structure*" – Helen D. Megaw [1]

The year 2021 marks the 100th anniversary of the discovery of the phenomenon of ferroelectricity, defined to be a spontaneous electric polarization that is switchable by an applied electric field. This is a wonderful centennial for crystallographers to celebrate because it is a property of crystals in which functionality and structure – specifically the existence of a polar axis – are intimately related. The key characteristic of a ferroelectric is a hysteresis loop of polarization versus electric field; we reproduce the data from the original discovery by Joseph Valasek in Fig. 1 [2]. The similarity with the magnetization behavior of ferromagnetic iron in a magnetic field is clear – just like the well-known hysteretic magnetic field induced switching between the two stable states of opposite magnetization in a ferromagnet, a ferroelectric material has two stable states of opposite *electric* polarization associated with opposite relative displacements of its anions and cations – and it's this analogy that gives ferroelectrics their name, in spite of the fact that most of them are not ferrous! We'll see in this article the key role played by crystallography in the evolution of ferroelectrics (a timeline of key discoveries is shown in Fig. 1), starting from the identification of polar materials, through establishing the nature of ferroelectric phase transitions, to providing crucial information for the development of today's accepted theories of ferroelectric polarization.

## 1. Beginnings

We begin our story in 300 BC, when Theophrastus, the successor to Aristotle in his School of Philosophy, made an early mention of the electrostatic behavior of certain rocks in his treatise *On Stones*. Particularly intriguing is his report that "lynx-urine stone" (lyngourion) is better able to attract bits of straw, wood and metal if the stones are derived from wild male lynx because of their superior diet and exercise regime [4]. The behavior he described is known today as *pyroelectricity* and has the same crystallographic symmetry requirement as that of ferroelectricity – the presence of a polar axis – although not all pyroelectrics are switchable by an applied field. For a discussion of the early history of the electrostatic behavior of pyroelectrics, we refer the reader to reference [5].

The first genuine crystallographic study of the pyroelectric effect occurred in 1785, when Abbé René Just Haüy, affectionately referred to as the father of crystallography [6], turned his attention to the unusual pyroelectric behavior of the mineral tourmaline. The Abbé had by this time established basic laws of crystal form and symmetry through painstaking fragmentation of minerals to reveal their underlying crystallographic planes and angles. He, therefore, set about relating the pyroelectric behavior of tourmaline to its crystalline structure. First, he showed that the electricity in tourmaline was strongest at the poles of the crystal and became imperceptible at the middle, although when divided each new sub-crystal again had electricity at its poles. He also established that the existence of pyroelectricity correlates with an absence of symmetry in the crystal, which we now know provides the polar axis. In an early example of rational materials design, his finding led him to discover the effect in a number of other minerals. Charmingly, when his colleagues secured his release from imprisonment during the French Revolution, he was so absorbed in a crystallographic study that he refused to leave the prison until the next morning. We encourage PhD students to reflect on this on Friday afternoons, although we should perhaps disclose that the prisoners who remained were guillotined the following week.

## 2. Rochelle salt

Haüy's flurry of discovery of new pyroelectrics was continued in particular by David Brewster, who, significantly for our discussion, identified pyroelectric behavior in sodium potassium tartrate tetrahydrate, more commonly known as Rochelle salt [7] [Fig. 1(*a*)]. First made in the 17th century by adding sodium hydroxide to the tartar residue left in wine barrels, Rochelle salt was developed by the Seignette pharmaceutical dynasty of La Rochelle for use as a laxative. Thankfully, these authors can not comment on its effectiveness in this application.

In the century after pyroelectricity was identified in Rochelle salt, a substantial body of experimental observations on this material accumulated. Some of the findings, particularly an anomalously large dielectric permittivity and electro-optic Kerr effect [8, 9], as well as hysteresis both in the piezoelectric response with pressure [10] and in the capacitance with charging direction [11], suggested an electrical resemblance to the ferromagnetic properties of iron [12]. Valasek set about exploring the analogy by measuring the charge on the plates of a Rochelle salt capacitor – a proxy for the polarization of the material – as a function of applied electric field. This simple experiment led him to discover the hysteresis of polarization with applied field, reproduced in Fig. 1 [2], which is strongly reminiscent of the magnetization – magnetic field behavior of a ferromagnet. Sadly, there was a resounding lack of interest given such a remarkable result, in part because Rochelle salt is unstable against dehydration and the results were not reproducible, and also because the behavior was not understood. Theoreticians were confounded by the crystal symmetry, which was incorrectly assigned on the basis of morphology to a non-centrosymmetric but non-polar space group. With 112 atoms per unit cell, a full X-ray crystal structure determination was attempted only 20 years later [13]. Even more unfortunately, the X-ray structure, being transparent to the symmetry-lowering water molecules, confirmed the incorrect assignment! {Not until decades later, when the first neutron diffraction structure on single crystals showed the positions of the polar water molecules that line up to provide the polarization, was the correct polar symmetry identified [14].} And a peculiar behavior observed in only one badly behaved material offered only a rather rickety bandwagon for researchers to jump onto.

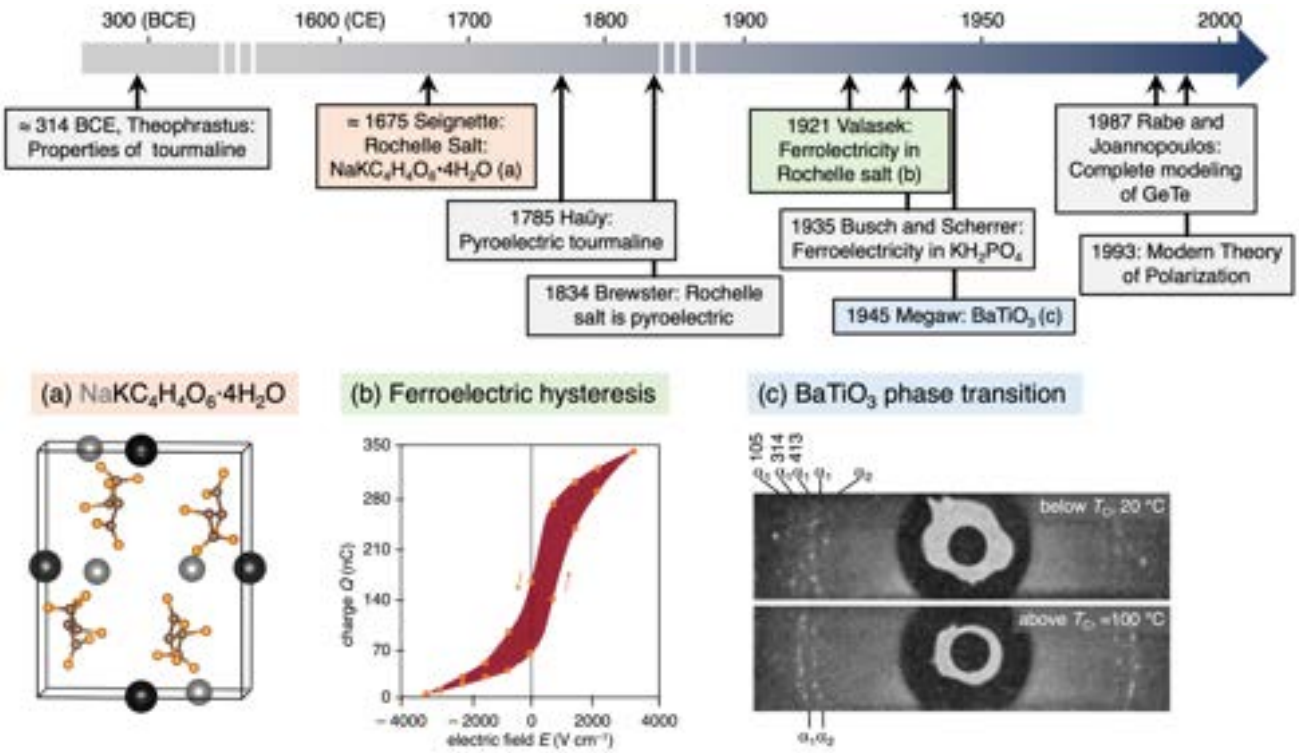



Figure 1. Upper panel: a timeline of some key events in the history of ferroelectrics, from 300 BCE to the present time. (*a*) The crystal structure of the paraelectric phase of Rochelle salt. Hydrogen atoms and water molecules are omitted for clarity. (*b*) The ferroelectric hysteresis loop of Rochelle salt recorded by Valasek in 1921 {data from reference [2]}. (*c*) A reproduction of the X-ray images recorded by Megaw with Cu $K\alpha_1$ and $K\alpha_2$ radiation [3] of high-angle reflections in $BaTiO_3$ powders above and below the ferroelectric phase transition.

## 3. The development of ferroelectrics in the 20th century

The lonely situation improved somewhat in 1935, when ferroelectricity was identified in a second, more tractable material, potassium dihydrogen phosphate, $KH_2PO_4$ or KDP [17]. (Note that the authors called it a seignette-electric in homage to the La Rochelle Seignettes.) In this case, the crystal structure, except for the hydrogens, had already been determined using X-ray diffraction [18], and later neutron studies found the hydrogen positions [19] and showed that their ordering triggered the onset of ferroelectricity.

The most significant breakthrough for the field, however, had to wait until the Second World War, when materials with high dielectric constants were in demand as capacitor components in devices for military communications and detection. One such proprietary condenser sent to the UK Government from the United States mysteriously found its way into the Philips Materials Research Laboratory in Surrey, where it was pulled apart to yield a sample of barium titanate ceramic. Dielectric measurements quickly revealed a very high and temperature-dependent susceptibility, with a number of peaks suggestive of phase transitions. Powder X-ray diffraction was performed by Helen Megaw, who found a tetragonal structure at room temperature and the ideal cubic perovksite structure above 120°C [20], about which she writes “I can still remember my thrill of pleasure in the darkroom when I looked at the photo and saw that the lines were those of a simple cubic material!” [21]. Soon after, ferroelectric hysteresis was measured [22] and single crystals were produced [23]. A robust material, with only five atoms per unit cell and ferroelectric properties, at room temperature, barium titanate $BaTiO_3$ and related compounds were almost too good to be true, allowing for detailed characterization as well as theoretical modeling. In particular, lead titanate $PbTiO_3$, which displays a relatively simple transition from the paraelectric cubic phase to a ferroelectric tetragonal phase just below 500°C (Fig. 2), remains one of the highest-performing ferroelectric materials to date. Collectively, the ferroelectric titanates form the basis of an enormous industrial enterprise, with applications as diverse as transducers, capacitors, data storage and computer memories [24].

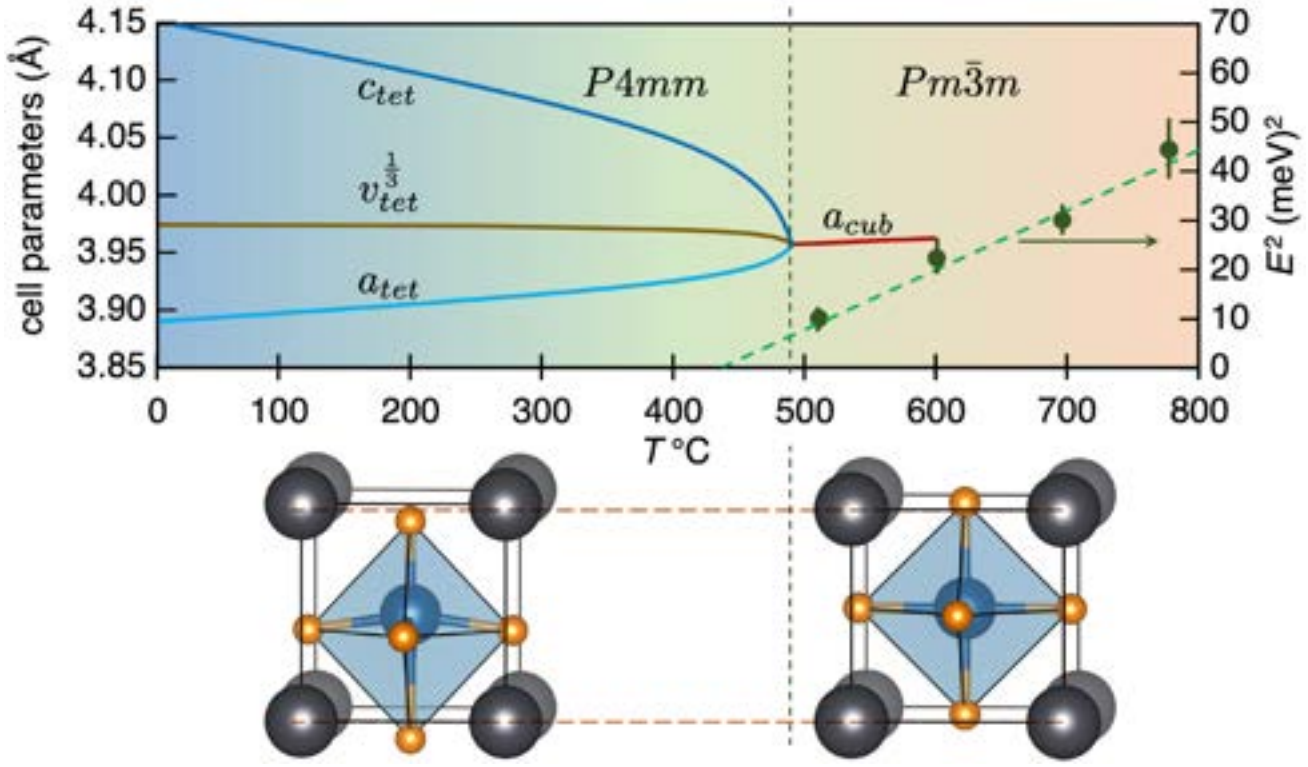



Figure 2. Variation of the cell parameters of perovskite $PbTiO_3$ as a function of temperature across the phase transition {data adapted from reference [15]}. In the high-temperature cubic phase, all three unit-cell lattice parameters have the same value, $a_{cub}$. In the low-temperature ferroelectric phase, there is a tetragonal elongation with the lattice constant along the polar direction, $c_{tet}$, larger than those perpendicular to it, $a_{tet}$, and a small increase in volume, $V$. Data associated with the right ordinate depict the square of the soft-mode energy as obtained from neutron scattering {data adapted from reference [16]}. The energy (or frequency) of this mode goes to zero near the transition temperature. The corresponding crystal structures of tetragonal ferroelectric $PbTiO_3$ at room temperature, and the cubic structure above the Curie temperature are depicted below. The Pb ions are shown in gray, the Ti in blue and the oxygen in orange. The loss of the dipole within the unit cell above the transition temperature is clearly seen from the oxygen octahedron around Ti becoming regular, as is the decrease in the *c* lattice constant from the ferroelectric phase.

Crystallographic measurements of the detailed atomic positions in $BaTiO_3$ were essential in developing the theory of ferroelectricity. In particular, the rather small displacement of the positively charged Ti ion relative to the negatively charged oxygen ions [25] showed that the polarization arose from rather subtle atomic displacements of the atoms, rather than the reorientation of discrete polar entities, and indicated the importance of the ionic polarizability in stabilizing the ferroelectric state [26]. This understanding enabled, in turn, the development of the so-called soft-mode theory of ferroelectricity [27], which recognizes that a crystal is only stable if displacements of its ions result in restoring forces that push them back to their starting positions. As the crystal approaches a phase transition, these forces become weaker, and the frequency of the corresponding atomic vibrations lowers, or softens. In the ferroelectric titanates, it's the polar phonon corresponding to the Ti–O relative displacement that softens and reaches zero frequency at the phase transition (Fig. 2). Verifying this prediction was, of course, more of a source of excitement for spectroscopists than for crystallographers [28, 29]. With the advent of advanced electronic structure methods and improved computing capabilities, a full first-principles/statistical mechanical description of the ferroelectric phase transition became feasible and verified the soft-mode picture [30].

Intriguingly, the most significant modern theoretical development – the so-called modern theory of polarization – shows that, in fact, only *changes* in polarization (for example, between the cubic paraelectric and polar ferroelectric structures) are single-valued, and the general polarization in crystalline solids is in fact a *lattice* of values [31, 32, 33]. This elegantly closes the loop between experiment and theory, with the concepts of crystallography that were so essential in defining and identifying ferroelectricity also proving relevant in its detailed quantum-mechanical description.

## 4. Evaluating the history of ferroelectrics

So why did ferroelectricians have to wait until 100 years ago for the emergence of their field? First, from a crystallographic standpoint, the fact that the polar state is usually formed on cooling from a non-polar parent phase leads to the formation of domains of different polar orientations. These polarization textures can be on the scale of tens of nanometers, rendering multidomain crystals difficult to distinguish crystallographically from their centrosymmetric parent phases. In this context, powder diffraction can often be more revealing than single-crystal experiments. Indeed, the advent of the Rietveld method [34], high-resolution X-ray powder diffraction at synchrotron sources [35] and the joint use of X-ray and neutron diffraction [36] have collectively played an invaluable role in advancing the understanding of ferroic materials.

From a technical point of view, crystallographic measurements in finite electric fields are challenging, as (to quote Arthur von Hippel) “the crystallographer has thus to accept the unusual situation that the *c* axis of the $BaTiO_3$ crystal can be turned around at will by the application of an electric field” [25]. From a practical point of view, the engineering component in the definition of ferroelectricity – that is the ability to switch with an electric field – can not be assessed from knowledge of the crystal structure alone, and indeed before the availability of high-quality thin-film samples the voltages required for switching could be prohibitive.

While much is now understood about the phenomenology of ferroelectricity, there remain many open questions that are particularly relevant to crystallographers. Measurements that probe local structure, such as the use of pair distribution function analysis [37] and aberration-corrected electron microscopy [38, 39], are starting to reveal previously hidden order such as polar nanodomains that are inaccessible to bulk techniques. Exotic non-uniform polarization arrangements reminiscent of skyrmions have been stabilized [40] and incommensurate phases, in which the polar distortions do not match the crystallographic periodicity, identified [41]. In the latter context, a general understanding of why most structural distortions are commensurate is still lacking, and a detailed knowledge of the crystallography of representative incommensurate phases would be invaluable. The excitement in recent decades in the area of multiferroic materials and phenomena has been fuelled by intricate details of crystal structure revealing novel mechanisms for ferroelectricity, such as the improper geometric behavior of $YMnO_3$ [42] or the polar magnetic ordering in $Cr_2BeO_4$ [43].

While many ferroelectrics in various crystal classes are now known, identifying new ones remains an active research area. Here, crystallographic databases provide invaluable input to modern high-throughput searches and machine-learning studies. And finally, while ferroelectrics must be electrical insulators so that an external electric field has a chance to switch the polarization, there is considerable current interest in polar metals, sometimes loosely referred to as ferroelectric metals [44], because of their potentially exotic transport and superconductivity behaviors [45].

At its 100th birthday, the field of ferroelectrics is still thriving, with new phenomena and materials continuously being revealed and remaining to be discovered. Doubtless, it will keep us busy for the next 100 years.

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EDITORIAL

# A new home for studies of crystal growth and characterisation




Alexander J. Blake, Marc de Boissieu, Ashwini Nangia, Elena Boldyreva, Andrzej Katrusiak, Tatyana B. Bekker, Karah E. Knope

Crystal growth is fundamental to much of crystallography, but in contrast to articles describing the crystallization of biological macromolecules, which are catered for by *Acta Crystallographica Sections D* and *F*, those dealing with the growth of crystals of nonbiological 'small' molecules or those of extended organic, inorganic or hybrid materials have lacked equally obvious routes to publication in IUCr journals. It is therefore timely to emphasize that *Acta Crystallographica Section B* offers such a route, by devoting a segment of the journal to relevant submissions. The ambition is to attract high-quality articles on crystal growth that are of broad interest and high impact, and there will be no arbitrary limit on the number of such articles.

Studies of crystal growth submitted to *Acta Crystallographica Section B* should align with the general scope of the journal, the subtitle of which is 'Structural Science, Crystal Engineering and Materials'. *Structural science* studies typically begin with the preparation of a crystal or other sample suitable for a diffraction experiment, but the importance of crystal growth in this context is far greater than simply producing a well diffracting crystal. Significant efforts are devoted to studying the mechanisms of crystal growth, as well as the melting, sublimation and dissolution behaviour of crystal forms. Moreover, control of crystal growth in aspects of both morphology (shape and size) and crystal polymorphism, based on the interplay of thermodynamics and kinetics, is fundamental to understanding the factors that determine crystal structure at the atomic, molecular, and macroscopic levels, and their impact on physical and chemical properties. *Crystal engineering* deals with the design, synthesis and growth of solid-state molecular assemblies with the aim of producing materials with defined desirable properties, since the bulk properties of many molecular materials are determined by the solid-state arrangement of the molecules. The method is based on a comprehensive understanding of intermolecular interactions and how they can be utilized to assemble extended crystal structures. A very wide range of intermolecular interactions, including hydrogen bonding, halogen bonding, π–π interactions and van der Waals interactions have been exploited for crystal growth in crystal engineering. The range of materials and properties (*e.g.* hydrogen storage, catalysis, gas separation, pharmaceuticals, optoelectronic materials) which have been designed is so extensive that even a much longer list would still not be representative. *Materials* are often composed of crystals, either as individual large single crystals with sizes in the range of centimetres and even metres, or with polygrain structures. Even at the other extreme of the size range, nanocrystals also need to be grown prior to being studied. Growing single or polygrain crystals, mastering their defect concentration (either to avoid defects or to engineer defects for given physical properties) is at the heart of material science. The understanding of the growth process, its modelling,

the detailed characterization of the atomic structure of the obtained crystals with defects, stoichiometry *etc.*, are key prerequisites to achieving an understanding of structure–property relationships.

Crystallization applies to most of condensed matter, including the naturally-occurring minerals, metals in almost infinitely various forms, organic compounds and many others. Recently we have witnessed enormous progress in crystallization methods, such as the special environments capable of reproducing in the laboratory the thermodynamic conditions of interstellar space, through hydrothermal baths, to the centre of Earth, extremely slow annealing in vacuum and ultrafast nucleation under terapascal (TPa) shocks, the size effects remarkably evident in nanotechnology, refined co- crystallizations, as well as the advanced methods of investigating the most subtle structural effects by super-intense microbeams combined with highly sensitive detectors. All this on one hand provides new information for better understanding the science and art of crystallization, and on the other hand opens new routes for obtaining new unprecedented forms of matter that can foster progress in scientific and technological development.

Some studies of crystal growth from more theoretical viewpoints have appeared in *Acta Crystallographica Section A* or *Journal of Applied Crystallography*, and we expect this to continue. However, as there are likely to be borderline cases, potential authors are encouraged to consult the Notes for Authors or contact a Main or Section Editor to discuss the most relevant journal for their submission.

The products of the crystal growth procedures, which may be single crystals, microcrystals, powders, nanocrystals, defect structures, incommensurate structures, *etc*., must be characterized by direct structural analysis based on diffraction or atomic scale imaging techniques. An article which describes a technique for growing crystals of a material but lacks a distinct crystallographic component or a complete and convincing structural characterization is unlikely to be suitable for *Acta Crystallographica Section B*. Other existing journal criteria also apply to crystal growth papers: these include relevance to the scope of the journal, clear and complete descriptions of experimental procedures, evidence that demonstrates that the method described is reproducible and whether the resulting sample is homogeneous or otherwise.

To emphasize that *Acta Crystallographica Section B* actively welcomes such submissions, the Notes for Authors contains a completely new section (§3) on crystal growth papers which present some examples of possible research topics. Our interest in publishing such papers is further signalled by the recent appointment of two Co-editors, Tatyana Bekker (Institute of Geology and Mineralogy, Siberian Branch of Russian Academy of Science, Novosibirsk, Russian Federation) and Karah Knope (Department of Chemistry, Georgetown University, Washington, DC, USA), who are experts in crystal growth. We are commissioning feature articles which will explain further the types of papers the journal is seeking to attract. Examples of crystallization under unusual conditions (high-pressure, cryogenic, levitation, confined medium, template-assisted, electric and magnetic field, laser irradiation, *etc*.) are welcome. A special collection of recent articles on crystal growth will be collated, followed later by a special issue comprising new papers.

These initiatives and developments demonstrate that *Acta Crystallographica Section B* is now a natural home for publications related to studies of crystal growth, and we invite the relevant communities to submit their work to the journal.

This article was originally published in *Acta Cryst*. (2021). B**77**, 456–457.

COMMENTARY

# Approximate symmetry in the third reported structure of a metal complex of L-DOPA

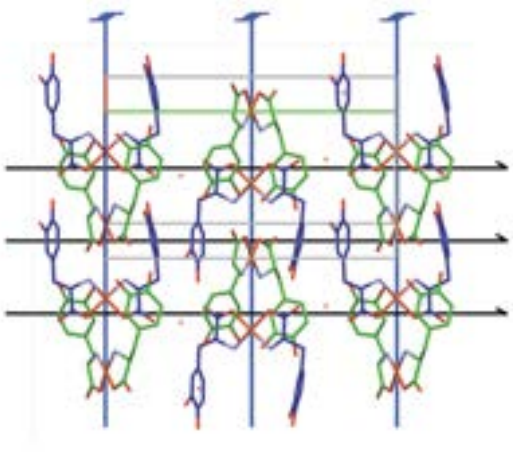


Figure 1. Projection along *a** of part of a layer of molecules having centroids in the range $0 < x < 1$. The projection of the *a* axis is visible because β = 104.7°. The crystallographic $2_1$ axes are shown in black and the approximate $4_2$ axes are shown in blue.

Carolyn Pratt Brock

The 2021 article '*cis*-Bis(L-DOPA-$^2$*N*,*O*)copper(II) monohydrate: synthesis, crystal structure, and approaches to the analysis of pseudosymmetry' by O'Brien *et al.* (2021) is notable for two reasons. First, it reports only the third crystal structure of a metal complex containing the medicinally important ligand L-DOPA (3,4-dihydroxy-L-phenylalanine). Second, the article includes a tutorial on finding approximate symmetry relationships between molecules that are chemically equivalent but crystallographically independent. Identification of approximate symmetry in molecular crystals is not yet a solved problem; rather, it is an area of considerable recent activity. Rekis (2020) proposed a method for finding approximate inversion centers; Brock & Taylor (2020) described software for finding approximate translations; Brock (2020) identified layers that have higher approximate symmetry than does the whole crystal; Baggio (2019, 2020) outlined a general method that can be used to find approximate symmetry of all types and applied it to a large number of $Z' = 4$ structures.

L-DOPA is a major drug that has been used for more than 50 years to treat the motor symptoms of Parkinson's disease; global sales of its various forms are measured in the billions of USD. It is then very surprising to find that the Cambridge Structural Database (CSD; Groom *et al.*, 2016) contains only two structures (FETTON and FETVEF; Suzuki *et al*., 1998) in which L-DOPA is coordinated to a metal, and two more (XOYXUH and XOYXUH01; Shemchuk *et al*., 2019) in which it is part of an ionic cocrystal (with LiCl).

There are numerous structures in the CSD of metal complexes containing the naturally occurring amino acids tyrosine (Tyr; 4-hydroxy-l-phenylalanine) and phenylalanine (Phe) that are the biological precursors of L-DOPA. In almost all of those structures the amino acid coordinates through both its amino and carboxylate groups. The CSD version of May 2021 includes 61 different $R \leq 0.075$ structures of metal complexes of Tyr and 83 of Phe; there are, however, only two structures of any precision of L-DOPA complexes. Given its medical significance and market value it seems certain that many attempts have

been made to grow diffraction-quality crystals of metal complexes of L-DOPA, but if so, then most of those attempts failed. It would seem that the addition of the second hydroxy substituent on the phenyl ring must be determining, but why?

O'Brien *et al.* note that they had difficulty finding a crystal that diffracted well. Shemchuk *et al.* (2019) mention the low quality of the data for XOYXUH; their structure of the polymorph XOYXUH01 was determined from powder data. The *R* factors for FETTON and FETVEF (0.073 and 0.079, respectively; Suzuki *et al.*, 1998) are surprisingly high. The small number of L-DOPA structures with metals and the problems with data quality suggest that some feature of L-DOPA interferes with crystal packing. Looking at the hydrogen-bonding tendencies of *vicinal* hydroxy groups located on phenyl rings might be a way to approach the problem.

The reported structure is particularly unusual in having two amino acid ligands. Of the 61 Tyr complexes in the CSD, only 18 have two Tyr ligands; of the 83 Phe complexes, only 9 have two Phe ligands. In many of those structures, especially those containing $Cu^{2+}$, the free O atom of a carboxylate group from a neighboring molecule completes the fivefold coordination sphere consistent with a $d^9$ ion capable of exhibiting a Jahn–Teller distortion. Sometimes, two such O atoms complete a sixfold coordination sphere. In the structure reported by O'Brien *et al.*, it is the 3-hydroxy O atom on the phenyl ring that fills this role, although one of the independent Cu—O distances is considerably shorter than the other (2.74 *versus* 2.97 Å). In none of the 18 $M^{n+}(\mathrm{Tyr})_2L$ complexes is the phenyl-ring OH group coordinated in that way.

The other notable feature of this structure is its approximate symmetry. There are hydrogen-bonded columns of molecules along *c* that have easily recognizable, although quite distorted, $4_2$ axes. O'Brien *et al.* explain how to find and quantify this relationship using the *MATCH* routine in *Crystals for Windows* (Betteridge *et al*., 2003) along with software they developed locally. The *FIT* routine in *PLATON* (Spek, 2020) gives essentially the same results for the rotation angle, the rotation axis, and the r.m.s. deviation (rmsd) for the best fit of the two molecules. When using *PLATON* (and perhaps when using *MATCH*) it was necessary to remove the 3-OH substituents on the phenyl rings so that the program recognized the two complexes as independent rather than bonded, with each of them having approximate twofold symmetry.

Approximate symmetry relating independent molecules is often associated with approximate symmetry that is periodic in at least two dimensions (Brock, 2020), but O'Brien *et al*. point out that the approximate $4_2$ axis does not lead to any supergroup description. In a three-dimensional supergroup including a $4_2$ axis the crystallographic $2_1$ axis along *b* would have to be accompanied by an at least approximate $2_1$ axis along *a*, but along *a* there is no symmetry of any kind other than translation (Fig. 1). Layers having approximate symmetry that is periodic in two dimensions are also impossible because layer groups cannot include *n*-fold rotation or screw axes, $n > 2$, that lie within the layer or any screw axis perpendicular to the layer. Any such axis within the layer could relate molecules through which it passes, but the axis would have to be local because if periodic it would move adjacent unit cells out of the layer.

The article concludes with a tribute to the first author, Professor Paul O'Brien of the University of Manchester, who passed away in 2018 while the manuscript was being formulated but before it could be completed. Since the delay was probably the result of the difficulty in finding a crystal that gave an acceptable diffraction pattern, the authors are to be applauded for their persistence. The finished article is a fitting tribute to Paul O'Brien.

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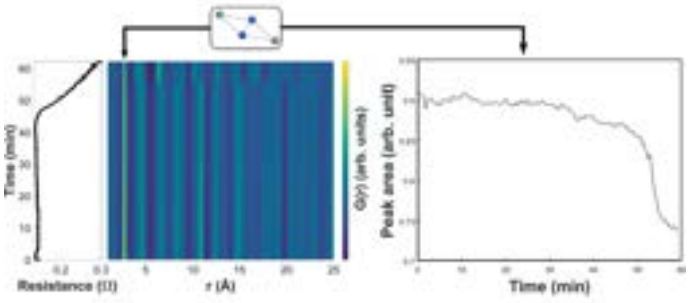
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This article was originally published in *Acta Cryst*. (2021). C**77**, 441–442.

EDITORIAL

# Operando structural science of functional materials




C. Richard A. Catlow

Classical structural science examined samples using diffraction and/or spectroscopic techniques under ambient conditions. Studies as a function of temperature have, of course, become routine and high-pressure experiments also have made a major contribution to structural science. In recent years, however, the availability of more sophisticated sample environments has enabled a rapid growth in the use of '*in situ and operando*' techniques, in which a functional material, such as a catalyst, is probed under conditions which resemble as closely as possible those used under the real operating environment, with measurements often being made with time resolution. The field is growing rapidly in sophistication and poses exciting challenges to, and opportunities for, the structural science of materials.

A terminological confusion can arise from the use of both '*in situ*' and '*operando*', with the latter denoting an experiment where the structural measurements are made together with measurements relating to its functional performance, which, for example, for a catalytic material would be the reaction product yield and distribution. A good illustration of an *operando* structural study was reported recently in **IUCrJ** by Rabøl Jørgensen *et al*. (2020), who investigated the thermoelectric material $Zn_4Sb_3$ using a setup which permitted simultaneous measurements of X-ray diffraction data and electrical resistance on samples subject to an electric current. From the analysis of the data, they were able to infer that zinc ions are mobile and also to demonstrate sample degradation at higher current densities. Their work paves the way for further *in operando* studies of thermoelectrics – materials of growing importance in energy technologies – and, as the authors comment, of solid-state battery and piezo- and ferroelectric materials.

The most extensive applications of in situ/operando techniques are probably those in catalytic science, using synchrotron-based X-ray diffraction and X-ray absorption spectroscopy. Here they have had a huge impact in recent years and good reviews of earlier work in this area are available from Beale *et al*. (2010), Newton & van Beek (2010) and Bentrup (2010). Several recent examples can be found in the *Faraday Discussions* held this year on *Reaction Mechanisms in Catalysis*, including the study of Bugaev *et al*. (2021) which examined the industrially relevant reaction of ethylene hydrogenation using palladium catalysts, monitored by both XRD and XAS, illustrating the well established and increasing trend to apply multiple measurement techniques during *in situ* experiments. The data obtained allow the structural evolution of the working catalyst to be monitored as well as the transitions between metallic, hydride and carbide phases of palladium as the catalytic reaction progresses. Another excellent illustration of the current state of the art is provided by the recent study of van Ravenhorst *et al*. (2021) on the topical Fisher–Tropsch $Co/TiO_2$ catalyst (which catalyses the synthesis of hydrocarbons from syngas, i.e. $CO/H_2$ feed). They again combine synchrotron-based XAS with both synchrotron- and laboratory-based XRD to

study the evolution of the catalyst on stream for 48 h. They are able to follow the formation of cobalt carbide as the reaction progresses, although the product distribution is largely unaffected. Interestingly, the formation of carbide is detected in XAS before XRD, as the former is more sensitive to short-range order. The paper nicely illustrates how detailed structural information on the evolution of complex catalytic systems can be obtained by this type of multi-technique *operando* study. A further recent example showing technical innovation is provided by the work of Matras *et al*. (2021), who used advanced tomographic imaging techniques in an *operando* study of a Ni–Pd/$CeO_2$–$ZrO_2$/$Al_2O_3$ catalyst during the partial oxidation of methane. The study gives detailed information both on the evolution of the metal and oxide species during the catalytic reaction and on the role of the heterogeneity in the catalyst particles.

A significant recent development is the ability to undertake experiments within a catalytic reactor, which is nicely illustrated by the work of Nieuwelink *et al.* (2021), again from the recent *Faraday Discussions.* The study followed hydrogenation reactions in a microreactor and was able to probe single particles during the reaction. 'In reactor' experiments can also give spatially resolved information as in the recent work of Decarolis *et al.* (2021) who investigated the widely studied selective catalytic oxidation of ammonia using a catalyst comprising palladium supported on alumina. The study shows that the nature of the catalytic species changes along the reactor and that these changes can be correlated with product distribution. This type of spatial analysis will grow in importance as the ability to undertake spatially resolved experiments develops.

*In operando* methodologies are not confined to X-ray techniques; they have been exploited, albeit less widely, using neutron scattering. Good examples in catalytic science include the work using neutron spectroscopy which was reported by Parker (2011), who clarified the role of surface hydroxyls in CO oxidation over a model palladium catalyst; the study also illustrates the power of neutron spectroscopic techniques to probe catalytic systems, which would be difficult to study by photon-based spectroscopies. A further illustration is provided by Youngs *et al.* (2013) who employed neutron total scattering methods to probe time-resolved catalytic chemistry in a Pt/$SiO_2$ catalyzed benzene hydrogenation reaction.

Finally, we should note that although the extensive range of applications in catalysis have had notable impact, *operando* techniques can be applied to many other classes of functional material, such as energy materials, highlighted earlier. Structural studies of functional materials will increasingly be made under '*operando*' conditions. **IUCrJ** would welcome submissions in this exciting and growing field.

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This article was originally published in *IUCrJ* (2021). **8**, 703–704.

COMMENTARY

# Lipid transport across the mycobacterial cell envelope

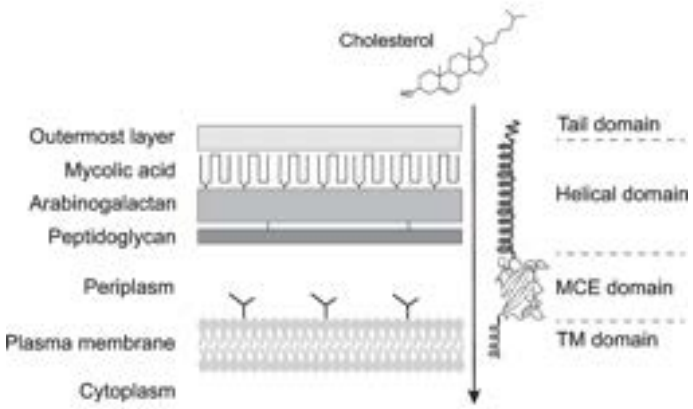



Figure 1. Schematic representation of the results reported by Asthana *et al.* (2021) within the context of cholesterol import through the *M. tuberculosis* cell envelope. Mce4A is shown to be monomeric in solution, likely forming a hetero-hexameric arrangement with other Mce4 proteins to form a tunnel for lipid transport. A schematic model of the *M. tuberculosis* cell envelope is also shown for comparison, adapted from Chiaradia *et al.* (2017). The schematics are not drawn to scale.

Ghader Bashiri

Tuberculosis is a devastating disease that has afflicted humans since antiquity. Known as Phthisis (Greek), the ‘white plague’ or consumption, tuberculosis appears as a common theme in art, music and literature, and has shaped many elements of human social history (Daniel, 2006). Even today, one-quarter of the world’s population is estimated to carry latent infections by *Mycobacterium tuberculosis*, the bacterium that causes tuberculosis (Getahun *et al*., 2015). So why is this disease so recalcitrant to treatment? This is, in part, due to the distinctive cell envelope of *M. tuberculosis*, which provides a physical barrier against antibiotics (Batt *et al*., 2020). Furthermore, this envelope also helps *M. tuberculosis* survive attacks by the host immune system (Batt *et al.*, 2020), allowing the bacterium to persist in a non-replicating (‘dormant’) state in the host cells (Gengenbacher & Kaufmann, 2012). A key feature of *M. tuberculosis* is its ability to acquire and metabolize lipids, notably cholesterol and fatty acids, from its human host (Wilburn *et al.*, 2018). These lipids provide the bacterium with the essential carbon and energy sources to maintain viability over many years (Warner, 2014). Understanding how these lipids are transported into *M. tuberculosis* may expose vulnerabilities that could then be exploited to develop new therapeutic agents against tuberculosis.

Despite the significance of lipid metabolism in the survival and pathogenesis of *M. tuberculosis*, it is not clear how lipids are transported into the cell, with no structural or mechanistic details on mycobacterial lipid transporters being available. The genome sequence of *M. tuberculosis*, and subsequent studies, have identified four homologous mammalian-cell-entry (Mce) multiprotein complexes that are proposed to play crucial roles in translocating various lipid molecules across the cell envelope (Cole *et al.*, 1998; Casali & Riley, 2007). These membrane-bound assemblies, however, have so far defied structural analysis. In the September 2021 issue of **IUCrJ**, Asthana *et al.* (2021) now provide the first insights into the structure and potential assembly of the Mce1 and Mce4 proteins in *M. tuberculosis*.

Mce proteins play crucial roles in *M. tuberculosis* pathogenesis through reimporting fatty acid and mycolic acid (Mce1), and importing cholesterol from the host cells (Mce4) (Pandey & Sassetti, 2008; Nazarova *et al*., 2017). Each mce operon encodes proteins with various roles in the formation of their

respective Mce complexes, including six Mce proteins (MceA, MceB, MceC, MceD, MceE and MceF) that act as substrate-binding proteins (SBPs) (Casali & Riley, 2007). The homologous SBPs from *E. coli* (Ekiert *et al.*, 2017; Isom *et al*., 2020; Liu *et al*., 2020; Coudray *et al*., 2020) and *Acinetobacter baumannii* (Kamischke *et al*., 2019; Mann *et al*., 2020) have been shown to form hexameric structures, leading to their central role in lipid transport through either a tunnel- or ferry-based mechanism.

The results presented by Asthana *et al.* (2021) reveal several advances in our understanding of the Mce proteins in *M. tuberculosis*. They used sequence analysis and secondary-structure prediction to show that all SBPs of Mce1–4 display a conserved four-domain architecture. This arrangement comprises an N-terminal transmembrane (TM) domain, the MCE domain, a helical domain and a tail domain of variable size. They also showed that all these individual domains, except for the MCE domain, require detergents for solubility and stability. Interestingly, the full-length and individual domains of *M. tuberculosis* Mce1A and Mce4A are predominantly present as monomers in solution. This was further confirmed by the crystal structure of the single MCE domain present in Mce4A ($Mce4A_{39–140}$), indicating that this domain could not form homo-hexamers due to steric clashes between monomers. This is a notable difference from the previously reported hexameric SBPs observed in E. coli (Ekiert et al., 2017; Isom et al., 2020; Liu et al., 2020; Coudray et al., 2020) and A. baumannii (Kamischke et al., 2019; Mann et al., 2020). Finally, using small-angle X-ray scattering (SAXS) experiments and structure-based modelling, they showed that the helical domains of Mce1A and Mce4A interact with the detergent micelles, implying that they either interact with the membrane or the lipid substrates.

These results have consequently led to a proposed model on the likely assembly of the Mce proteins in *M. tuberculosis* (Asthana *et al.*, 2021). Based on this model (Fig. 1), the six MCE domains of MceA–F SBPs may interact with each other to form hetero-hexamers, with the helical domains of each polypeptide coming together to form a long and hydrophobic channel for lipid transport. This structure would be held in between the plasma membrane and the cell surface *via* interactions with the TM domains and the tail domains, respectively. This model resembles the tunnel-based mechanism described in *Ec*-Pqi (Ekiert *et al.*, 2017), providing the first experimental model towards the Mce-mediated lipid transport in *M. tuberculosis*.

The proposed model by Asthana *et al*. (2021) establishes a unique foundation for future studies of the Mce multiprotein complexes, elucidating structural and mechanistic details of lipid transport in *M. tuberculosis*. Such endeavours may also facilitate the development of specific compounds to target cholesterol import as a therapeutic intervention, particularly restricting *M. tuberculosis* growth and survival during persistence.

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This article was originally published in *IUCrJ* (2021). **8**, 711–712.

EDITORIAL

# An open-access future for Journal of Synchrotron Radiation – Editorial from the Main Editors and IUCr Journals Editor-in-Chief




Kristina Kvashnina, Yoshiyuki Amemiya, Dibyendu Bhattacharyya, Ingolf Lindau, Andrew J. Allen

The entire *Journal of Synchrotron Radiation* (*JSR*) editorial team would like to take this opportunity to inform all our readers, authors and supporters about the coming transition to open access. All papers submitted to *JSR* after 1 October 2021 will be for open-access publication. By taking this step, *JSR* is supporting a journey towards open science in general.

*JSR* was founded in 1994 (Hasnain, Helliwell & Kamitsubo, 1994) with the aim of providing comprehensive coverage of the entire field of synchrotron radiation. Almost immediately its coverage also started to include free-electron laser research (Doniach, 1996) including instrumentation, theory, computing and scientific applications in areas such as biology, nanoscience and materials science. Just in the last year, authors from 35 different countries published in the journal, the top five countries represented being the USA, Japan, Germany, China and France. Throughout the past 26 plus years *JSR* has been an up-to-date information resource for scientists and engineers in the field of synchrotron radiation.

Now, the *JSR* Main Editors, with the International Union of Crystallography (IUCr) Journals Editor-in-Chief and the IUCr Editorial Office, supported by the IUCr Executive Committee, believe that switching to open access will benefit research in this area by disseminating it more easily and rapidly to the global synchrotron and free-electron laser science communities. The clear goal of this initiative is to induce the smoothest and most research-oriented transformation possible of *JSR* from a behind-paywall subscription-based publishing model to an open-access-based publishing model. All of us increasingly work under conditions in which open access supports researchers in every aspect of their workflow. New detailed instructions on general policies for submission, possible open-access discounts, and other guidelines and templates are discussed at https://journals.iucr.org/s/services/openaccess.html. However, we wish to emphasize that all open-access articles will undergo initial editor assessment and the same rigorous peer review process as at present. The management of the peer review process will continue to focus on the high standards and rapid publication expected for IUCr journals.

At this time, we wish to express our enormous appreciation of *JSR*'s supporting (facility) institutions. We also hope that more supporting institutions will join with *JSR* as we move forward from here. *JSR* supporting institutions are entitled to a certain number of open-access article processing charge (APC) vouchers per year for papers reporting work carried out at their facilities. Alternatively, if the contact

author's home institution is included in a transformative or read-and-publish arrangement with the IUCr's publication partner, Wiley, those authors will be able to publish open-access research or review articles in *JSR* with no direct (APC) charge. Currently, such arrangements exist in Austria, Finland, Germany, Hungary, Ireland, Italy, Liechtenstein, the Netherlands, Norway, Spain, Sweden, Switzerland and the UK. Contact authors with connections to IUCr (Associates, members of national affiliates, World Directory of Crystallography, *etc*.) will receive modest APC discounts. Meanwhile, discounts (50%) for authors from lower middle income countries and waivers (100%) from low income countries will be issued. However, please note that a major difference from present arrangements is that all submitting authors will need to apply for such discounts and waivers at the time of submission and payments will be handled via Wiley authors services (for more details see https://journals.iucr.org/s/services/openaccess.html).

As the transition to open access proceeds, the *JSR* Editorial Board welcomes feedback from the *JSR* research community, especially from authors, on the new open-access procedures, and is keen to know what is working well and what needs some adjustment.

The change to open access is made with every faith in the future. The Editors fully believe that the publishing of scientific research with global open access providing worldwide visibility without barriers demonstrably leads to more downloads, citations and more impact for authors. The increased citation of open-access articles published in *JSR* since 2018 is shown graphically in the figure.

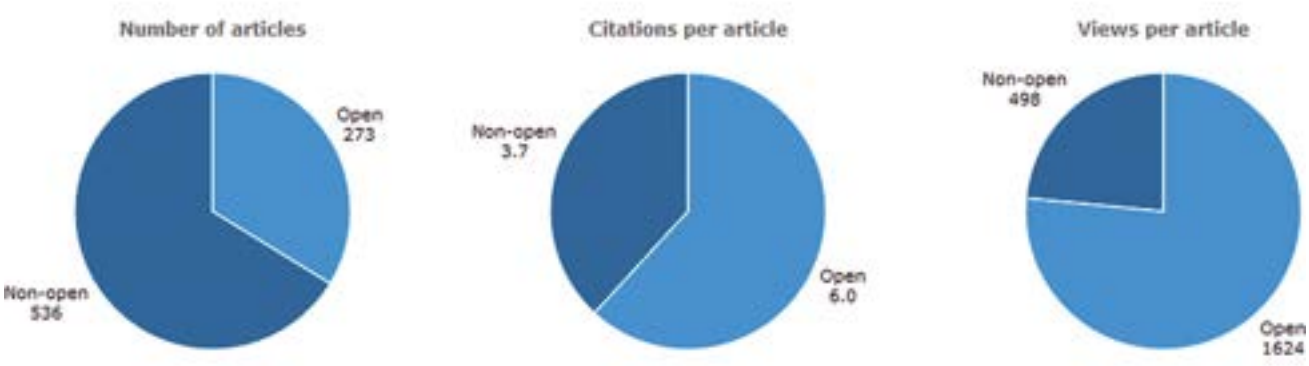



Figure 1. Graphic showing the increased citation of open-access articles published in JSR since 2018.

The *JSR* Editors embrace both the idea and concept of making research freely available to all researchers, and are committed to coordinate and establish best principles to facilitate a smooth transition from subscription to open access. The Editors wish all the best to all *JSR* authors for the future and look forward to seeing your forthcoming high-quality open-access research submissions to *JSR*.

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This article was originally published in *J. Synchrotron Rad.* (2021). **28**, 1273–1274.

OUTREACH

# Eighth Polish Crystal Growing Competition for Schools 2021



A selection of winning and distinguished entries to the Eighth Polish Crystal Growing Competition for Schools 2021. Photo credit: Dr Marcin Łaciak, Faculty of Science and Technology, University of Silesia.

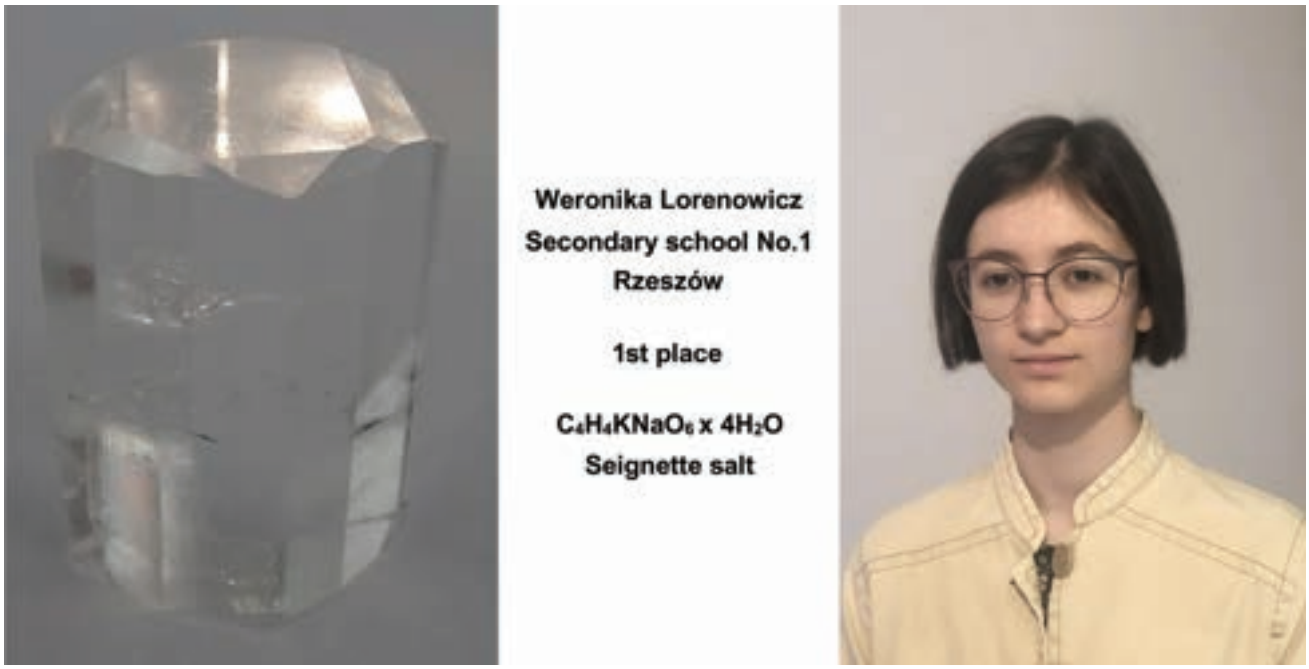
Ewa Talik

The competition was organized at the Institute of Physics, University of Silesia, Poland, at the invitation of the IUCr in 2014 and IUCr Commission on Crystal Growth member Dr Hanna Dabkowska. For the 2021 competition, 620 people were registered, and 289 sent 456 crystals from 72 towns. The jury selected 3 winners and more than 20 works were distinguished. The awards ceremony took place on 17 June 2021 during the seminar "Crystals: order, beauty and usefulness" at the Institute of Physics, University of Silesia, Chorzów.




The poster advertising the scientific seminar and award ceremony.

The Director of the Institute of Physics, University of Silesia, Professor Sebastian Pawlus, and the Dean of the Faculty of Mathematics, Physics and Chemistry, Professor Danuta Stróż, opened the Seminar connected with the award ceremony. The patronage of the competition included the Polish Society for Crystal Growth and the Committee of Crystallography, Academy of Sciences (detailed in the poster above). The organizing committee included Professor Ewa Talik, Dr Magdalena Szubka, Dr Monika Oboz, Dr Adam Guzik, Dr Aneta Szczygielska-Łaciak and Dr Marcin Łaciak.






The three winners of the competition. Photo credit: Dr Marcin Łaciak, Faculty of Science and Technology, University of Silesia.

| | |
|---|---|
| Students awarded distinctions | |
| Town | |
| Compound | |
| Magdalena Ruszkowska | |
| Poznań | |
| $KCr(SO_4)_2{\cdot}x12H_2O$ | |
| Kinga Abrachamczyk | |
| Rybnik | |
| $KAl(SO_4)_2{\cdot}x12H_2O$; $CuSO_4{\cdot}x5H_2O$ | |
| Gabriela Drzewiec | |
| Siedlce | |
| $K_3[Fe(CN)_6]$ | |

Patrycja Mleczek

Klecza Dolna

$(NH_4)_2Fe(SO_4)_2$

Dominik Janczyk

Czerwionka-Leszczyny

$KAl(SO_4)_2{\cdot}x12H_2O$

Karolina Rozpędek

Katowice

$KCr(SO_4)_2{\cdot}x12H_2O$; $CuSO_4{\cdot}x5H_2O$

Maksymilian Kozik

Gliwice

$KAl(SO_4)_2{\cdot}x12H_2O$

Beata Niedbała

Nowa Sarzyna

$KAl(SO_4)_2{\cdot}x12H_2O$

Artur Rolnik

Ruda Śląska

$C_{12}H_{22}O_{11}$

Weronika Lorenowicz

Rzeszów

$(CH_3COO)_2Cu{\cdot}xH_2O$

Julia Białek

Chorzów

$K(Cr,Al)(SO_4)_2{\cdot}x12H_2O$; $CuSO_4{\cdot}x5H_2O$

Wiktoria Golian

Lublin

$KCr(SO_4)_2{\cdot}x12H_2O$

Anastasiia Afonina

Ostrowiec Świętokrzyski

$KCr(SO_4)_2{\cdot}x12H_2O$

Barbara Stefaniuk

Siedlce

$Mg(SO)_4{\cdot}xH_2O$

Bianka Wyrobek

Paniówki

$KAl(SO_4)_2{\cdot}x12H_2O$

Natalia Bartkowiak

Poznań

$C_6H_8O_7$

Alicja Mizera

Pniewy

$CuSO_4{\cdot}x5H_2O$

Wiktoria Marosik

Kostrzyn nad Odrą

$CuSO_4{\cdot}x5H_2O$

Aleksandra Babiuch

Przeginia

$CuSO_4{\cdot}x5H_2O$

Szymon Flis

Tychy

$KAl(SO_4)_2{\cdot}x12H_2O$

Marcel Tomaszek

Stargard

$CuSO_4{\cdot}x5H_2O$

Jan Chomiuk

Rybnik

$CuSO_4{\cdot}x5H_2O$

Franciszek Kubuj

Otwock

$CuSO_4{\cdot}x5H_2O$

Dominika Kucharska

Grójec

$CuSO_4{\cdot}x5H_2O$

For full details, please see here.




The prizes awarded in 2021.

Three lectures devoted to the subject of crystals were delivered during the seminar:

“Crystallography” – Professor Maria Gdaniec (Department of Chemistry, Adam Mickiewicz University, Poznań, Poland)

“Luminescent crystals” – Dr hab. Dobrosława Kasprowicz (Poznan Faculty of Technical Physics Institute of Materials Research and Quantum Engineering Division of Optical Spectroscopy, University of Technology, Poznań, Poland)

“Looking inside the materials” – Dr Maciej Zubko (Faculty of Exact and Technical Sciences Institute of Materials Science, University of Silesia, Chorzów, Poland).

**The organizing committee expresses its thanks to Professor Juan Manuel García-Ruiz from the University of Granada, Spain, for sharing the video “The Mystery of the Giant Crystals” presented at the seminar.**

Before the COVID-19 pandemic, winners participated in a scientific visit to CERN, Geneva, Switzerland, and the Solaris Synchrotron Radiation Centre, Kraków, Poland. For a full list of winners since 2014, please see here.

FEATURE ARTICLE

# Growing a thriving international community for small-angle scattering through collaboration

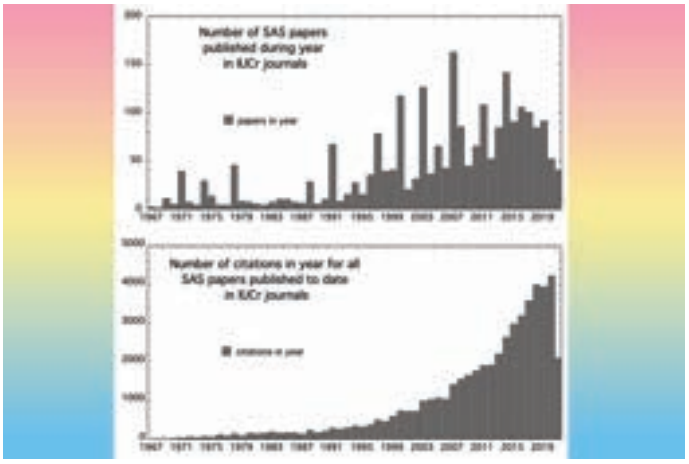



Fig. 1. Small-angle scattering papers published in IUCr journals, and their associated citations (in all journals) from 1967 to mid-2021.

Jill Trewhella

Small-angle scattering emerged as a tool for studying noncrystalline structures from early observations around 1930 that there was a relationship between the extent of the scattering and the size of the scattering object. André Guinier, a leading figure in the development of the field, noted in his summary findings from the first Conference on Small Angle Scattering in 1958 that the technique would be of value to study 'submicroscopical inhomogeneities' and further provided a means of 'observation [that had] in the past restricted the field of application of the X-ray method.' In 1965 the first of what became a highly successful series of Small-Angle Scattering (SAS) meetings held approximately every three years took place in Syracuse, NY, USA, and many of these ongoing meetings published their proceedings and highlights in the International Union of Crystallography (IUCr) *Journal of Applied Crystallography*. Since the early 2000s, the relationship between the international SAS community represented at the triennial SAS meetings and the IUCr has been strengthened and deepened through formal cooperation and collaboration in a number of mutually beneficial activities that have supported the growth and health of the field and the IUCr.

## 1. A brief history of an emerging small-angle scattering community

The first published observations of small-angle X-ray scattering (SAXS) were in the *Indian Journal of Physics* by Panchapakesa Krishnamurti, whose series of papers from 1928 to 1930 culminated in a publication detailing the relationship between particle size and molecular weights and the extent of SAS (Krishnamurti, 1930). In 1939, André Guinier published his landmark paper (Guinier, 1939) describing a simple approximate mathematical relationship between the rate of decrease in intensity of the SAS signal at the smallest angles and the size of the scattering particle. This relationship, known as the Guinier approximation, could be readily exploited for studying a broad range of materials and resulted in a significant uptick in applications of SAS. Guinier thus became widely known as the 'father' of small-angle scattering.

The influential monograph *Small-Angle Scattering of X-rays* co-authored by Guinier with Gérard Fournet (Guinier & Fournet, 1955), translated from the original French into English, provides a comprehensive review of the state of the field by the mid-1950s. The monograph covers the enabling theoretical

frameworks, such as the Debye approximation (Debye, 1915) relating the total scattering from a particle to the scattering from volume elements within it, as well as advances facilitating laboratory measurements, such as the development of laboratory-based X-ray cameras with pin-hole or slit collimation, and further breakthroughs in data interpretation and analysis such as the Kratky plot (Kratky & Porod, 1949), used to assess flexibility in a scattering particle, and the Porod law (Porod, 1951) describing the high-angle scattering. The scattering of neutrons is also covered in the monograph, as researchers were beginning to consider how the distinctive properties of neutrons might be used to expand the opportunities for SAS. However, it would be a couple of decades before small-angle neutron scattering (SANS) would begin to grow, notably aided by the advocacy of Bernard Jacrot (Jacrot, 1976) and neutron scattering facilities at the Institut Laue–Langevin, Grenoble, France.

In 1958, the first conference dedicated to SAS was held in Kansas City, Missouri, USA (September 23–25), and the proceedings were published in the *Journal of Applied Physics* (*Small Angle X-ray Conference Proceedings*, Vol. 30, Issue 5, May 1959). Final summary remarks were contributed by André Guinier (Guinier, 1959), who notes 'In conclusion, it can be said that the progress in our understanding of small angle scattering, as well as in other methods of observation have in the past restricted the field of application of the X-ray method, but we are now able to make a better use of this method as in cases where it is still the best tool to study submicroscopical inhomogeneities.'

In many ways, the 1958 conference could be considered the zeroth meeting for what would become the very successful series of meetings that would bring together the international SAS community across all fields to advance and grow SAS applications. It was just seven years on, in 1965, that what is regarded as the first of this series was held in Syracuse, New York, USA. Since then, the international SAS community has come together to meet approximately every three years (see Table 1). These triennial meetings are a major event for the field, consistently attracting >400 attendees since the 2006 Kyoto meeting. They highlight SAS contributions to biology, chemistry and materials science, much as Guinier envisioned with the technique becoming broadly applicable for materials characterization.

Table 1
Small-angle scattering conferences and associated proceedings and special issues 1958–2020.

The Roman numerals indicate the recognized triennial meetings, with the 1987 and 1958 meetings and their proceedings listed for completeness.

| Sequence | Conference, location | No. attendees | Proceedings/special issues | No. papers, pages |
|---|---|---|---|---|
| XVII | SAS2018, Traverse City, MI, USA | 411 | – | – |
| XVI | SAS2015, Berlin, Germany | 424 | *J. Appl. Cryst.* **49**, *Small-angle scattering special issue*, December 2016, highlighting contributions at SAS2015, bringing together articles originally published between October and December 2016 | 12 papers |
| XV | SAS2012, Sydney, Australia | 426 | *J. Appl. Cryst.* **47**, *Small-angle scattering special issue*, February 2014, highlighting contributions at SAS2012, bringing together articles originally published between October 2013 and February 2014 | 25 papers |
| XIV | SAS2009, Oxford, UK | 500 | – | – |
| XIII | SAS2006, Kyoto, Japan | 600 | *J. Appl. Cryst.* **40**, Part s1, April 2007 | 139 papers, 705 pages |
| XII | SAS2002, Venice, Italy | 380 | *J. Appl. Cryst.* **36**, Part 3, Number 1, June 2003 | 69 papers, 319 pages |
| XI | SAS-99, Upton, NY, USA | 310 | *J. Appl. Cryst.* **33**, Part 3, Number 1, June 2000 | 69 papers, 319 pages |
| X | SAS-96, Campinas, Brazil | 210 | *J. Appl. Cryst.* **30**, Part 5, Number 2, October 1997 | 69 papers, 319 pages |
| IX | SAS-93, Saclay, Paris, France | 190 | *J. Phys. IV* **3**, C8, 3–526 (1993) | 109 papers, 523 pages |
| VIII | SAS-90, Leuven, Belgium | 150 | *J. Appl. Cryst.* **24**, Part 5, October 1991 | 69 papers, 461 pages |
| – | 1987, Argonne, Lemont, IL, USA | – | *J. Appl. Cryst.* **21**, Part 6, December 1988 | 43 papers, 302 pages |
| VII | SAS-87, Prague, Czechoslovakia | 120 | *Macromol. Symp.* **15**, 1–372 (1988) | 372 pages |
| VI | SAS-83, Hamburg, Germany | 100 | – | – |
| V | SAS-80, Berlin, Germany | 150 | – | – |
| IV | SAS-77, Gatlinburg, TN, USA | 140 | *J. Appl. Cryst.* **11**, Part 5, October 1978 | 71 papers and abstracts, 359 pages |
| III | SAS-73, Grenoble, France | 70 | *J. Appl. Cryst.* **7**, Part 2, April 1974 | 58 papers and abstracts, 144 pages |
| II | SAS-70, Graz, Austria | 65 | *J. Appl. Cryst.* **4**, 406–427 (1971) | 64 extended abstracts, 21 pages |
| I | SAS-65, Syracuse, NY, USA | 25 | *Small-Angle X-ray Scattering* (edited by Harry Brumberger), Gordon & Breach Science Publishers, New York (1967) | 32 papers, 509 pages |
| – | 1958 Kansas City, MO, USA | – | *J. Appl. Phys.* **30**(5), 601–674 (1959) | 74 pages |

## 2. Growing a vibrant international community through collaboration with the International Union of Crystallography

The early triennial SAS meetings were arranged on an ad hoc basis by successive host institutions whose representatives convinced the preceding conference delegates that they could do the job. In part because SAS at the time was viewed primarily in terms of being a sub-discipline within its application field, these meetings were organized independently from the IUCr. Even so, there was a strong IUCr connection that recognized SAS as a field of crystallography, with the publication of conference proceedings for many of the meetings in *Journal of Applied Crystallography* (Table 1). Further, IUCr journals have been a favoured destination for what are highly cited SAS studies (Fig. 1); the 2335 SAS papers published in IUCr journals since 1967 have generated 50 960 citations in 30 416 citing papers for an average citation per paper of 21.85.

In the early 2000s, Jan Skov Pedersen, John Barnes, Gernot Kostorz and other founding members of the IUCr Commission on Small-Angle Scattering (CSAS) saw value in greater collaboration between the IUCr and the triennial SAS meeting organizers. An agreement was negotiated wherein the international SAS community would adopt IUCr standards for speaker diversity and venue selection for the triennial SAS conference and the meeting would become an IUCr-sponsored meeting. That sponsorship would include support for students to attend and to enable participation from developing countries, as well as the establishment of the IUCr Guinier Prize of USD 5000, honouring the pioneering work of André Guinier. The Guinier Prize would be awarded for ‘lifetime achievement, a major breakthrough, or an outstanding contribution to the field of small angle scattering.’

As part of these negotiations, and in response to IUCr concerns to avoid scheduling clashes, the triennial SAS conference was moved out of the Congress years: hence the four-year break between the 2002 conference in Venice, Italy, and the 2006 conference in Kyoto, Japan (Table 1). Since then, successive CSAS Chairs have sought to solidify the connections between the IUCr and the triennial SAS meetings, in addition to supporting other independent SAS groups like canSAS (Collective Action for Nomadic Small Angle Scatterers; an ongoing activity to provide the small-angle scattering user community with shared tools and information http://www.cansas.org/) that have grown spontaneously from the SAS conference community.

## 3. A successful framework for sustaining meaningful collaboration

An important mechanism to strengthen and sustain the IUCr–international SAS community connection has been to include IUCr CSAS members in processes for future conference venue selection and the selection of the Guinier Prize recipients. To this end, a formal process was established for venue selection for future meetings based on proposals submitted at least two meetings prior. The proposals are reviewed by representatives of the current triennial SAS meeting organizing committee members and members of CSAS. Qualified proposals based on a set of criteria agreed by the review committee (*e.g.* ensuring geographical diversity, programme committee diversity, local organizing capacity, adequate conference facilities, affordable accommodation, accessible food outlets, budgets with cost estimates *etc.*) are presented to the conference participants for a vote. In this way, the participants in the meeting still choose their future venue, but with the assurance that the proposed hosts are fully prepared for the challenging job they will have and with a full six years to prepare.

As with venue selection, nominations for the Guinier Prize are considered jointly by members of CSAS and members of the organizing committee for the meeting where the prize will be presented. It is often the case that the meeting will cosponsor the prize by providing registration and travel support for the prize winner. The number of SAS luminaries who are Guinier Prize recipients (Table 2) is testimony to the success of the selection process, a credit to the field, and the prize constitutes a worthy recognition to the honouree.

**Table 2**
Guinier Prize recipients.

| | |
|---|---|
| 2018 | Dmitri Svergun (EMBL, Germany) |
| 2015 | Sow-Hsin Chen (MIT, 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) |

In addition to the above formal mechanisms for collaboration on specific tasks, there are many ways in which CSAS and the triennial meeting organizers interact and exchange ideas, for example through engagement in the international advisory committee, programme and local organizing committees for the triennial meetings, and of course strong participation in the meetings. At the triennial SAS meetings, there is generally an open meeting of CSAS where the activities of CSAS are reported and there is opportunity for input to CSAS and exchange of ideas on initiatives. As the tenures of CSAS members tend to span multiple meetings, CSAS engagement offers some historical context and memory for what has worked well. The meeting organizing and programme committees also have been a source of excellent recruits to the membership of CSAS, with proven records for community engagement and effort.

Significantly, in 2020 when issues arose with the planned SAS2021 meeting in Campinas, Brazil, that would conflict with the COVID-19-postponed 25th IUCr Congress in Prague, the Chair of CSAS requested and was granted approval from the IUCr Executive for an additional CSAS member, specifically a representative of the SAS2021 meeting organizing committee.

Through this mechanism, it was possible to work through the issues for both meetings with transparency and good will, which led to SAS2021 becoming SAS2022 with the 25th IUCr Congress moving to 2021 and the prospect of two successful meetings as the pandemic hopefully fades.

## 4. Examples of how the IUCr–SAS community collaboration advances science

The SAS conferences and associated open CSAS meetings were important venues for discussion and dissemination of information by CSAS members who were leading efforts to establish a universal SAXS intensity standard (Allen *et al.*, 2017). The International Organization for Standardization (ISO) first published the *SRM 3600 – Absolute Intensity Calibration Standard for Small-Angle X-ray Scattering* ISO best practice measurement standard in 2015, and it was revised in 2020 (see https://www-s.nist.gov/srmors/view_detail.cfm?srm=3600). This standard was drafted in ISO/TC 24/SC4/WG10 where Technical Committee (TC) 24 is ‘Particle characterization including sieving’, sub-committee (SC) 4 is ‘Particle size characterization’ and Working Group (WG) 10 is ‘Small angle X-ray scattering method’. The same working group is now focused on surface area measurement.

Another example of the effectiveness of the collaborative relationship between the IUCr CSAS and the triennial SAS meeting organizers is the effort to establish community endorsed standards for biomolecular SAS. Around the mid-1990s, biomolecular SAS evolved rapidly from a technique practiced predominantly by a select community of SAS experts to a tool for the broad structural biology community. From 1950 to 1995, biomolecular SAS accounted for 4% of the total publications using SAS, from 1996 to 2016 it was 12% and since 2017 it has been 18% (Web of Science Core Collection search on small-angle scattering and protein compared with small-angle scattering alone). This growth trajectory was the subject of considerable discussion at a meeting of CSAS in Kyoto at SAS2006, where several participants suggested that standards for the publication of biomolecular SAS data and models were

needed. The topic was predictably controversial as there were legitimate concerns that efforts to have standards could have unintended consequences and restrict publication in ways that were not appropriate. With the seeds of this effort having been planted in Kyoto, however, there were many follow up discussions at subsequent triennial SAS meetings and IUCr Congresses. These discussions were expanded with the establishment of a SAS validation task force (SASvtf) (Trewhella *et al.*, 2013) by the world-wide Protein Data Bank (wwPDB). Eleven years after the discussions in Kyoto, guidelines for publication of biomolecular SAS studies, including standards for data presentation and model validation, were presented at the 24th IUCr Congress in Hyderabad, India (Trewhella & Guss, 2017), and published in *Acta Crystallographica Section D* (Trewhella *et al.*, 2017). These guidelines are being adopted, as shown by the 11 964 downloads (as of 23 July 2021) since going online.

Among the recommendations of the SASvtf and the 2017 publication guidelines was that biomolecular SAS data should be deposited in a public archive. Subsequently, the wwPDB Integrative/Hybrid Methods validation task force (IHMvtf) (Sali *et al.*, 2015) recommended that, for the new generation of biomolecular structures that use multiple types of data and computational methods, all the data types with their metadata should be publicly available in data archives. Furthermore, the data formats should facilitate seamless exchange between archives of different data types to support data and model validation. In response to these recommendations, there was further development of a SAS extension of the IUCr core Crystallographic Information File (sasCIF) (Kachala *et al.*, 2016; Malfois & Svergun, 2000), which was then utilized by the SAS Biological Data Base (SASBDB) (Valentini *et al.*, 2015) in establishing a searchable curated repository of experimental SAS data deposited together with the relevant experimental conditions and sample details. Because the SASBDB uses sasCIF, there can be seamless exchange with the wwPDB, and SAS was the first technique beyond crystallography, NMR and electron microscopy to be formally included in the wwPDB efforts to establish the capacity to accept structures determined using integrative/hybrid methods (Burley *et al.*, 2017; Berman *et al.*, 2018, 2019).

## 5. Looking to the future

There are good reasons to continue to expand and deepen the connection between the IUCr and the international SAS community represented at the triennial meetings and at the IUCr Congresses.

The international SAS community continues to be very active in the IUCr Congresses, with CSAS sponsoring a keynote speech, five microsymposia (including a number cosponsored with other commissions) and a special session (Advances in data and model validation in biomolecular small-angle scattering: impacts on data and meta-data recording and data archiving) for the upcoming 25th Congress (14–22 August 2021, Prague, Czech Republic). IUCr journals will continue to be a popular choice for leading SAS research, with an array of journals that are natural homes for SAS applications across chemistry, biology and materials, as well as for further technical developments in instrumentation, analysis and standards development. While the era of conventional conference proceedings in IUCr journals is effectively over, special issues of *Journal of Applied Crystallography* that collect up to 25 full-length high-quality peer-reviewed papers associated with work presented at the conference have worked well for both SAS2012 and SAS2015. It will be beneficial for each SAS conference organization to continue to work with IUCr journals to facilitate open access for such special issue papers.

The IUCr pioneered the development of standards, common data formats, open-access data and education in the field of crystallography, diffraction and scattering, and the SAS community represented at the triennial SAS meetings is very active in tackling these issues in their respective subdisciplines. Ongoing cooperation in these activities will be of great benefit, providing for knowledge transfer and collaboration and, importantly, the avoidance of multiple divergent efforts to achieve the same goal.

## Acknowledgements

Thanks to Andrew Allen for the data on SAS publications in IUCr journals and for the information and references on the development of the ISO SAXS intensity standard. Also thanks to Andrew for his notes on the early interactions among the IUCr CSAS members and the SAS community represented at the triennial SAS meetings and to Iris Torriani for confirmation of particular events. Thanks to Pete Jemian and Jan Ilavsky for the data on the triennial SAS meeting attendees. Thanks also U-Ser Jeng (current CSAS Chair), members and consultants of the Commission on Small-Angle Scattering for their review of the text and comments.

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This article was originally published in Trewhella, J. (2021). *J. Appl. Cryst.* **54**, 1029–1033.

Some errors in the original publication are corrected in https://journals.iucr.org/j/issues/2021/06/00/jl9024/index.html

SPECIAL REPORT

# Teaching crystallographic symmetry




Leopoldo Suescun

## 1. Introduction

I remember a particular day in 2010 vividly. Mois I. Aroyo had spent eight hours of that hot Saturday (summertime in Montevideo) talking about Representations of Space Groups, after a full Monday–Friday week of Space Group Symmetry shared between Mois and Massimo Nespolo, with short contributions by Ernesto Estevez Rams, Gustavo Echeverría and Eduardo Granado on Thursday. Mois showed no sign of exhaustion and remained enthusiastic. The surviving audience (~25 of the ~50 school participants), including tutors like myself, were all exhausted but tried to follow the lectures. I had met Mois and Massimo personally only eight days before, on Saturday 28 November, when they landed in Montevideo after a long flight from Madrid. Ernesto Estevez Rams had introduced me to them virtually and pushed me into agreeing to organize this International School on Fundamental Crystallography (ISFC2010). I was a recent Associate Professor at Universidad de la República, and having the chance to organize a regional School sounded like an excellent challenge and a good opportunity to establish a network on teaching fundamentals of crystallography that would complement the already existing School, centered on practical aspects of X-ray diffraction and crystallography.

December 2020 marked the 10th anniversary of that final day of the ISFC2010 MaThCryst School in Latin America [1] held at Facultad de Química, Universidad de la República, Montevideo, Uruguay. This was the first of a series of International Schools that I've since organized in Montevideo or helped to organize in Latin American institutions (before and within LACA's auspices). Joining the International Organizing Committee of the MaThCryst Schools significantly impacted my career, both in network-building and teaching activity aspects. In this article, I want to share some of the more important matters learned in this period, aiming to push colleagues from all over the world to lead similar initiatives that help build the community and educate our students and future colleagues.

Starting with a brief historical description of the development of the series of MaThCryst Schools in Latin America and the most important details of the organization of the events, a review of the outcome based on the opinions of students will follow. After this, I want to discuss how this activity has impacted my teaching work at the local level in the Crystallography "101" course and at symmetry lectures at applied crystallography schools. Finally, I want to share how I would like my teaching activities at all levels and future MaThCryst Schools to develop. The reason for this is that we need more crystallographers able to understand and use symmetry for their analyses. In addition, they have to profit from the modern tools that have been developed by generous colleagues in freely available web servers and databases, and by the IUCr, through the Teaching Edition of *International Tables for Crystallography* Volume A and the online version of *International Tables for Crystallography*.

## 2. MaThCryst Schools in Latin America. The birth of a periodic event

The International School on Mathematical and Theoretical Crystallography in Havana, Cuba, in 2007, was supported by the IUCr Commissions on Mathematical and Theoretical Crystallography (MaThCryst), Crystallographic Teaching and Inorganic and Mineral Structures, and locally organized by Ernesto Estevez-Rams, Arbelio Penton and Cristy Azanza (Havana University, Cuba) with the participation of Commission members Massimo Nespolo (Chair, Université de Lorraine, France) and Bernd Souvignier (Radboud University, The Netherlands) (among other Lecturers) [2]. After this event, the need for this kind of intensive School on Symmetry and the *International Tables for Crystallography* in Latin America became evident to the organizers. I was not involved in that School but had met Ernesto at the LNLS (Brazilian Synchrotron Light Laboratory) in the late 1990s and visited him at Havana University in 2003 for the X Summer School on Materials Science and Technology. This was followed by a short stay during my PhD studies to learn more about X-ray diffraction analysis of materials showing stacking faults. Some years later, when I was back in Montevideo after my postdoc, I joined the XVI Summer School of 2009, but this time as a Lecturer. During that School, Ernesto convinced me to organize a School on Mathematical and Theoretical Crystallography, similar to 2007, but in Montevideo, farther south in the Latin American region. I accepted his invitation with only one big but: the name of the School could not contain the words "Mathematical and Theoretical" if we wanted chemistry, structural biology, materials science and other students to attend. He put me in touch with Massimo Nespolo, and in January of 2010, we agreed to organize the International School on Fundamental Crystallography in December of that year [1]. Our deal was that I would help Massimo manage some schools of this kind in South America. I was hesitant, though, to have them too close in time since I ignored how much demand there would be for learning Crystallographic Symmetry and the financial resources for these schools. A plan was made to organize three such schools, in 2010 (Uruguay), 2012 (Brazil) and 2014 (Argentina), to cover the largest crystallographic communities of South America and put the idea and format of the School to the test. With this in mind, the ISFC2010 MaThCryst School I organized with Ernesto Estevez-Rams, Massimo Nespolo and Mois I. Aroyo also counted on the participation of Raimundo Lora-Serrano, Eduardo Granado and Gustavo Echeverría, future organizers of ISFC2012 and ISFC2014 in Brazil and Argentina, respectively (Fig. 1).



Figure 1. Photos from ISFC2010 (top line) and ISFC2016 (bottom) with organizers names in bold. Top left: **E. Estévez**, S. Cora, S. Píriz, M. Romero, A. Mombrú (LOC member of ISFC2010) and **G. Echeverría** (organizer of ISFC2014). Top right: **M. Nespolo**, **M. I. Aroyo**, R. Burrow, S. Píriz, **E. Estévez**, **R. Lora-Serrano**, **E. Granado** (organizers of ISFC2012) and **L. Suescun**. Bottom picture, front row: **L. Suescun**, **A. Penton**, **M. Nespolo**, **E. Estévez**, **M. I. Aroyo**. Behind L. Suescun are **C. Azanza** (local organizer of MaThCryst School 2007 and ISFC2016) and **R. Lora-Serrano**. Behind and to the right of M. Nespolo is **M. A. Macías** (organizer of ISFC2018).

The School in Montevideo (with 50 attendees from 6 countries) was followed by Uberlandia (2012) [3] (with another 50 attendees from 5 countries) and La Plata, Argentina (together with an IUCr/UNESCO OpenLab in 2014) [4] (with 37 participants from 9 countries), as planned. The success of the school series, measured by the number and diverse nationalities of participants, was demonstrated, so the ISFC MaThCryst School would continue and cover the rest of Latin America. To get back to the north of the region, Havana, Cuba, was selected again for 2016 [5], to celebrate a decade of MaThCryst Schools in Latin America (counting 35 attendees from 9 countries). Bogotá, Colombia, in 2018 [6] would follow, with Mario Macías – a participant in the Havana School – assisted by Elizabeth Jimenez and colleagues, organizing the event. The ISFC2018 School had Arbelio Penton replacing Ernesto Estévez in the International Organizing Committee and the Lecturers' team and involved 39 participants from 9 countries. The seventh MaThCryst School in Latin America was programmed for November 2020 in Lima, Perú but had to be canceled for reasons every reader knows and is now in the process of being rescheduled for 2022 (see below).

The number of attendees at ISFC Schools was on average 40, with a majority (about half of the participants) from the country where the School was held. From that group, between a third and a half came from the city or region of the university hosting the event. Gender balance is a natural characteristic of crystallography in the area and was easily achieved even before the IUCr policy was established. The number of non-local attendees was always limited by the generous but overall scant resources provided by the IUCr and other sponsors of each event. For example, in the case of Montevideo 2010, USD 4650 was given to 14 IUCr Young Scientist Awardees, and more than USD 7000 was spent to cover lodging and lunch for a total of 30 students from abroad. Still, the number of registered participants who could not attend due to lack of financial support was above 30.

## 3. ISFC format and outcome

The ISFC MaThCryst Schools developed with a consistent format. Lectures were taught in university classrooms with basic teaching media support and wireless internet. First, on Sunday, optional introductory lectures were given, primarily for local participants, on the basics of Fourier Transforms and vector and matrix algebra. Next, there were five days of lectures (Monday to Friday) on crystallographic symmetry. Finally, a one-day workshop (Saturday) was organized on a topic of interest to the local organizers. From Monday to Wednesday, point groups, 2D and 3D lattices, plane and space groups were covered, using the notation and examples from *International Tables for Crystallography* Volumes A and A1. Aroyo and Nespolo alternated lectures with the rest of the lecturers/tutors guiding the students with exercises. Thursday was devoted to symmetry of the diffraction pattern and space-group determination from diffraction data (details of the lecturers can be obtained from the events' websites [1–6]). Friday was the day for transformations, group–subgroup relations, and application of these and other concepts using the tools available by the Bilbao Crystallographic Server. The one-day Workshops on the final day (also of optional attendance) covered Representation Theory of Space Groups (2010) [1], Twinning (2012/2014) [3–4], Nanocrystallography (2016) [5] and Phase Transitions (2018) [6].

In the first three days of lectures, where the fundamental concepts and definitions are outlined, the classes consistently follow a pattern beginning with an introduction of a concept or definition: an example is solved, and exercises are given to the attendees for them to solve with the assistance of the lecturers and tutors. This mechanism is a very useful way to quickly diagnose the level of understanding of the students and promote cooperation among them. It also sets the pace of the lectures and keeps everyone active during long days consisting of seven hours of lectures (with two coffee and one lunch break). Even when the concepts or definitions have not been fully understood by the students, having the tutors around allows one-on-one explanations to be given that usually solve the problems and pushes the less active students to keep trying.

As is usual in IUCr-sponsored events, travel and local expenses support grants for non-local participants were given based on academic merit and the background of the applicants. This implied that, in most schools, non-local attendees were slightly more experienced (Master's/PhD students or Professors) and educated in crystallography than the locals, where undergraduate students were a majority. This may be a bit less accurate for large countries such as Brazil or Argentina, where the nationals may come from longer distances than some international participants and may require more expensive travel support. Still, after eliminating some applicants based on merit, a significant number of them had little or no knowledge of *International Tables for Crystallography* Volume A. Fig. 2 shows a collection of answers of the attendees in the post-school evaluation form where their background before the event was recorded. This is a central reason why MaThCryst schools have been so well received and are so needed in Latin America.

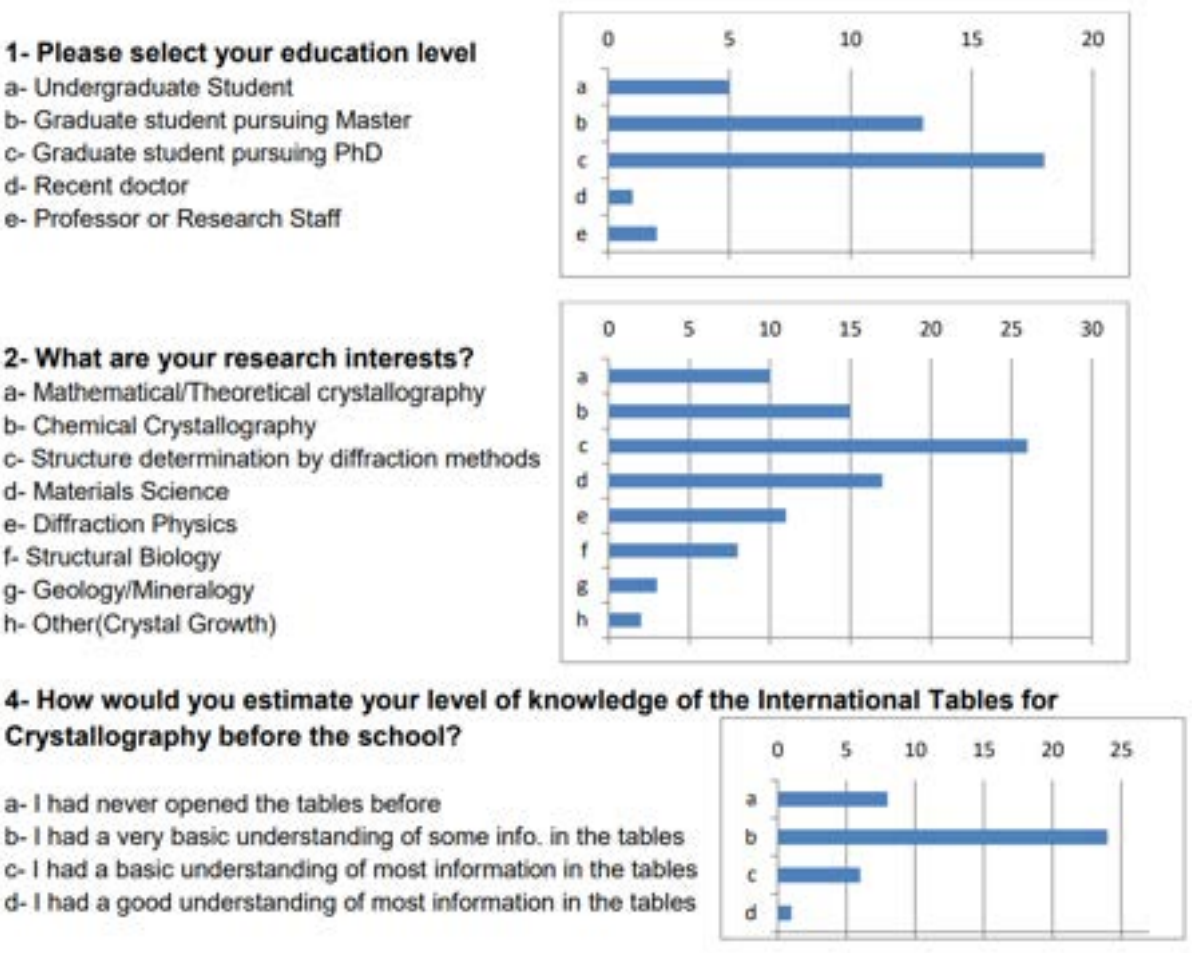



Figure 2. Education level, research interests and knowledge of *International Tables for Crystallography* before the ISFC2010 School in Montevideo. Note that about 80% of the attendees had little or no knowledge of *International Tables for Crystallography* Volume A before the School even after a strict selection.

Even though all of the participants are exhausted by the end of the week, and in every evaluation of the School a request for a free afternoon is repeated, the students evaluate the School very positively (Fig. 3). The need to profit as much as possible from the Lectures, devoting the maximum amount of time possible in the classroom, justified the non-stop program.

Over the years, I have seen a couple of dozen participants in the ISFC Schools become colleagues. Some of them are currently working at highly respected institutions, doing research, teaching and/or developing tools for data analysis. I have exchanged memories of the Schools with many of them, and they all have mentioned how good the ISFC was for their careers. This may just be a demonstration of politeness and respect, so in the following months, I plan to distribute an anonymous questionnaire to the ~200 participants of the different ISFC Schools. I would like to collect their opinion about the impact of ISFC Schools on their formation as crystallographers and ask them for suggestions on improving future Schools. This will probably justify a future article. It has already happened that former ISFC participants have organized a School. Eventually, they will become the Tutors and Lecturers of ISFC, so why not get them thinking about it now?!




Figure 3. Opinion of the participants about their learnings and goals achieved.

## 4. Teaching crystallographic symmetry in other Schools or regular courses

Before 2010 I had taught crystallographic symmetry (among other topics) over 10 different years (1996–2004, 2009) within the Crystallography "101" course at Facultad de Química, Udelar, only missing the course during my postdoc years. The course was intended for advanced undergraduate and graduate students in chemistry. The focus of the course was to introduce the participants to the most relevant theoretical and practical aspects of crystallography and X-ray diffraction (both single crystals and powder), giving them tools to analyze small-molecule structures or perform qualitative powder X-ray diffraction. The course was not expected to delve deeply into mathematical or physical aspects of the discipline, so every qualitative simplification of the topics was welcome (not only by the students but also by myself as I was learning on the road). I had based the initial course on some basic books (Stout & Jensen, 1968 [7]; Woolfson, 1969 [8]) and IUCr Teaching Pamphlets #13 and #14 [9–10], where I had learned about symmetry myself. I have to say that initially (~1996–1998), the quality of my teaching was poor but evolving in parallel with the knowledge I was acquiring during experimental work. Initially, during my Masters' studies (1996–1999), I solved small-molecule structures from single-crystal X-ray diffraction data collected on a point-detector diffractometer (twins, incommensurate or pseudosymmetric structures were not frequent problems). Later, during my doctoral studies (1999–2003), I used synchrotron X-ray and neutron powder diffraction data and the Rietveld method for refining perovskite structures with magnetic components, applying Shubnikov's space groups.

My classroom approach to crystallographic symmetry changed drastically after 2010. Even if I could not reproduce the same content Mois and Massimo taught at ISFC Schools (the audience was significantly different), some central ideas of their method became stepping stones of my classes (Fig. 4). Almost all crystallographic symmetry could be built based on the knowledge of symmetry operations and on the definition of a group. So starting in 2011, my symmetry classes, spanning eight 2-hour lectures, started by introducing symmetry operations that form simple crystallographic point groups and, using group axioms, add more symmetry elements to obtain more complex ones. This is done in a geometrical way first (easier to visualize for chemistry students used to representing molecules in space) and in algebraic form later, using matrices. Later, simple lattices in 2D are obtained from identical periodic rows of nodes and ideas on how to derive Bravais lattices from 2D lattices are introduced (not formally derived for time economy). Finally, symmorphic space groups are introduced by combining simple point groups with lattice translations. Later, symmetry operations with translation components are added, emphasizing

that space groups have an infinite number of periodically ordered symmetry elements. *International Tables for Crystallography* are used to show the progression of complexity of space groups and the standard notation. Feet and hand patterns from Teaching Pamphlet #14 are also used, first to learn how to search for symmetry operations, later to determine the coordinates of equivalent positions and the matrix-column pairs for symmetry operations, and finally to determine the space-group symbol.

The logic behind this approach is simple and straightforward, and it could be used to introduce crystallographic symmetry even in a couple of 2-hour lectures, as is the norm in single-crystal or powder diffraction schools of which I've been an Organizer or Lecturer. I have always devoted one class to point groups with geometrical and algebraic approaches finishing with a quick view of Bravais lattices and the final class to space groups and *International Tables for Crystallography* Volume A. For time economy, I discuss triclinic, some of the simpler orthorhombic and the monoclinic $P2_1/c$ space group, which is so frequently encountered (one out of three structures deposited at the CSD) [11] that the students have heard of it or will need it sooner or later. It may not be possible in such a short time to derive and study the more complex point or space groups or give an exhaustive classification of them. But an idea of how they could work them out remains.




Figure 4. Two of the most important slides of my Symmetry classes.

Some may say it is a waste of time to teach what is in *International Tables for Crystallography* Volume A, and what they are used for, in a couple of 2-hour lectures with at most 2-hours of exercises. I would agree that not much will be of use for an absolute beginner after the class. However, I emphasize the idea that even the most complex space groups could be deduced by adding symmetry operations to simpler space groups in a step-by-step procedure. The idea in the mind of all of the students is, in my humble opinion, that it is possible to understand crystallographic symmetry by patiently going through all steps, point groups, then lattices and then space groups. It will take them time, but it is possible.

## 5. Perspectives

With the cancellation of Schools during the COVID-19 pandemic, there will be a gap in the preparation of students regarding all aspects of crystallography, but especially of crystallographic symmetry, rarely covered in depth in local courses in the region. The slow restart of international schools in 2022 will generate increased demand, as has been already observed in online courses (the 3rd LACA School on Small Molecule Crystallography held online in December 2020 had 60 participants, but 120 registered).

The International School of Fundamental Crystallography has not been held for three years already, and we are planning the next for the last trimester of 2022, almost certainly at Anápolis, Goiás, Brazil, organized by MaThCryst member Hamilton B. Napolitano, Professor at the Universidade Estadual de Goiás. I expect that we will have well over 100 applications, but the number of attendees will not exceed 50 or 60. On one side, classroom space (and likely limitations to the occupancy of classrooms are in place) will prevent larger numbers. On the other side, larger audiences will be tough for Lecturers and

Tutors in the practical sessions. In addition, financial resources for travel support will also limit the attendance to those students that could pay for most of their expenses or will travel from shorter distances.

Other international and national schools held periodically in Latin America will also resume in 2022 and 2023. Hopefully, the hybrid in-person/virtual mode of attendance will be implemented to widen the audiences and reduce the financial costs of the organization. These will also have an increased demand in any of the modes, as the growth of the synchrotron and neutron users communities develop, with the stepwise opening of beamlines at the Sirius synchrotron source in Campinas, SP, Brazil [12] and the start of operation of the Laboratorio Argentino de Haces de Neutrones (LAHN) in Ezeiza, Buenos Aires, Argentina [13] in 2024.

On the bright side, this gap gave time for the significant efforts of Mois I. Aroyo (Editor), contributing authors and the IUCr Editorial Office in Chester to finish successfully the new Teaching Edition of *International Tables for Crystallography*: Crystallographic symmetry [14], which was published in April 2021. This book, better adapted for teaching and for individual study [15], will become an essential addition to the toolbox of Lecturers and Tutors and may play a significant role in the education of Latin American (and all around the world) students during the next decade if its availability (price, web access, *etc.*) is adequately adapted to the circumstances. I think an effort by the IUCr to give away e-copies of the book for free to school applicants/attendees and allow for access to the online tools of *International Tables for Crystallography* Volumes A and A1 and the *Symmetry Database* will be very important to support the efforts of Lecturers in our region.

In conclusion, after a decade of participation as a lecturer of crystallographic symmetry in X-ray diffraction schools and symmetry-focused schools in Latin America, I have witnessed the evolution of demand and progress in the community. The pandemic came with a toll, as reduced access to travel reduced the number of Schools and probably the quality learning for the virtual attendees of online events. A strong effort will be needed to restore the situation to pre-2020 levels in the next couple of years on the side of Lecturers and institutions organizing more and better Schools and on the side of students to adapt to reduced travel funds and mobility. However, the IUCr (accompanied by other institutions and companies that profit from the existence of many good crystallographers in the region) is capable of helping to defray the cost of this situation by promising more financial help and free access to educational resources. The MaThCryst Commission I represent and LACA are looking forward to working hard for more and better crystallography in Latin America and around the world.

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Editor's note: The IUCr is pleased to announce that it plans to put the Teaching Edition online. This resource will be made available free of charge to IUCr-supported schools along with the customary free access to International Tables Online and the *Symmetry Database*.

The paperback version of the Teaching Edition is available at low cost from Wiley [USD 40.00/EUR 33.90/GBP 29.99 excluding taxes (if applicable) and postage]. Bulk discounts may be available; please contact Wiley's Corporate and Custom Sales team.

MEETING REPORT (IUCR SUPPORTED)

# 55th Course of the International School of Crystallography: Molecular Crystal Engineering




The Erice21 participants "at" Venus Castle.

Annalisa Guerri, Paola Spadon

The organization of a course on Molecular Crystal Engineering began in 2015 with the aim to continue the series on the topic initiated in Erice in 1995. The course was originally planned to be delivered in the usual venue of the Ettore Majorana Centre in Erice. Due to the on-going COVID-19 pandemic and the gathering/travel restrictions associated with it, the course was delivered online instead.

The online version was scheduled taking into account several aspects: to allow for the different time zones of the participants, to avoid long periods in front of the screen and to convey the spirit of Erice. In light of these points, the official online school was restricted to 31 May–4 June 2021 with only a four-hour section per day but, to ensure a high-quality school, lectures were prerecorded and distributed to the participants three weeks before the official opening day while during the official meeting, discussion was favored. A “Preparation” week was also introduced from 24 to 28 May; during this week the students met daily in small groups to discuss each of the recorded lectures and to collect questions. The questions were then passed to the lecturers and addressed during the live-streamed sessions from 31 May to 4 June. These meetings were facilitated by the “orange scarves”, selected students appointed to help with organization. Each day was dedicated to a specific theme: Theory, Solid Forms, Analysis, Properties, Applications.

The scientific program was delivered by 23 lecturers chosen for their academic reputation as leaders in their respective field. Additionally, six outstanding guest speakers (Professors Angelo Gavezzotti, Mier Lahav, Lesley Leiserowitz, Carol Brock, Judith Howard and Bruce Foxman) gave an historic perspective on crystallography to the younger participants. In the “Preparation” week, two workshops were delivered by the CCDC.

Each day was concluded by a Panel Discussion amongst the speakers open to additional questions from the participants.

Participants were selected according to a set of criteria that guaranteed a diverse representation. Gender and geographical balance were pursued. Applicants in their mid/final years of a PhD were favored to ensure their active participation in the scientific discussion. A total of 36 participants presented their work during 2 poster sessions. Each presenter introduced their work with a 1 minute pre-recorded video

presentation.



The Erice2021 participants "visit" the San Domenicano Convent.

During the closing ceremony, poster prizes were awarded, supported by ELDICO, *CrystEngComm* and the International School of Crystallography. The prizes were awarded to Avital Wagner (Ben Gurion University, Israel), Divya Amin (UCL, UK) and Torvid Feiler (BAM, Germany), respectively. In addition, the Lodovico Prize was awarded to Marco Bardini (University of Mons, Belgium). At the end of the course, a questionnaire was circulated amongst all the participants with a 75% response rate. Overall, 37% of the responders ranked the course as the best online event in which they had participated, and an additional 52% ranked it amongst the best. In particular, the students really appreciated the opportunity to meet during the “Preparation” week, which helped make their participation in the school more fruitful.

IUCr funds were used to partially cover the cost of the set up of the online platform for the lectures and the poster sessions, and to allow the participation of four PhD students from Argentina, Estonia, Mexico and Morocco.

MEETING REPORT

# FEBS practical crystallization course "Advanced methods in macromolecular crystallization IX"



In the lab: Martin Caffrey (Trinity College Dublin, Ireland) explains crystallization experiments in lipidic cubic phase. Photo: Petr Pachl.

José A. Gavira, Pavlina Řezáčová, Ivana Kutá Smatanová

The ninth FEBS practical crystallization course "Advanced methods in macromolecular crystallization“ was planned to take place in Nove Hrady, Czech Republic, in May 2020. This was not possible due to the COVID-19 pandemic and, after a further postponement (October 2020), the final course date was set as 9–14 August 2021. This change also affected the choice of location, which was moved by about 30 km to Ceske Budejovice (Budweis), at the Faculty of Sciences of the University of South Bohemia. What did not change was the large amount of interest shown by students and postdocs in participating in the course, and the dedication and enthusiasm of the lecturers, tutors and organizers.

Ivana Kutá Smatanová (University of South Bohemia), Pavlina Řezáčová (Czech Academy of Sciences), Jeroen R. Mesters (University of Lübeck, Germany) and José A. Gavira (National Research Council & University of Granada, Spain) organized the course under the auspices and main support of the Federation of European Biochemical Societies (FEBS). In addition, eight companies sponsored the course mainly by providing their equipment and required material for the lab exercises and also by partially covering the registration fee of five selected students (YTF FEBS grant and a grant from the Czech Society for Structural Biology).



In the lecture hall: Lubica Urbanikova (Slovak Academy of Sciences, Bratislava, Slovakia) explains to participants that the nature of the protein is the most important variable during crystallization. Photo: Petr Pachl.

The course gathered 28 speakers/tutors from 11 different countries (11 female and 17 male) with outstanding records in the fields of protein crystallization and crystallography. They delivered 27 morning and 4 evening lectures to 25 students (12 female and 13 male). The course was opened with welcome speeches from the Vice-Chancellor of the University of South Bohemia, Professor Tomas Machula; the Dean of the Faculty of Science of the University of South Bohemia, Professor Hana Santruckova; and a FEBS representative, Professor Libor Grubhoffer. After the ceremony, the participants – PhD students and postdocs – were introduced to some of the advanced methods of protein crystallization. The first day ended with a welcome party in which the students had the opportunity to introduce themselves to the speakers on a 1:1 basis. The course followed from Tuesday to Friday with a very intense and packed agenda from morning (08:30) to night, with lectures and lab exercises covering everything from biophysical techniques used for the analysis of protein properties and sample quality before crystallization (DLS, protein labeling, *etc*.) to protein crystallization screening, crystal improvement, *in vivo* crystallization, *etc*. The students were able to perform experiments on their own proteins or use model proteins.




The three IUCr poster prize winners with the three members of the selection committee: l-r Stefan Djukic (student at Charles University, Prague, Czech Republic), Kevin Rollet (student at the University of Strasbourg, France), Juan Manuel Garcia-Ruiz (National Research Council and University of Granada, Spain), Kathrin Bach (student at Charles University, Prague), Crissy L. Tarver (Stanford University, CA, USA) and Radka Chaloupkova (Masaryk University, Brno, Czech Republic). Photo: Petr Pachl.

Time was allocated for poster sessions and discussions with the speakers. Three students' posters were awarded IUCr Journals poster prizes, which were presented in a short lecture during the closing ceremony on Friday. Saturday morning was devoted to crystal observation and final evaluation of the practical results.



The organizers, happy that the course could be organized after three shifts in time and one shift in place: l-r José A. Gavira (National Research Council and University of Granada, Spain), Ivana Kutá Smatanová (South Bohemian University, Ceske Budejovice, Czech Republic), Pavlina Řezáčová (Czech Academy of Sciences, Prague, Czech Republic) and Jeroen Mesters (University of Lübeck, Germany). Photo: Petr Pachl.

We would like to finish by thanking not only FEBS and the sponsors for keeping this course alive but also all the speaker/tutors, who really contributed to the very friendly atmosphere during the whole week.

HISTORY OF CRYSTALLOGRAPHY

# Images of Dorothy Hodgkin



Graham Sutherland’s sketch of Dorothy Hodgkin, 1980, Science History Institute (Meyer 2018 [4]).

Magdolna Hargittai, Istvan Hargittai

This Note is about two little-known photographic images of Dorothy Hodgkin (1910–1994). She was a world-renowned crystallographer with such famous structures in her oeuvre as penicillin, cholesterol, insulin and vitamin $B_{12}$. She was awarded the 1964 Nobel Prize in Chemistry “for her determination by X-ray techniques of the structures of important biochemical substances.” She was not only well known; she was also well loved. We met her for the first time in 1971 in Manchester, UK, at a crystallography meeting and for the last time at the 1993 IUCr Congress in Beijing, China. We have the warmest memories from all our meetings with her. In addition to her greatness in science, her popularity probably added to her becoming the subject of artistic expression. We mention this before introducing the two, what we think, are little-known photographic images.

Actually, we should mention the works of four artists. Patricia Fara (2002, [1]) has written about two of them, and both give emphasis to Hodgkin’s severely deformed hands due to rheumatic arthritis from youth. A drawing of Hodgkin’s hands by Henry Moore, 1978, is at the headquarters of the Royal Society of London. An oil painting of Dorothy Hodgkin by Maggi Hambling, 1985, is displayed at the National Portrait Gallery in London. The peculiarity of this image is that it shows the scientist at her desk with two pairs of hands. To us, Hambling sees Hodgkin in redoubled activities despite her handicap.

The Royal Society has another oil painting of Dorothy Hodgkin, and this is by Bryan Organ, 1982, and we can say a few more words about this project based on Sir John Cornforth’s narratives (1982 [2], 1998 [3]). The Council of the Royal Society decided in 1977 that the situation that no woman Fellow had any portrait yet should be remedied. Nonetheless, the Council offered no sponsorship for a Dorothy Hodgkin portrait.

A brief and most successful public subscription brought together the necessary Portrait Fund. Next, they had to find an artist, and this was Graham Sutherland OM (1903–1980). As it turned out, Hodgkin and Sutherland had met in person at what must be one of the world’s most exclusive luncheons. It is given once every five years by the Queen for the members of the Order of Merit, OM, whose membership is limited to 24. Both Hodgkin and Sutherland were OM members. Sutherland made sketches, one of which is at the Royal Society and the other is at the Science History Institute in Philadelphia (see the image above, Meyer 2018 [4]). These sketches eminently grasped Hodgkin’s demeanor. Alas, Sutherland fell ill and died before he could complete the painting. The task was then completed by another artist, Bryan Organ.

Now, about the two photographs.



Detail of a large group photo of the participants of the 1934 International Conference on Physics in London, UK: Dorothy Crowfoot (as Hodgkin was then) is eighth from the right, back row; Patrick Blackett is in front of her to the right, J. Desmond Bernal is in front of Blackett to the right, and Enrico Fermi is at the right edge of Bernal's row. The picture was taken by London Panoramic Company, and Gerhard Hund, Friedrich Hund's son, gave the photograph to Wikimedia Commons to be placed in the public domain (colorized by photomyne.com).

The detail of the photograph displayed above shows Dorothy Hodgkin among the participants of the 1934 International Conference on Physics in London, UK. The meeting was organized by the Royal Society, and the picture was taken in front of the then headquarters of the Royal Society at Burlington House. Friedrich Hund of Hund's rule for electron structures of atoms, was one of the many attendees of the meeting. Hund marked the names of famous physicists he recognized on the photographs. Even his partial list demonstrates the importance of the meeting. Note, however, that he failed to mark Dorothy Crowfoot, as she was then. At 24, she must have been one of the youngest attendees of the meeting. From among Hund's marks we quote: J. Desmond Bernal, Vilhelm Bjerknes, Patrick Blackett, W. Henry Bragg, W. Lawrence Bragg, Robert Brode, Blas Cabrera, Hendrik Casimir, Arthur H. Compton, Charles G. Darwin, Walter M. Elsasser, Paul P. Ewald, Enrico Fermi, Siegfried Flügge, William A. Fowler, Otto R. Frisch, Douglas R. Hartree, Frederick Hoffmann, Johan P. Holtsmark, Friedrich Hund, Erich Hückel, Egil Hylleraas, Frederick Joliot-Curie, Willem H. Keeson, Martin Knudsen, John Lennard-Jones, Fritz London, Theodore Lyman, Charles L. Manneback, Hermann Minkowski, Francis Perrin, George Placzek, Franco Rasetti, Lord Rayleigh, Owen W. Richardson, Stefan Rozental, Paul Scherrer, Erwin Schrödinger, John C. Slater, Adolf Smekal, Leo Szilard and Gian C. Wick.



Dorothy Hodgkin and Albert Szent-Györgyi, 1947, in Cambridge, UK. The photograph is courtesy of William B. Jensen and the Oesper Collection of the University of Cincinnati, OH, USA.

The above photograph of Dorothy Hodgkin with the biochemist Albert Szent-Györgyi was taken by an unknown photographer in 1947 in Cambridge, UK. Szent-Györgyi received the Nobel Prize in Physiology or Medicine in 1937 for his discoveries related to biological combustion and vitamin C. In 1947, he was an escapee from the emerging Soviet-type communist dictatorship in Hungary, on his way to the United States.




Dorothy Hodgkin on British postage stamps in 1996 (left) and 2010.

We augment this brief account by including two well-known images of Dorothy Hodgkin. She appeared on a British postage stamp for the first time soon after her death, in 1996, along with a few other women celebrities. Then, in 2010, there was another Dorothy Hodgkin postage stamp. It was in the framework of

celebrating the 350th anniversary of the founding of the Royal Society. She was the only woman depicted among the 10 illustrious Fellows. Listing the other 9 adds to the weight of this distinction: Isaac Newton, Edward Jenner, Joseph Lister, Benjamin Franklin, Charles Babbage, Robert Boyle, Ernest Rutherford, Nicholas Shackleton and Alfred Russel Wallace.

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HISTORY OF CRYSTALLOGRAPHY

# John Desmond Bernal: his contributions to crystallography



Christine Slingsby, Paul Barnes, John Finney, Vincent Casey

*The 50th anniversary of John Desmond Bernal's death (15 September 1971) seems an appropriate occasion to look at his fundamental role in the development and exploitation of crystallography. The following three articles explore his influence on structural biology, material science and our understanding of liquid structure (including water). The final article describes his recent scientific legacy in Ireland (the country of his birth), which is helping to realise his concern that science should be used for the benefit of society. These articles were originally published in the June 2021 issue of Crystallography News, and are reproduced with permission.*

## Bernal's early influence on biomolecular structure, and on protein crystallography

### Christine Slingsby, Birkbeck College, University of London, UK

Bernal had the satisfaction of determining the atomic structure of graphite in the early days of X-ray crystallography under the guidance of William H. Bragg while at the Davy–Faraday Laboratory at the Royal Institution in London (1923–1927). Bernal was less enchanted with the tedium of indexing the diffraction spots, and so he devised ways of aiding the interpretation of single-crystal rotation photographs by inventing Bernal charts. His first two papers published in 1924 and 1926 brought him early recognition as a leader in X-ray crystallography. However, recognising even then the potential of the technique, he wanted to extend the reach of X-ray structure analysis to the biological world. As both he and his colleague Bill Astbury wanted to tackle proteins, they divided the field between them, Astbury taking on the fibres, Bernal the soluble globular proteins.

In 1927 Bernal moved back to Cambridge as a Lecturer in Structural Crystallography in the Department of Mineralogy next door to the Cavendish. In his quest to understand the biological world he took advantage of the proximity of the biochemistry laboratory, led by Frederick Gowland Hopkins, who considered that enzymatic activity was responsible for the biological activity of living cells. Although little was known of protein structure at the time, it was known that chemically they were chains of amino acids. In the summer of 1928 Bernal travelled around European laboratories to catch up on progress in protein research. He began work on amino acids and related compounds using crystals prepared by A. Leese in Biochemistry. Atomic resolution structures of these molecules were beyond reach at that time, but Bernal had shown from his Cambridge student days that he had the kind of geometric mind that could independently derive all 230 space groups (see Paul Barnes's article below). He therefore embarked on optical and X-ray characterisation of the unit cells to reveal likely models of the amino acids

and how they packed together in the crystals. The final paragraph of the survey published in 1931 reads: “A detailed account of the crystallographic investigation with a discussion of its chemical implications will be published at a later date. In the meanwhile, in view of the difficulty of obtaining crystals of these compounds the author would be very grateful if anyone possessing such crystals of a magnitude of 0.01 mm or over would send a few mgms of them to him for examination”.

In the early 1930s Solly Zuckerman and J. B. S. Haldane steered Bernal towards new crystals of the sex hormone oestrin. He was able to figure out from unit-cell dimensions, symmetry and optical properties that all steroids were likely to have a similar structure, which was not in agreement with the ideas of the leading chemists of the day. Bernal’s observations led to a rapid reappraisal of sterol chemistry. The sterol success boosted Bernal’s reputation among chemists and filled his desk with samples. In 1932 an Oxford graduate Dorothy Crowfoot joined him as a PhD student. He invited her to sort out his desk and she became his principal photographer of biological samples.

## Protein crystallography begins

The key experiment performed by Bernal in 1934 that started protein crystallography is described in *Nature*, with an opening sentence that reads like a diary entry: “Four weeks ago, Dr G. Millikan brought us some crystals of pepsin prepared by Dr Philpot in the laboratory of Prof. The Svedberg, Uppsala.” On exposure to air the crystal birefringence diminished and X-ray photographs taken in the usual way showed nothing but a vague blackening. So Bernal drew a new pepsin crystal into a fine glass tube, surrounded by its mother liquor. To his delight this time there was a pattern of spots extending all over the film that sent him wandering the streets of Cambridge through the night. From the intensity of the spots near the centre, he proposed that protein molecules are relatively dense globular bodies, separated by relatively large spaces which contain water. From the intensity of the more distant spots, he inferred that the arrangement of atoms inside the protein molecule is of a perfectly definite kind, although without the periodicities characterising the fibrous proteins, as determined by Astbury. Several more pepsin photographs were taken by Dorothy, but further interpretation was abandoned. The molecular basis of the activity of the enzyme pepsin was within grasp, but not yet.



Figure 1. Dorothy Hodgkin discussing things with Bernal. Courtesy of S. Jane Bernal.

Although Dorothy was not in Cambridge the day the first photo was taken, her name was on the publication. She remained in close contact with Bernal when she moved to Oxford later that year and acquired some insulin crystals. He mentored her through the X-ray work and pushed her to publish a single-author paper in *Nature* in 1935 on her early results on dry insulin crystals. In 1935 in correspondence with Bernal on insulin she says: “It was clear that Bernal was thinking of the possibilities of changing zinc in the crystal for heavier metals and using the change to find the phase angles necessary for the direct calculation of an electron density map of insulin – the method of isomorphous replacement.” Bernal’s steering allowed her at a young age to be in a position to pursue independent research. Her subsequent application of X-ray crystallography to solve penicillin, vitamin $B_{12}$ and insulin changed the way chemists approached the development of medical therapeutics.

## Viruses and haemoglobin

Isidor Fankuchen came to work with Bernal in 1935, ostensibly to work on sterols, having been trained in crystallography by W. Lawrence Bragg in Manchester. Instead Bernal had him work on plant virus particles, such as tobacco mosaic virus (TMV), in collaboration with N. W. Pirie, who had purified a ‘crystalline protein’ from infected tobacco leaf sap. Bernal was keen to define the nature of the crystallinity. By measuring diffraction data from wet and dry gels and solutions, they showed that the sample was in fact liquid crystalline. They deduced that the virus particles formed long rods that probably contained some RNA. Most of the diffraction peaks originated in the internal structure of each rod, for which Bernal, typically, had this to say: “A complete interpretation of this intramolecular pattern has not yet been possible; but a rough survey indicates that the molecule is made of piles of sub-molecules of dimensions 22 Å x 20 Å x 20 Å, somewhat smaller than the normal protein molecule, and themselves divided into nearly identical units with half these dimensions.” But there was little interest in the substance, other than it was possible to follow the wake of a goldfish swimming in a solution of it when viewed under polarised light.

Bernal and Fankuchen were also intrigued by spherical viruses, and X-rayed crystals of tomato bushy stunt virus (TBSV). In Dorothy Hodgkin's Royal Society Biographical Memoir of Bernal, she tells us that: "The photograph was necessarily a powder photograph, though of wet crystals, and showed at first only two lines. With a pair of dividers and a ruler on the wet film, in great excitement, Bernal measured these lines and, assuming as was likely from the form of the crystal, that the lattice was cubic body-centred, deduced a cubic unit cell of edge 390 Å, containing two particles of diameter 340 Å and a molecular mass of the order of 10 000 000." However, the more reflections they recorded, the less good the fit with any symmetrical model, so the work was paused.

When Max Perutz arrived in Bernal's lab in 1936, he wanted to work on a biological molecule. With enthusiastic support from Bernal, he used his own initiative to get crystals of horse methaemoglobin, with the first diffraction photographs taken in late 1937. With help from Fankuchen, a *Nature* paper was rapidly published in March 1938. Wet crystals of both haemoglobin (and chymotrypsin) diffracted X-rays to high resolution with perfectly definite reflections at spacings as low as 2 Å, proving the internal regularity of the protein molecules down to atomic dimensions. Furthermore, controlled dehydration of the crystals caused variation in the intensities which Bernal saw may make possible the direct Fourier analysis of the molecular structure once complete sets of reflections became available in different states of hydration.

## Birkbeck College

Bernal had ambitions to form an Institute of Biomolecular Research that would be organised at the boundaries of classical disciplines, but as he was disliked by the Head of the Cavendish, Ernest Rutherford, and out of favour with the University establishment, he could not progress his career in Cambridge. Early in 1938 Bernal was appointed Professor of Physics at Birkbeck College, University of London, taking over from Patrick Blackett. Formerly the London Mechanics' Institution, Birkbeck was organised so that working people could study for degrees in the evening, and so was more aligned with Bernal's politics.

The department was housed in the ancient Bream's Buildings, close to Chancery Lane. W. L. Bragg moved to Cambridge in 1938, having been appointed Cavendish Professor in succession to Rutherford who had died unexpectedly. Bragg and Bernal shared out the Cambridge apparatus and students. Max Perutz wished to stay in Cambridge with his crystals and Bragg was keen to keep him. Fankuchen went with Bernal to Birkbeck with the virus projects, soon to be joined by Harry Carlisle, a student from Burma.

Bernal summarised his thinking on proteins at a Friday evening discourse at the Royal Institution in January 1939, which was published the following April in *Nature*. He proclaimed that the phase problem can only be resolved by some physical artifice, such as the introduction of a heavy atom, or the observation of intensity changes on dehydration. The paper also includes his thoughts on "how the behaviour of the hydrophobe groups of the protein must be such as to hold it together… a force of association is provided which is not so much that of attraction between hydrophobe groups, which is always weak, but that of repulsion of the groups out of the water medium". Charles Tanford judged this overview as "a pioneering and prophetic paper, a glimpse into a rare moment when one man's insight was able to encompass simultaneously all the strands of a complex problem, much of which the rest of the protein community would not understand for another twenty years".

What Bernal – or anyone else – did not foresee at the time, was that a polypeptide chain could form a regular helix with a non-integral number of amino acids per turn. In the early 1950s Linus Pauling proposed the alpha helix which respected the planarity of the peptide bond, had around 3.7 amino acid residues per turn, with a predicted distance repeat of 5.4 Å, close to, but not exactly matching, the 5.15 Å repeat measured by Astbury in unstretched keratins.

In the summer of 1939 Bernal undertook an extensive trip to the US, visiting labs and giving lectures, including to the Rockefeller Institute. He made contact with Moses Kunitz, who specialised in crystallising enzymes, and this may have been the occasion at which he gave Bernal a tube of ribonuclease crystals.

Bernal cut short the trip to return to the UK as war was coming. He evacuated his students and equipment to Dorothy in Oxford. (Harry Carlisle took his aging cholesteryl iodide crystals to her lab and she guided him through the calculation of a 3D map phased from the heavy atom. The structure, announced in Cambridge in 1943, was the first time a complex, asymmetrical biological molecule was visualised directly by X-ray analysis and opened a new path to determining unknown chemical structures.) Bernal feared that outbreak of war would stop all work on proteins in Europe, so he dispatched Fankuchen to the US with most of his materials in the hope he could continue there. At the start of the London Blitz, Fankuchen submitted a paper (from MIT, Cambridge) on X-ray diffraction of dry crystals of ribonuclease, while in the early hours of 25 September 1940 Birkbeck College was hit by incendiary bombs.

## The war interlude

In 1939 Bernal suspended his direct academic activities. He joined the Civil Defence Research Group, and in spite of his MI5 dossier, was recruited by a member of Chamberlain's cabinet, Sir John Anderson, who is quoted as saying "even if he is as red as the flames in hell, I want him". In 1942 Bernal became Scientific Adviser to Combined Operations under Lord Louis Mountbatten.

As an Austrian, Perutz became a refugee in 1938. He was arrested in Cambridge as an enemy alien in May 1940 and transported under appalling conditions to Canada where he was interned in a camp. He was able to return to his red crystals in Cambridge at the start of 1941, but was desperate to join the war effort. Bernal recruited him onto the doomed Habbakuk floating ice strip project (see Paul Barnes's article below), based under Smithfield Market, a role which required Perutz to travel to the US for demonstrations. The Home Office was pressed in 1943 to grant Perutz naturalisation, issuing him a British passport with a US visa. Max was soon able to network with US protein scientists like John Edsall at Harvard and Martin Buerger at MIT, and to showcase his successful haemoglobin work. Meanwhile Bernal told Pauling he had "left the field myself for the duration to spend my time being an engineer, architect and explosives expert". However, Bernal went on to make more charts, this time for the landing beaches of Normandy. After D-Day, Bernal was sent to Ceylon. While testing bombs for jungle use alongside Wing Commander John Kendrew, he convinced Kendrew of the central importance of protein structure to understand the biology of living systems and directed him to Perutz in Cambridge.

## The 'new' lab in a (very) old building

In 1947 the MRC agreed to support Perutz, Kendrew and a small team in a Unit within the Cavendish for five years. Francis Crick joined in 1949. Meanwhile Bernal's group was dispersed in temporary accommodation until his Biomolecular Research Laboratory, at 21–22 Torrington Square, was opened in July 1948 by W. L. Bragg. The research strategy was to exploit and enhance physical methods to understand the structure and function of soluble proteins and viruses, as well as to pursue research into materials and liquids (see articles by Paul Barnes and John Finney below) in conjunction with the development of computing methods. Bernal was looking for a link between long-range interactions of proteins and biological systems, though was pessimistic about being able to buy the newly invented electron microscope. All this was expected to take place in a pair of partially repaired bomb-damaged Georgian houses in Bloomsbury that was supposedly temporary, but lasted for 20 years.



Figure 2. The Birkbeck Laboratory from 1948 to 1966.

Although Bernal, like most scientists in the early 1940s, considered genes might be proteins, he recognised that chromosomes and viruses share a similar chemical composition. In his post-war Birkbeck lab, he directed Harry Carlisle to supervise PhD student Sven Furberg, who solved the structure of a nucleoside, cytidine. The puckered pentose ring was perpendicular to the flat base ring, an observation noted by Watson and Crick in their DNA double-helix paper.

Getting a biological crystalline sample from an outside laboratory inside a well-equipped X-ray lab and into the hands of a brilliant investigator was the recipe for advancement in the early days of biological structure analysis. In 1953 Bernal invited Rosalind Franklin to bring her fellowship to Birkbeck to follow up his pre-war studies of the rod-shaped TMV. Her X-ray diffraction photographs of oriented gels of TMV, which showed evidence of helical diffraction, enticed Aaron Klug to join her team in early 1954. They established the helical symmetry of TMV, showing it had 49 subunits per three turns of the helix. In 1955 Don Caspar, an American biophysicist on a fellowship in MRC Cambridge, came to work in Franklin’s lab. They showed from a comparison of X-ray fibre diagrams of oriented gels of intact TMV and the reassembled isolated protein labelled with heavy atoms that the RNA was inside the helical protein shell, embedded in the protein subunits. Shortly before her early death in 1958, Franklin began work on poliomyelitis virus, sourced from Wendell Stanley, Berkeley. Klug and Caspar continued the polio work and raided the fridge for Bernal’s old samples of spherical viruses. Caspar took the TBSV crystals while Klug and John Finch worked on the turnip yellow mosaic virus (TYMV). All turned out to have icosahedral symmetry, with 180 subunits. Caspar and Klug wrote their classic paper in 1962 on quasi-equivalence, taking inspiration from Buckminster–Fuller’s construction of geodesic domes. This paper brought new ways of thinking about loosening regularity in modular protein assemblies, as well as a basis for the interpretation of virus electron micrographs. In 1962 Klug with John Finch and Ken Holmes, who had both gained their PhDs at Birkbeck in 1959, moved to the newly opened MRC Laboratory of Molecular Biology (LMB) in Cambridge.

## Bernal, Booth and the double helix

During the Birkbeck 2004 degree ceremony, Andrew Booth, a Bernal protégée, and a founder of computer science, whose multiplication algorithm sits in many contemporary laptops, was being made an honorary fellow. In his acceptance speech, Booth reported a story involving himself, Bernal, Rosalind Franklin and the Nobel laureates Crick and Watson.

When Rosalind Franklin was at King's College before she moved to Birkbeck, Bernal arranged a meeting of these five to discuss the state of knowledge of the structure of DNA. The meeting was friendly and relaxed, and the participants were fully aware of the possibility of DNA assuming a double-helix shape, since this was openly discussed at the meeting. Crick and Watson published their DNA double-helix theory shortly afterwards. If this account is true, and I have no reason to think it is not, it may not rewrite history, but it adds to our knowledge of the timeline that led to the double-helix discovery.

**Paul Barnes**

The phase problem was solved by Perutz in 1953, not by dehydrating crystals, but by introducing the physical artifice of a heavy-atom derivative into haemoglobin enabling him to calculate the first two-dimensional electron density projection of haemoglobin. Kendrew was the first in 1960 to calculate a high-resolution tertiary structure of a protein, monomeric myoglobin, showing rather unexpectedly that the alpha helices were not lying in an orderly parallel fashion. Perutz's team calculated the quaternary tetrameric structure of haemoglobin in 1968.

In 1950 David Harker accepted the challenge of building a team to solve a protein structure for USD 1 million. The Protein Structure Project was set up at the Polytechnic Institute in Brooklyn, following successful lobbying by Fankuchen, with ribonuclease as its target. A 2 Å structure was published in *Nature* in 1967. Without the luxury of such a large budget, Bernal and Carlisle started work on ribonuclease in the post-war years, trudging the painful path of searching for heavy-atom derivatives. The structure was published in 1974.

Bernal had a mission to establish crystallography as a distinct branch of science. In 1951 he began his difficult campaign with Birkbeck to extricate crystallography from physics into a separate department. In 1963 Bernal got his Chair in Crystallography, but the splitting of the two departments was delayed until 1967. Bernal's first stroke, which he largely recovered from, was in the summer of 1963. In late summer 1965 he suffered a disabling stroke, but his brain remained highly active, and he could, though with difficulty, communicate with colleagues, give lectures and write papers and books. He retired in 1966, just as the new crystallography laboratories were opened, and Harry Carlisle took the Chair. A couple of years later, Bernal had a heart attack and suffered a further stroke that left him essentially speechless for two years until his death on 15 September 1971. Carlisle continued protein research on ribonuclease, and new projects on plasma transferrins and eye lens crystallins were begun. Protein crystallography underwent rapid expansion when Tom Blundell, a former colleague of Dorothy Hodgkin, joined the department in 1976, taking the Bernal Chair in 1978.

## Concluding comments

Bernal was clearly a – if not the – major driving force in the genesis and early development of biomolecular crystallography. The work he himself did in the 1920s and 1930s laid much of the foundations that have been built on by others. And his legacy can also be seen through the achievements of those he collaborated with and inspired – an impressive list that includes Caspar, Fankuchen, Finch, Franklin, Hodgkin, Holmes, Kendrew, Klug and Perutz. You can count the Nobel Prizes yourself.

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## Bernal, material science and problem solving

## Paul Barnes, Birkbeck College, University of London, UK

I first met Sage, as Bernal had been known since his undergraduate days, in his house, in 1968, after joining his Liquids Group as a postdoctoral researcher. There is a record of that first meeting in Andrew Brown's biography (Brown, 2005), a book I recommend to anyone interested in Bernal. Though my meetings with Bernal were, due to his deteriorating health, intermittent over the three remaining years of his life, I drew inspiration from them and felt that I had touched greatness.

In my opinion he was one of the most powerful scientific thinkers of the 20th century, an intellectual giant within the vast field of structural science. He possessed a natural curiosity, endless enthusiasm, prodigious knowledge and brilliance with which he could explore the more remote regions of a subject outside the conventional boundaries of science. These are lofty claims so if any reader suspects exaggeration, a quick glance at the informed comments of eight Nobel laureates (see box) should surely set the record straight.

**Comments on Bernal by eight Nobel laureates (from Brown, 2005)**

Aaron Klug (1982)

"...was one of the most influential scientists of the 20th century."

James Watson (1962)

"...the extraordinary Irishman whose genius inspired the birth of molecular biology."

Francis Crick (1962)

"I regarded Bernal as a genius."

Linus Pauling (1954)

"...one of the greatest intellectuals of the 20th century."

Dorothy Hodgkin (1964)

"His greatest gift was his power to inspire others."

Hermann Muller (1946)

"One of the best, if not the best scientific mind in the world."

Lawrence Bragg (1915)

"He has been the pioneer who pushed the frontier forward."

John Kendrew (1962)

“It seems to me you’ve fathered 5 Nobel Prizes this year alone.”

In this article, I take a quick look at some relevant events during his formative years, his search for research positions that complemented his skills and the turning points in his development. I am intrigued to see whether we can learn anything new from his choice of research projects or the open manner in which he carried them out.

## The early days

While the boy Bernal showed clear signs of exceptional promise, his stellar potential only became fully recognised during his first year at Emmanuel College, Cambridge. There he would be able to sample the full Cambridge educational package comprising not only the necessary science components but also the essentials of a fully rounded academic life with activities such as political discussion, debating, networking and meeting influential thinkers. Bernal thrived in such an environment which in turn brought out the very best in him, accelerating his transition towards the later accomplished version that we would eventually recognize.

Bernal enjoyed listening to lectures that explored the dimensionality of a given topic and was clearly developing a flair for solving geometrical problems. In 1921 he set himself the challenging project of deriving the 230 crystallographic space groups using the quaternion mathematical representation. His wife to be, Eileen Sprague, assisted by typing the 80-page document which was then shortened to a 60-page version full of densely packed mathematics with no diagrams. It won him a shared first prize of £30 from Emmanuel College but the effort and time that he devoted to it may have cost him the first class honours he wanted so badly.

Bernal's next task was to find a post that fitted in with his areas of expertise in crystallography and that could provide him with a salary. The Cavendish could not help him. However, he did succeed in getting some temporary support from his old college, Emmanuel, which kept him and Eileen alive until he was able to join W. H. Bragg at the Royal Institution (RI).

## Bernal at the Royal Institution

Bernal’s stay at the RI from 1923 to 1927 comes across as a happy period despite a clumsy start. The laboratory exuded a friendly informal atmosphere which encouraged cooperation. In addition, Bernal and Eileen melded effortlessly into the London social and political scene which in turn was well-suited to the Bernals’ unconventional lifestyle. Bernal started slowly in the lab, but then excelled himself by making significant improvements to the X-ray equipment and production of crystallographic manuals and charts and teaching aids. However, these endeavours were all over-shadowed by one single Bernal breakthrough, the determination of the structure of graphite. This was hugely significant for several reasons:

Several big names in crystallography had tried to solve its structure, but all had failed. Bernal, however, demonstrated he had the patience to succeed and even constructed a home-made X-ray diffraction camera from various discarded objects (brass tubing, bicycle clips, alarm clock, bent nail – see Fig. 1). Needless to say, he had to perform all his calculations by hand!

There was a long-standing paradox regarding diamond and graphite. Both are made from carbon alone, yet diamond was the hardest material known to man while graphite is a very soft material. Bernal’s explanation resolved the paradox on the basis of the differences in structure: diamond has strong covalent bonds in a tetrahedral arrangement, thereby imparting strong interactions in all directions, whereas the graphite structure consists of hexagonal layers that can slide over each other like cards in a deck, resulting in softness and a tendency to smear. Obvious now to us as we accept that properties are related to structure (hence crystallography!), but this was not realised before Bernal explained it.



Figure 1. Bernal’s prototype rotation camera!

Following his four years at the RI, Bernal held a lectureship in Mineralogy and Crystallography at Cambridge until he moved to Birkbeck College in 1938, where he was to remain until his death.

## Bernal, Birkbeck and World War II

With the likelihood of war against Hitler’s Germany, Bernal felt compelled to voice his protest at the lack of preparation for mounting any form of response against an initial attack. He and Solly Zuckerman, a medic turned bio-scientist and anthropologist, became an effective duo in challenging the politicians’ official lines. Perhaps in line with US President Lyndon Johnson’s infamous remark about preferring his antagonists to be inside rather than outside his tent, Lord Louis Mountbatten in April 1942 invited Bernal to become his scientific advisor in Combined Operations. Bernal accepted and set about designing arrangements that would enable him to work on Combined Operations while remaining in contact with Birkbeck and crystallography *via* some kind of partial leave of absence. The complex arrangements, formal and informal, considerable deputising, delegation and shuffling of duties eventually brought about a successful arrangement, but not without heavy negotiations and quite a few headaches.

Bernal’s scientific interests were extremely broad, both with respect to science itself and the use to which science could be put. His Birkbeck department reflected this broad range, with research groups focussing on Structures of Biological Systems (see article by Christine Slingsby above), Materials Science Structures (initially headed by Helen Megaw, and which I took over following the retirement of Jim Jeffery), Structures of Liquids (see article by John Finney below) (the group which I originally joined) and Generalised Crystallography (in which Alan Mackay was a major collaborator).

This eclecticism is reflected not only in the strictly structural problems of, for example, graphite, bronze, metals and alloys, proteins, viruses and liquids, but also in the application of scientific methodology to ‘real’ issues exampled by many of the problems he was concerned with during the war.

## Some war-related projects of Bernal’s

Efficacy of gas masks with respect to poisonous gases

Action of incendiary bombs on building top floors

Design of effective shelters

Blast management

Physics of explosions

Biological effects of explosions

Resistance of concrete and other materials to explosions

Handling of unexploded bombs (Bernal risked his life many times by defusing real unexploded bombs)

Physics of air raids

Statistical analysis of bombing raids

Effects of breakwaters in sinking boats

Properties of pykrete

A synthetic iceberg as an airstrip for bomber operations

While much of what Bernal accomplished was not crystallographic – even within his wide interpretation of the subject – I give below an example of his non-crystallographic war work, followed by one war project which certainly did relate to materials and their functionality. Finally I say something about the work that involved me – the polywater story.

## (a) Statistical analysis of bombing raids

If there was one area of science above all others in which Bernal, as scientific advisor, had left his distinctive mark it would, I believe, be on his use of statistical analysis; one might call it Bernal's war-time legacy. Bernal took its implementation to a new level, in terms of both the surveying and the interpretation of the raw data. This was particularly so for assessing the effectiveness of bombing raids of both sides in causing both physical and psychological damage.

An assortment of volunteers and students, as well as professional statisticians, were used to establish relationships between damage, tonnage of bombs deployed, type of environment ascertained from observation, previous conflicts and accurate maps. Similar methodology was used to measure loss of morale using medicinal changes and increased alcohol consumption as indicators of population morale. Interestingly, the loss of morale in the affected population was generally less than expected. The rigour and neutrality behind the data from the statisticians were a breath of fresh air compared with the unverified and sometimes exaggerated claims made by those carrying out the ‘work’, such as flight crews. One issue of great irritation to the statisticians was the ignoring of their predictions, meaning the lives of pilots were sometimes being put in danger unnecessarily as they carried out bombing missions that statisticians had already shown to be of little value.

I close this section with an anti-intuitive statistical gem: more damage was inflicted on RAF aircraft parked on the ground than during aerial combat.

## (b) Habakkuk

Habakkuk was an Old Testament prophet who invited the heathen to look upon his unbelievable works in fear (“I will work a work in your days which ye will not believe, though it be told you”); Brown (2005) also suggests the name might have been chosen after a character in Voltaire’s *Candide* “who was, like Mountbatten, capable of anything”. So, it was perhaps appropriate that Habakkuk was used as the name for one of the most imaginative and ambitious projects of World War II: to produce man-made icebergs to serve as gigantic aircraft carriers. The reasons for Habakkuk were not obvious, nor was it obviously feasible. However, consider the following remarkable attributes:

The cost of the building materials is virtually zero.

The breakup cost is virtually zero since the main component simply melts away.

The project is ecologically 'green'.

The project is economically independent of how long the structure operates.

The structure is mobile with a range of thousands of miles, and so can be positioned to match war requirements.

The structure would need to last for only a few years, unlike a commercial equivalent.

With these points taken into account the project does appear attractive. The iceberg would be powered by a number of propelling units, and steered by a combination of those units and a gigantic rudder. Inside the structure would be refrigerator units supplementing the natural environmental chill, keeping it at sub-zero temperatures. To give some idea of the feasibility of staying cool, a scaled-down model was built that took two years to melt naturally.

The icebergs were to be built using pykrete – a mixture of 15% wood shavings and 85% ice. This material, named after its inventor Geoffrey Pyke, has remarkable strength, and melts very slowly. The plan was to make each of these icebergs the size of 25 conventional aircraft carriers.

If correctly constructed, pykrete blocks are stronger than concrete by weight and their thermal conductivity is lower than ordinary ice by an order of magnitude – the wood shavings behave as thermo-mechanical barriers. It was what we now know as a composite material; then, it was 40 years ahead of its time.

It was a controversial project. Some believed it was outlandish. Others including Churchill thought it had sufficient merits to be worth a try. The pykrete block was tested by Max Perutz (see article by Christine Slingsby above) – the same Perutz who went on to win with John Kendrew the Nobel Prize for his work on haemoglobin.

A somewhat amusing scene took place in a briefing session on board the Queen Mary (see Fig. 2). A block of pykrete and a block of ordinary ice were presented to an audience, which we believe included Churchill. One General Arnold attempted to break the ice block with an axe. He succeeded easily. When trying to break the pykrete block, not only did the axe fail to cause any damage, but also the resulting shock-wave travelling up Arnold's arm caused him to gasp in pain.

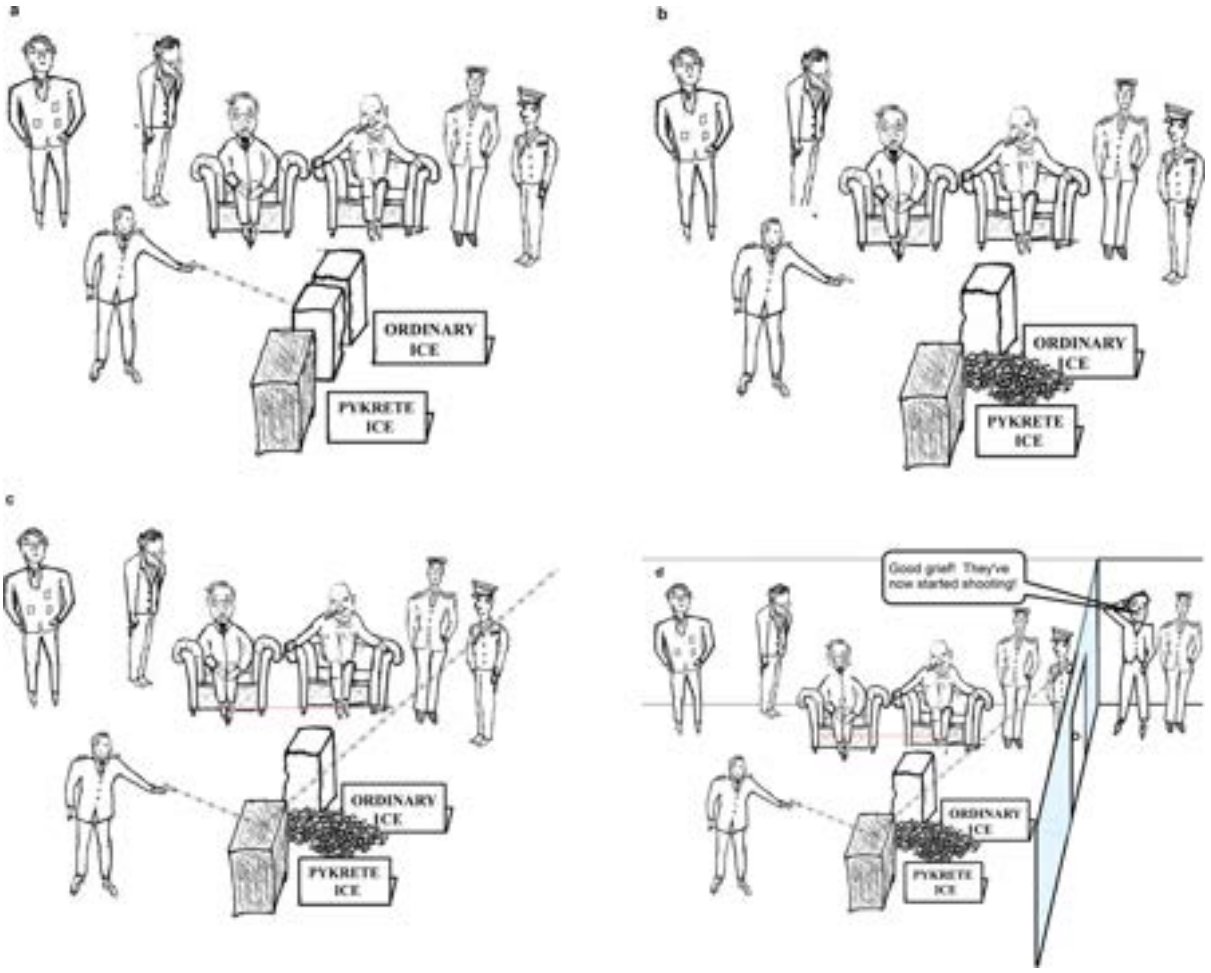



Figure 2. The pykrete test!

In typical dramatic style, Lord Mountbatten decided to repeat the experiment, but with a revolver. The ordinary ice shattered. In contrast, the pykrete block suffered little damage. Moreover, the bullet ricocheted off, narrowly missing one of the observing officers. It is said that pykrete was demonstrated by Churchill at dinner parties. He would present two apparently similar blocks of ice, but one of them would appear not to melt, or would melt only very slowly, presumably amazing his guests.

Despite these positives, the project was turned down after the requirements expanded. The required travelling range grew to 7,000 miles, the required lifetime expanded to 3–5 years, and the weight to be carried grew from that of fighter planes only to bombers and fighters. These increases in requirements, and the increase in distances that aircraft could fly without refuelling, combined to result in the project's cancellation.

## (c) Polywater

Polywater was the name given to an allegedly new structural form of water being worked on by the Russian group of Boris Deryaguin, and was being followed up by various international groups. I was aware of the work of Deryaguin mainly because of his classic publication on the DLVO theory of surface interactions. He had a high reputation in the scientific world, so when he made the surprising claims regarding the existence of a new from of water, he was given the benefit of the doubt. He claimed that water could be persuaded to condense onto confined silicate surfaces (*e.g.* glass or quartz capillaries) in a form that exhibited significant changes in its physical properties, such as boiling point, freezing point and density–temperature profile. Deryaguin's claims went further: he said that when ordinary and polywater droplets co-existed in proximity but with no physical contact, the polywater droplets could grow at the expense of the ‘ordinary’ water droplets. He was saying ordinary water could transform easily into polywater, implying polywater was a more stable form of $H_2O$.

When I was a postdoctoral researcher in Cambridge in 1968, I attended a lecture on polywater given by Deryaguin who was visiting the UK. I then wrote to Bernal at Birkbeck and asked if I could join his team which I had heard was to start research into polywater. Thus my four decade career at Birkbeck began. So in some ways Deryaguin was responsible for my association with Birkbeck. This is somewhat ironic, since he later tried to get me – and the rest of the team (including John Finney, current editor of *Crystallography News*) – sacked.

At the time I joined Birkbeck, the scientific community was full of polywater rumours and counter-rumours, and these extended into the wider academic world, industry and government. The issue started to feature in mainstream newspapers and TV. I was even interviewed about polywater, along with some of my Birkbeck colleagues, by the BBC.

Several *ad hoc* scientific meetings were arranged to address the issue. I remember one in particular in 1969, attended by about 40 physicists and chemists. The morning session consisted of everyone having an opportunity to describe their experiences making or using the alleged polywater. Reports fell into three categories:

I am trying hard but cannot make any polywater.

I can make polywater, and I am trying to measure some property (*e.g.* X-ray spectra, boiling point, freezing point).

Beware of the misleading effects of surface chemistry.

At the end of that meeting, various follow-up meetings were arranged to take place depending on the outcomes of further work. It may be that none ever took place...

Further communications on the subject became progressively quieter and less frequent when the Birkbeck group decided it was time we went public with the results of our work on the problem: that all such ‘sightings’ were nothing more than surface effects and contamination. And there was no real danger to our planet, as there would have been had ‘polywater’ indeed been a more stable form of $H_2O$ as claimed by some polywater adherents. We said polywater did not exist; what others had found were just ‘polypollutants’ (Bernal *et al.*, 1969; Barnes *et al.*, 1971).

After publication we waited for the response which we expected would be an onslaught of invective against anything found carrying the name of Birkbeck. In the event it was the opposite: near-silence except for a letter from Deryaguin to Bernal urging that the Birkbeck team should be sacked for incompetence! Communications on polywater then appeared to vanish altogether, its champions seeming to have given up and moved onto other projects. Polywater was dead, killed by a deafening silence.

I think Deryaguin had been reporting what he believed to be true, based on information given to him by his experimental colleagues. He himself was more of a theoretician. A few years after our paper, Deryaguin, to his credit, retracted his previous claims in print.

## A final comment

A question about Bernal: one long mission or multiple short-term solutions?

While waiting for a bus I sometimes play my mind game *Categorize* in which I try to place all members of a chosen group into the lowest possible number of categories. For example if the group comprised all famous crystallographers, dead or alive, known by me, I might try to sub-divide the members into two categories: those who are driven or inspired in their research as part of a life-long mission (*e.g.* seeking a cure for cancer; developing a new quantum theory) and those who are driven by the repeated thrill of solving many short-term problems (*e.g.* regularly solving the *Times* crossword). I can usually make an informed choice between the two but when the case of Bernal comes up, clarity disappears, as I uncover signs in his history that can apply to either of the two forms of inspiration. This ambiguity might in turn be related to another of Bernal’s renowned abilities, that of *multi-tasking*, evident long before the

modern use of the term had been invented.

So the answer to the above question is that Bernal was in both categories.

## Concluding remarks

In my opinion, Bernal was one of the greats, both for who he was and for what he did. His abilities included an amazing memory, a fertile imagination and a determination to make science work for the benefit of mankind.

I rate him as one of the four founding pillars of X-ray crystallography. Röntgen discovered X-rays; von Laue confirmed X-ray diffraction; the Braggs used X-rays to reveal crystal structure. For me, the fourth pillar is Bernal, since he linked the above discoveries and systematised X-ray crystallography.

A case could be made that Bernal should have been awarded whole or part of four Nobel Prizes: a share in the one awarded to Dorothy Hodgkin, a share in the one awarded to Max Perutz and John Kendrew, and two prizes that were never awarded; one for his founding work linking X-ray diffraction with the structure, properties and function of crystalline material, and another for his founding work on liquids, water and hydrogen bonding. As to why Bernal was never a Nobel laureate, the reasons offered range from political forces to being active in too many different areas of science. These reasons are still debated now, 50 years after his death.

One final remark. Over the last 18 months we have seen scientists around the world work together for the benefit of mankind as they delivered vaccines effective against COVID-19. The level of cooperation and the speed with which life-saving results have been delivered has been unprecedented and remarkable. This is exactly the role that Bernal wanted for science. If had lived to see this, he would have been very happy.

## Acknowledgements

This article is derived from my own experience plus a host of discussions and written sources too numerous to mention, with the exception of Andrew Brown’s excellent biography (Brown, 2005), which has been my main written source. Special thanks are due to Michael O’Callaghan, friend and colleague, who has been a huge help in the research and writing of this article.

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## Bernal, water and liquids

## John Finney, University College London, UK

Though Bernal threw out ideas for others to develop, he kept the development of his approach to liquids for himself. Initially, his interest was sparked by the biological relevance of water, saying “My interest in the subject... came about... through my biochemical interests, in that all living structures are mostly composed of water” (Bernal, 1964). This interest was to lead to a landmark paper in 1933 published in the first number of the *Journal of Chemical Physics* (Bernal & Fowler, 1933). Bearing in mind that this was only 20 years since X-rays were first diffracted from a crystal, and barely a decade since the first methods of working back from a diffraction pattern to a crystal structure were successful, to tackle the much more difficult problem of liquids, where there was inadequate understanding of how to interpret the broad diffraction patterns that liquids gave, was ambitious to say the least.

Arguing that the charge distribution on a water molecule was near-tetrahedral, Bernal proposed that as opposite charges would attract, the local molecular structure should be essentially tetrahedral (see Fig. 1).

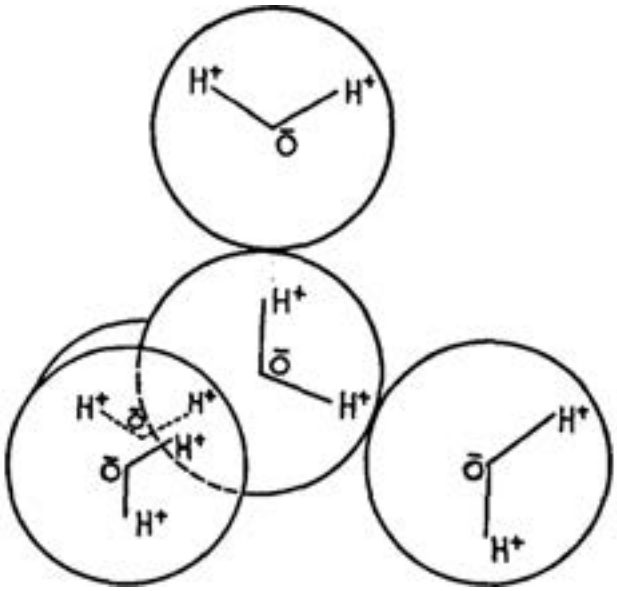



Figure 1. The ideal local tetrahedral arrangement of water molecules as first envisaged by Bernal in 1933. Two of the four molecules surrounding the central one are in the plane of the paper, while one is above and one is below.

Noting that silica ($SiO_2$) also forms similar local structures, he developed 'disordered' versions of two silica structures (quartz and tridymite) to fit the X-ray data. With this model, he was able to explain a wide range of the properties of water and ionic solutions, as listed in the short summary heading that paper shown in Fig. 2.

## Short Summary

ON the basis of the model of the water molecule derived from spectral and x-ray data and a proposed internal structure for water, the following properties of water and ionic solutions have been deduced quantitatively in good agreement with experiment.

(1) The crystal structure of ice.
(2) The x-ray diffraction curve for water.
(3) The total energy of water and ice.
(4) The degree of hydration of positive and negative ions in water.
(5) The heats of solutions of ions.
(6) The mobility of hydrogen and hydroxyl ions in water.

And the following inferred in a qualitative way.

(7) The density and density changes of water.
(8) The explanation of the unique position of water among molecular liquids.
(9) The dielectric properties of water and ice.
(10) The viscosities of dilute ionic solutions.
(11) The viscosities of concentrated acids.

Figure 2. The ‘Short summary’ of Bernal’s classic 1933 water paper.

It is also worth noting that the not-quite tetrahedral charge distribution he used is essentially the father of the effective pair potentials used today in simulations of water and aqueous systems.

In Birkbeck, he returned to the water problem in the 1950s, when he recognised that his 1933 approach “was, frankly, one of crystal structure, trying to picture water structure as that of a mixture of the analogous four co-ordinated structures of… quartz and tridymite”, and that “This was ultimately to prove rather a delusive approach, postulating a greater degree of order… in the liquid than actually exists there” (Bernal, 1964).

However, rather than return to the specific problem of water, he recognised that he first needed to understand the structures of simpler liquids. Theoretical approaches to the liquid state at the time treated liquids either as disordered (crystalline) solids (as Bernal had done in the 1933 water paper) or as dense gases. Though the disordered crystal approach was mathematically tractable and could yield correct densities, it assigned too much order to the liquid – the predicted entropy was too low. On the other hand, treating liquids as dense gases required unphysical mathematical approximations: though the entropy could come out OK, the densities that could be handled were too low to be representative of real condensed-phase liquids.

Bernal found both these approaches unsatisfactory. So instead he tried to find an approach that:

was a concrete picture of the structure (Bernal was a crystallographer and so naturally would want to visualise the atomic arrangement);

was consistent with Ockham’s razor (‘entities should not be multiplied beyond necessity’);

was homologous to that of the crystalline solid as well as radically different in kind;

had a general quality of homogeneity without the assumption of any special groups.

The most general hypothesis he came up with was to treat the liquid “as *homogeneous, coherent* and *essentially irregular* assemblages of molecules containing no crystalline regions” (Bernal, 1964). This concept he realised in the laboratory with assemblies of steel ball bearings, contrasting liquids as irregular *heaps* of molecules as against crystals as regular *piles*. Fig. 3 illustrates the differences!

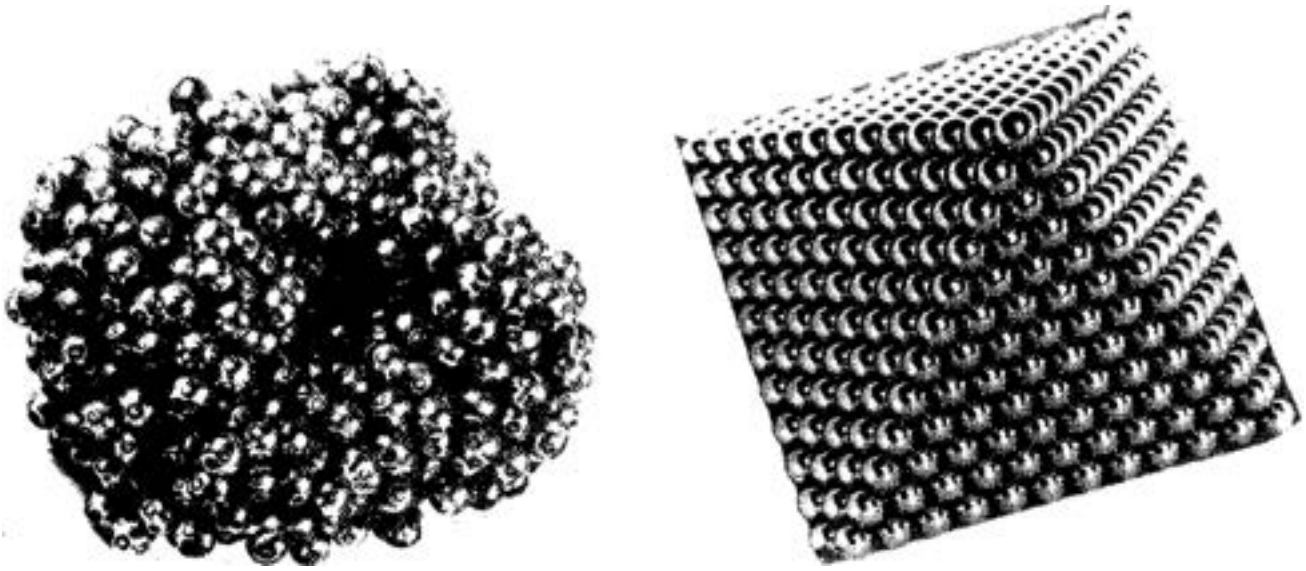


Figure 3. Bernal’s concept of the simple liquid as an irregular heap of molecules (left) compared with the regular pile of the crystal (right).

This structural approach was indeed radically different from that of other workers on the liquid problem – and indeed Bernal apologised to “the modern theoretical physicist for introducing such a simple way of

looking at things, but I believe on the whole that it is better to start with a model that has some resemblance to reality" (Bernal, 1959).

And indeed the model was successful for simple liquids such as those of the inert gases. It gave correct densities, explained density changes on melting, had the right degree of disorder and essentially predicted the observed X-ray scattering. In the late 1960s, it was also successful in explaining structures of amorphous metal alloys. The coordinates of a large laboratory model on which much of the later work was based continues to be requested and used for a variety of theoretical and practical purposes (Finney, 1970). And John Ziman, a key theoretician of liquids in the second half of the 20th century, commented that "This simple idea… is now seen to be the key to any qualitative or quantitative understanding of the physics of liquids" (Ziman, 1979). Similar comments were made in 1970 by John Rowlinson of Imperial College, one of the foremost theoretical chemists who has spent a lifetime working on liquids:

"*It has therefore been hard to admit that the form or even the existence of the attractive forces has little direct effect on the structure of a liquid, as described, for example, by the pair distribution function g(r). The recent realization of this truth has followed the extensive studies… of the properties of assemblies of hard spheres without attractive forces*" (Rowlinson, 1970).

Recent work is also suggesting that Bernal's model can explain the behaviour of liquids above the critical point, where the liquid/vapour coexistence line that vanishes at the critical point continues into the supercritical region as a line of maximum heat capacity (Finney & Woodcock, 2014).

So how did Bernal move from this 'irregular heap' model to the more complex water problem? Simply by recognising that a disordered non-crystalline arrangement could also be built up of molecules interacting in the essentially tetrahedral fashion of Fig. 1 to produce a random *network* of molecules, as against the random *packing* of the spherical molecules of simple liquids. Fig. 4 shows a ball and stick visualisation of a random network compared to the ordered crystalline arrangement of hexagonal ice.

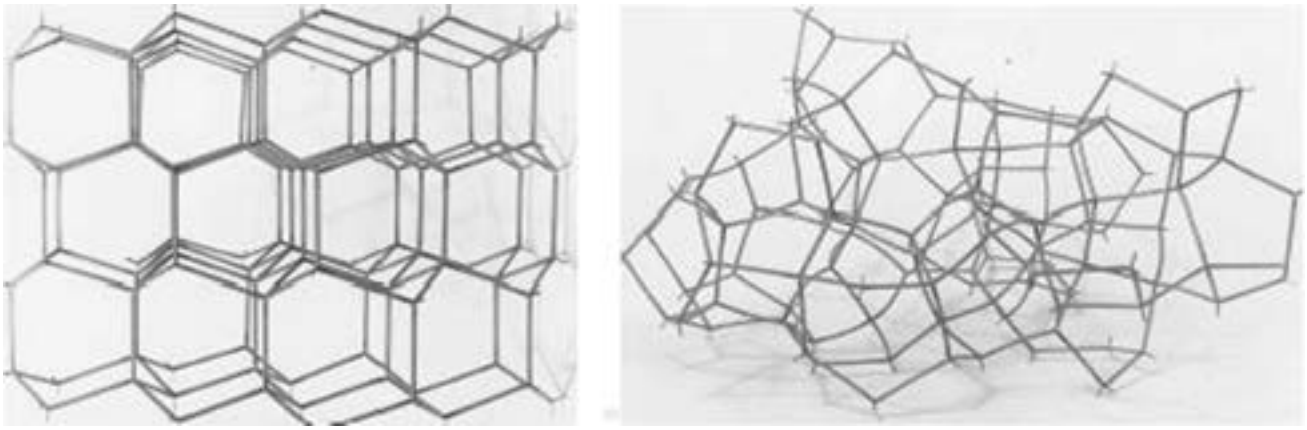


Figure 4. A 'spaghetti model' visualisation of (left) crystalline ice $I_h$, compared with (right) the 'random network' arrangement of liquid water.

So Bernal's final view of water (Finney, 2007, 2015) was that:

Water is essentially a 'random network' of water molecules.

Each molecule interacts with its neighbours in an approximately tetrahedral geometry.

Local coordination is ideally fourfold, but with some variation.

And it compared well with experimental results. It explains the main properties of water such as expansion on freezing, the temperature of maximum density and other so-called anomalies, the mobility of hydrogen, and structural changes with temperature and pressure. It is consistent with current state-of-the-art experimental and computational work, which demonstrates that Bernal's random network concept is essentially correct. And it has indeed helped us to understand water's biological role – the reason that Bernal started working on the problem in the first place.

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*For a fuller account of the development of Bernal's ideas on the structure of water.

**For an updated account of our understanding of water.

## Bernal's Irish connections and his enduring legacy at the Bernal Institute

**Vincent Casey, University of Limerick, Ireland**

John Desmond Bernal was born at the dawn of the 20th century (1901) and subsequently became one of the pioneering scientists associated with that century and the renaissance in science which it heralded. He addressed the big questions of his day. For instance: what is life and how did it originate? (Bernal, 1967). He was convinced that the answer lay within the structure of matter and the molecules where form and function would be closely entangled. Those he inspired were subsequently involved in the nascence of structural biology (see article by Christine Slingsby above) and were duly acknowledged for their pioneering work by the Nobel Committee. It is fair to assert that Bernal laid the groundwork that ultimately led to the extinction of the 'vital force' postulate in much the same way that Einstein's relativity theories banished the luminiferous ether. Bernal was very much aware of the important role played by science in history, as is clear from his four-volume book set of that title (Bernal, 1969), and of its critical role in shaping society and so was active in pioneering the use of scientific methods to solve major social (Cross & Price, 1988) and political (Bernal, 1958) problems of his time. For a comprehensive account of his life and work, Andrew Brown's biography is a most interesting and engaging read (Brown, 2005).

Bernal was born in Nenagh, Co. Tipperary, before Ireland won its independence. He was therefore a British citizen. Nevertheless, he was deeply 'imprinted' by his Irish childhood. He traced his roots to a mid-nineteenth century Sephardic Jewish migration. His grandfather, John Bernal, ran a successful auctioneering business in Limerick city and was also active in Limerick city politics. Around 1900, John Bernal's son, Samuel George Bernal (J. D. Bernal's father) bought a 147 acre country estate on the outskirts of Nenagh, complete with estate house, Brookwatson (National Inventory of Architectural Heritage, 2004). Considering those class-conscious times, this is likely to have placed the Bernals amongst the Anglo Irish landed gentry ruling class. The 1911 Irish census lists those staying at Brookwatson to include his parents, siblings, two relatives and four servants (Casey, 2021).

## Bernal commemoration

Until recently, Bernal was largely unknown in Ireland although better established, if neglected, among our colleagues in the UK. In recent times, Bernal's contributions to science and to society have been acknowledged and he has been commemorated in various ways both in the UK and in Ireland. On the occasion of the unveiling of a plaque (on 20 July 2005) in honour of J. D. Bernal by the National Committee for Science and Engineering Commemorative Plaques in his home town of Nenagh (see Fig. 1), Martin Bernal (J. D. Bernal's son) wrote: "Now, 35 years after his death, John Desmond Bernal has received this ultimate accolade". In 2006, the Munster branch of the Institute of Physics in Ireland organised a conference entitled 'John Desmond Bernal: Science and Society' with keynote speakers who knew and worked with Bernal (Finney, 2007; Mackay, 2007). Earlier, in 2001, an English Heritage blue plaque was erected at 44 Albert Street, London, where Bernal lived. And in 2013, after some years of planning, the magnificent Bernal Institute was opened by the Taoiseach (Irish Prime Minister), Mr Enda Kenny, on the University of Limerick riverside campus.




Figure 1. Pictured (photograph by V. Casey) in front of the science and engineering commemorative plaque to John Desmond Bernal are (left to right) Professor Alan Mackay, Dr Martin Bernal, Professor John Finney, Professor Helena Sheahan and Dr Andrew Brown.

## The Bernal Institute, University of Limerick

In October 2010, the University of Limerick, arising out of ongoing work with Atlantic Philanthropies, prepared an ambitious Science and Engineering development plan under the stewardship of Professor Kieran Hodnett. Coinciding with the publication of Andrew Brown's book (Brown, 2005), the University of Limerick (UL) had an ideal opportunity to celebrate the life and achievements of a local lad (Nenagh lies just 35 km from the University of Limerick campus), who was one of the most influential scientists of the 20th century. This was the genesis of the Bernal Project, and preliminary planning for the Bernal Institute (BI) started in early 2011.

The Bernal Project ultimately resulted in a €100+ million investment in people and buildings to enable spatial integration of Science and Engineering disciplines at UL. The vision was to support synergies that result from close multidisciplinary collaborations and strong links with an Irish and international industrial base forged through UL's 'cooperative education' programme, an industry–academia education partnership. The overall project objective is to address societal challenges that require disruptive new technologies enabled by materials science and engineering.

The preliminary proposal envisaged the recruitment of ten Bernal Professors to act as international leaders in their fields and to develop the resources, equipment and infrastructure needed for the successful pursuit of both fundamental and applied research. Bernal was an extraordinarily generous mentor to younger colleagues. In appointing Bernal Professors UL therefore sought out candidates who would exhibit this same generous spirit of mentorship. A preliminary schedule of accommodation for the new building envisioned ten specialised laboratory suites in a 6,000 $m^2$ building, with associated office and write-up space, seminar rooms, lecture theatres and social space. The total cost of the project was projected to be €52 million. The BI project proposal was accepted for funding and an activity whirlwind of building design and recruitment followed with a three year timeline.

In September 2013, prior to the official launch of the Bernal Project, the President of the University of Limerick hosted a dinner for some family members and friends of Professor Bernal. The attendees included Gay-Caroline Bernal (Bernal's granddaughter) who regaled the company with her childhood memories of visiting her grandfather in his offices in Birkbeck College, London, and playing with his modelling materials including, we suspect, those used in models to support his theory of liquids (see John Finney's article above). Friends present included John Finney, UCL, a former external examiner to UL's applied physics degree and a previous student of Bernal's. Also present was R. J. Polle (Jack), just retired from active service as a vet having served the Brookwatson farm for over 70 years. He remembered meeting Bernal in 1950 when even at that stage, Bernal predicted how global warming caused by $CO_2$ emissions would lead to climate change.

By the time the Bernal Project was officially launched in October 2013 by 'An Taoiseach', seven of the ten Bernal Chairs had been filled (Fig. 2) and the building aspect of the project was concluded in 2016 (Fig. 3). Three additional chairs have subsequently been filled in composite materials, process engineering and structured materials.



Figure 2. Official launch of the Bernal Institute: (left to right) Professor Edmond Magner, Professor Don Barry (President UL), Professor Bartek Glowacki (Bernal Chair in Energy), Professor Gavin Walker (Bernal Chair in Pharma Powder Engineering), Professor Ursel Bangert (Bernal Chair in Microscopy and Imaging), An Taoiseach Enda Kenny TD, Professor Jacques Huyghe (Bernal Chair), Jon O'Halloran (BI Operations Manager), Professor Michael Zaworotko (Bernal Chair in Crystal Engineering), Professor Harry Van den Akker (Bernal Chair in Fluid Mechanics), Mary Shire (Vice President Research, UL). Photograph courtesy of UL.



Figure 3. The sun about to rise over the Penrose tiling patterns that decorate ‘The Cube’, the large lecture hall in the Analog Devices Building of the Bernal Institute. Photograph courtesy of M. J. Zaworotko.

## Bernal’s enduring science legacy – the Bernal Institute

Bernal had many and varied interests, making key scientific contributions in his own time. He remains highly influential in many scientific domains. His early work on graphite (Bernal, 1924), for instance, laid the foundation for the recent burgeoning research related to graphene (Sandhya *et al.*, 2021). For a comprehensive bibliography of Bernal’s writings see Dorothy Hodgkin’s Royal Society Biographical Memoir (Hodgkin, 1980).

## Some ongoing Bernal Institute projects

Under the umbrella of ‘structures matter’, the BI has developed five research clusters that span the full range of matter length scales and materials: (i) biomaterials, (ii) molecular and nanomaterials, (iii) 3D electron diffraction, (iv) composite materials, (v) process engineering. Each of these clusters engages in research projects targeted at societal challenges related to sustainability and/or human health. The clusters are supported by world-class imaging facilities and molecular modelling research groups. Some highlights of the research in these clusters, several of which involve the study of crystals including crystal growth, are summarised below.

## (i) Biomaterials

The Biomaterials Research Cluster focuses on structured biological materials, and systems based on molecular and cellular building blocks that range from molecular ensembles to cells/cell aggregates, tissues and organs, and the interface with non-biological materials such as implants, drugs and their combination. Professor Tewfik Soulimane studies how diphenylalanine (FF) demonstrates a robust ability to self-assemble at the nanoscale forming a variety of structures ranging from nanospheres to nano- and microtubes resulting in outstanding functional properties including pyro- and piezoelectricity. FF nanotubes mimic the structure of β-amyloid fibrils characteristic of Alzheimer's disease and thus can serve as a model material in biology and medicine. Water confined in self-assembled FF peptides displays the properties of both bulk and nanoconfined water – confined water is critical to understanding the role of water in molecular biology where almost all water can be considered as confined water and/or mixed with ions and other molecules in aqueous solutions. Professors Tofail Syed and Damien Thompson study how piezoelectricity can be controlled through the self-assembly of amino acids. Piezoelectricity has attracted recent interest due to its manifestation in biological molecules such as synthetic polypeptides or amino acid crystals, including γ-glycine. It has also been demonstrated in bone, collagen, elastin and the synthetic bone mineral hydroxyapatite. Piezoelectric coefficients exhibited by these biological materials are generally low, limiting technological applications. Research guided by quantum mechanical calculations has changed this situation, with the demonstration that the amino acid crystal β-glycine possesses a piezoelectric coefficient similar in magnitude to materials such as barium titanate or lead zirconate titanate that are in commercial use.

## (ii) Molecular and nanomaterials

The expansion of computational power, the development of better/faster characterization methods and the evolution of self-assembly and supramolecular chemistry have enabled the dream of ‘materials by design’ to approach fruition. This ‘materials design revolution’ is poised to profoundly influence society by impacting, amongst other matters, energy sustainability and drug development. Simply put, by finding the right chemistry, materials scientists and engineers can gain the level of power that architects have when they design new buildings. Crystalline materials by design – crystal engineering – is one of themes of this cluster.



Figure 4. ‘Skyscrapers’ – an image of crystals of coordination polymers taken by Bernal Institute post-graduate student Shi-Qiang Wang. This image was awarded a prize in the Crystal in Art Virtual Competition 2020 of the British Association of Crystal Growth (BACG). Photograph courtesy of M. J. Zaworotko.

Bernal Chair of Crystal Engineering Professor Michael Zaworotko leads the crystal engineering research group that he established upon joining UL in 2013. Crystal engineering is enabled by one of Bernal’s legacies: the Cambridge Structural Database, CSD. The CSD was proposed by Bernal (Bernal, 1960*a*,*b*) and constitutes perhaps the first scientific database. There are two main areas of study in the crystal engineering group: advanced porous materials and multi-component pharmaceutical materials. Recent highlights include articles in *Science* in 2016 and 2019 that dealt with improving the energy efficiency of ethylene purification, the largest volume product of the petrochemical industry. Dr Matteo Lusi leads a research group that studies mixed crystals – solid solutions of two or more molecular compounds that are characterized by structural disorder that enables variation of stoichiometry in continuum. Often such variation results in modulation of structural and physicochemical properties which, in turn, facilitates fine-tuning of bulk properties. Such behaviour is particularly important in pharmaceutical, agrichemical and optoelectronic materials. Professor Kevin Ryan leads a team that studies colloidal nanocrystals with particular emphasis on semiconductor nanorods and their device scale assembly by directed (electric fields) or non-directed approaches for scalable applications in photovoltaics. The team has pioneered routes whereby the nanorods can be assembled from solution such that each rod is vertically aligned and close-packed. A second work programme addresses low-cost solution synthesis of silicon and germanium nanowires by seeded and non-seeded strategies, targeted particularly towards generating wires in high yield. Control over nanowire length for the formation of nanorods was achieved by a modification of this synthesis protocol.

## (iii) Timepix and 3D electron diffraction

Dr Andy Stewart leads a 3D Electron Diffraction group. Structure determination of nanocrystals of organic pharmaceutical compounds by 3D electron diffraction (3DED) at room temperature is possible using the Timepix direct electron detector with increased quantum detection efficiency (Genderen *et al.*, 2016). A convolution of the electron density and the nuclear charge makes it possible to determine the position of hydrogen atoms in structures without the need to grow millimetre-sized crystals for neutron diffraction studies. It is hoped that the ability to investigate nano-sized crystals will answer the question of why so few crystal polymorphs are currently observed despite the many possible based on crystal structure predictions. Is it a question of kinetics – the crystals cannot be grown to sufficiently large size or in sufficient numbers to be used in X-ray studies? It is hoped that the fast developing field of 3DED will help reveal the deeper crystal physics behind this question.

Until recently, structure determination of organic compounds by transmission electron microscopy required data collection at liquid-nitrogen temperatures to reduce the effects of radiation damage. The novel Timepix detector combines a high dynamic range with a very high signal-to-noise ratio and single-electron sensitivity, removing the need to freeze the specimens to obtain data good enough to enable *ab initio* phasing of beam-sensitive organic compounds. Low-dose electron diffraction data ($\sim$0.013 $e^{-}$ $Å^{-2}$ $s^{-1}$) collected at room temperature were of sufficient quality for structure determination using software developed for X-ray crystallography which includes the appropriate electron scattering factors.

The Stewart group showed that 3DED coupled with a sensitive Timepix detector allows fast and efficient three-dimensional crystal structure analysis of organic pharmaceutical compounds at room temperature. The technique will allow higher-throughput examination of nanometre-sized samples in a transmission electron microscope complementing standard single-crystal and powder X-ray diffraction. The recent advances in 3DED have led to an interesting new question for crystallography. Electron microscopes are sensitive enough to observe the transition from nucleation to particles and onto crystals during the growth process. This raises the question as to how many unit cells are sufficient to produce an observable crystal, the structure of which can be determined by 3DED methods. This question is particularly pertinent for crystal growth and phase-change materials. Currently, the International Union of Crystallography's definition of a crystal does not provide a quantitative number to declare an object as a crystal. The Stewart group has been working on this question [a talk on this was given at the 2021 BCA-BACG Spring Meeting (*Crystallography News*, 2021)] and will be shortly publishing a paper suggesting a methodology to quantitatively define an object as crystalline.

## (iv) Process engineering

Professor Gavin Walker leads the Process Engineering Cluster, which is aligned to UN Sustainable Development Goals in Health, Energy and the Environment/Sustainability. Improving population health and well-being is critically dependent on the development of new biopharma solutions which, in order to complete the drug approval process and have maximum impact, must be amenable to scalable processing and manufacture, this often demanding innovative processing. Ensuring access to affordable, reliable, sustainable and modern energy for all will require a substantial increase in the share of renewable energy in the global energy mix. Advanced energy storage solutions are sought and can help here through C-storage/conversion but so too can process synthesis and integration. Fostering and supporting responsible consumption and production ensuring sustainable consumption and production patterns can reduce waste generation through prevention, reduction, recycling and reuse. Reduced solvent waste can be achieved through solvent-free processing of advanced materials and through multiphase, bio- and hybrid processing.

### (v) Composite materials

Professor Paul Weaver leads this cluster, a major aim of which is to help develop new industry in Ireland based on new intellectual property. To meet this aim the cluster focuses on two main application areas: renewable energy and sustainable aircraft transport. Research involves a combination of materials development, manufacturing technology and design methods where each will be pursued in balance and harmony with one other so that equi-fidelity contributions to technology are made. In terms of renewable energy, Dr Maurice Collins leads a research programme pursuing the development of new carbon fibre materials from sustainable resources (lignin). Once large-scale production of these materials is achieved, composite materials will be developed using thermoplastic matrices from natural, sustainable supplies. Research in manufacturing and design methods both contribute in a complementary manner to application areas such as renewable energy and sustainable transport. A research focus of the cluster is on the development of methods of manufacturing composites that will help reduce the cost of energy for wind turbine blades and lead to cheaper, better-performing aircraft (less fuel burn). Design methods will focus on advanced stress analysis development and subsequent tool development that exploits our new combination of materials and manufacturing technology.

## Concluding remarks

The future focus of the Bernal Institute is likely to include, in addition to current themes, areas such as sustainability in food, energy and water, three areas of application where the Institute has growing momentum. It seems inevitable that the structure–function relationships of crystalline and non-crystalline materials that inspired Bernal will continue to guide its direction. We are now at the stage where crystal engineering can have major positive impact on our future. Engineered crystals that can efficiently separate mixed gases and abstract specific molecules from the atmosphere have been demonstrated. An application of this technology with obvious world-saving potential is one-step separation of $CO_2$ from the air at ambient temperature and pressure to combat global warming. Another crystal engineering system currently under trial could help solve the developing world’s need for potable water by direct extraction from the atmosphere [these last two projects were described in Michael Zaworotko’s Plenary talk at the 2021 BCA-BACG Spring Meeting (*Crystallography News*, 2021)].

The field of crystallography has expanded enormously since the 1920s when Bernal commenced his pioneering research at Cambridge elucidating the structure of life’s molecules using XRD. A century later, it is clear that crystals and the science of crystallography will continue to expand the social function of science to create a more equitable and harmonious society. With what has already been achieved and the exciting future prospects, we may, with good reason, hope that crystals are going to play a major role in saving the world.

## Acknowledgements

I would like to acknowledge contributions to this article by Professor Kieran Hodnett, Professor Edmond Magner, Professor Michael Zaworotko, Dr Matteo Lusi, Dr Tofail Syed, Professor Tewfik Soulimane and Dr Andrew Stewart, all of whom gave freely of their time and support. Jon O’Halloran, BI Operations Manager, has also been most supportive.

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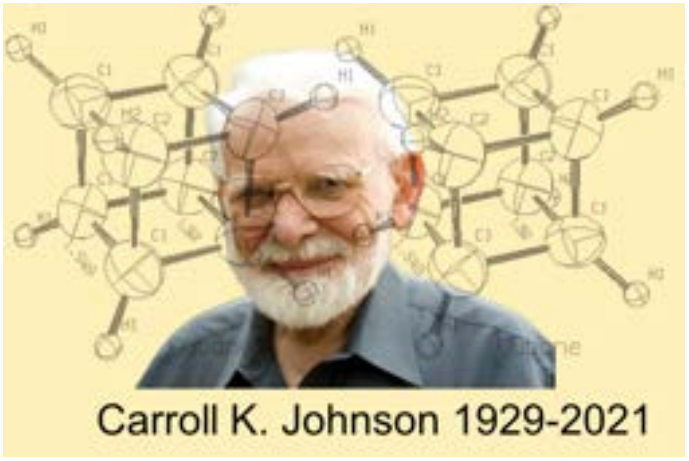
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OBITUARY

# Carroll K. Johnson (1929–2021)




Bryan Chakoumakos

Carroll Johnson's professional career adhered to the postulate that one should change directions every five years to avoid stagnation. His career profile, in approximately five-year segments as he described himself, starting in 1955, was as follows: (1) graduate school (MIT-PhD): biophysics & fiber diffraction theory, (2) postdoctoral positions: X-ray crystallography (Institute for Cancer Research, Philadelphia) & neutron diffraction (Oak Ridge National Laboratory), (3) staff member at ORNL until retirement in 1996: thermal motion & computer graphics, (4) crystal physics, (5) artificial intelligence concepts, (6) artificial intelligence applications, (7) machine vision engineering & project management, and (8) crystallographic topology & neutron diffraction. Carroll remained very active in retirement. His post-retirement activities involved "crystallographic topology, personal computing and enjoying life." He personally noted selected professional activities in his career: 1975–1976 sabbatical, studying artificial intelligence at the Computer Science Department, Stanford University, 1977 President of the American Crystallographic Association (ACA), 1981 studying more artificial intelligence at the Naval Research Laboratory, 1996 Chairing the ACA General Interest Group, and 1997 receiving the ACA Buerger Award.

He was a native of Colorado and started a pre-med program in 1947 at the University of Colorado, which was put on hold by four years in the United States Marine Corps, which included a year in the Korean war. Afterward, he completed a BS degree in Animal Nutrition and Physiology (1955) from Colorado State University. He went on to complete a PhD degree in Biophysics (1959) at MIT, doing his thesis on polypeptide packing studied by X-ray diffraction. After that, he did a postdoc (1959–1962) at the Institute for Cancer Research (now the Fox Chase Cancer Center), Philadelphia, with Lindo Patterson, whose group at that time included Jenny Glusker, Dick van der Helm, Max Taylor, Eric Gabe and Jean Minkin. During his postdoctoral work in Philadelphia, he began working on the computer program for plotting atomic thermal ellipsoids, which he later finished at ORNL.



Carroll started at ORNL in 1962, beginning as a postdoctoral assistant working on neutron diffraction in Henri Levy's group. After converting to a staff scientist, he spent his entire professional career in the Chemistry Division at ORNL, retiring in 1996. Over the years at ORNL, his immediate colleagues and his group's mission changed a lot. In the early days, he worked with Henri Levy, Bill Busing and George Brown. Later he worked with Michael Burnett on many programming applications. He was an independent thinker, mathematically inclined, and pursued projects always at the forefront and often beyond current thinking. Carroll Johnson's scientific success grew markedly with the publication of his most celebrated computer program, *ORTEP* (Oak Ridge Thermal-Ellipsoid Plot Program). This rapidly became a favorite of crystallographers and protein crystallographers to make illustrations of crystal structures for conference presentations and publications. *ORTEP* was first released in an ORNL Technical Report in 1965; see historical note https://www.umass.edu/molvis/francoeur/ortep/ortepnews.html. A key strength of *ORTEP* was its capacity to generate stereoscopic images automatically. *ORTEP* 2 was released in 1976 and *ORTEP* 3 in 1996. The latter is still available from the official *ORTEP* website, https://ornl-ndav.github.io/ortep/ortep.html. *ORTEP* is one of the most cited and miscited publications in crystallography, with over 30,000 citations. *ORTEP* even is commonly used as a word for a crystal structure drawing with atomic displacement ellipsoids, whether it was made with *ORTEP* or not. A Google search on "ORTEP" returns 798,000 hits. Atomic displacement ellipsoid drawing options are now standard in all the major crystal and molecular structure drawing programs. Carroll was an expert on thermal motion analysis as studied by diffraction and sought better and new ways to use this aspect of crystal structure analysis.

Bill Busing, Henri Levy, Hal Smith and Pete Peterson had built and were operating a three-circle neutron diffractometer at the Oak Ridge Research Reactor for several years working on chemical crystallography prior to Carroll Johnson's arrival at ORNL. Data collection by that diffractometer was controlled by a computer program run on the ORACLE (an early main-frame computer at ORNL), which prepared a paper punch control tape conveying the angle settings to the motors. The intensities were also recorded on

paper punch tape. When minicomputers became available in 1965, Busing and Levy programmed a PDP-5 computer to control a four-circle Picker diffractometer, first as an X-ray instrument and then as a neutron instrument at ORNL's High Flux Isotope Reactor (HFIR), which had just started operating. Seminal crystal structural studies of hydrogen-containing compounds were done with the HFIR diffractometer. Still, the science it could do was limited because no low-temperature sample cooling device was available on the instrument. The HFIR four-circle diffractometer was operated only intermittently through the 1980s. After a 3+ year safety review shutdown of HFIR ending in 1990, Carroll Johnson and Michael Burnett upgraded the neutron diffractometer at HFIR, with a new Huber goniometer, a low-temperature sample stage (5–300K), and a new 7-pixel array detector. I was responsible for the powder diffractometer at HFIR at that time but was interested in doing structural studies with single crystals as well, so I worked with Carroll to learn as much as I could about the operation of his single-crystal diffractometer. The interest in single-crystal neutron diffraction studies by the Chemistry Division kept dwindling with each retirement and passing of Levy's old group members. Carroll had been the youngster in that group when he started, but now he was ready for a change: retirement was next, and it was at that time that he transferred the HFIR four-circle diffractometer to me in the neutron scattering group. Since then, we have upgraded that instrument multiple times, and it has grown with the help of a new younger generation of scientists hired in the ORNL Neutron Scattering Division to be extremely productive doing research in materials physics and chemistry. I thank Carroll for setting us on that course.

The American Crystallographic Association was his professional society home. He began attending ACA meetings in 1964 and, with few exceptions, attended all their meetings until he retired, and some after that. The ACA was an extended family for him, and he often planned family vacations around the annual meetings, bringing along Carol, his wife, and his children, which numbered five in the end. He gave back to the ACA by presenting many talks and posters, serving on the Crystallographic Computing Committee in 1965–1967, then as President-elect in 1976, President in 1977, Past-President in 1978, and Chairing the General Interest Special Interest Group 1994–1996.

Carroll Johnson was married to Carol for 69 years and had five children, ten grandchildren and seven great-grandchildren. His family especially enjoyed how his curiosity and inquisitiveness were not limited to his work and touched all aspects of his life.

OBITUARY

# John Sinclair Reid (1942–2021)




Moreton Moore

Crystallography was one of the many and varied subjects that John Reid taught at the Natural Philosophy (alias Physics) Department of the ancient University of Aberdeen, UK. He was born in wartime Chorleywood (North London) in 1942, and after his early schooling, he was sent as a boarder to Robert Gordon's College, Aberdeen. Aberdeen was where he spent the rest of his life, graduating three times at the University and becoming, in turn, Assistant Lecturer, Lecturer and Senior Lecturer in Natural Philosophy. He had an unusual combination of degrees and an impressive collection of professional fellowships: BSc, MLitt, PhD, FRAS, FRMetS, FInstP, CPhys. He had a keen interest in astronomy, meteorology and the history of physics, and he was a founder member of the Scientific Instrument Society. He was Curator of the Natural Philosophy Collection of Historic Scientific Instruments in Aberdeen. It was while demonstrating practical physics in one of the teaching laboratories that he met the undergraduate who later became his wife. Their joint interests in astronomy and navigation were useful practical skills in pursuing their love of sailing.

John's father had survived the World War I trenches of Belgium and France, and was later a business accountant, who loved carpentry. His mother had been a secretary. They lived in a house with plenty of books, which included volumes in French, German, Italian, Gaelic and Russian. It was aged 10 that he made his first valve radio and while at Robert Gordon's College, John made crystal sets (wireless receivers); he indulged in photography; and he constructed and flew model aircraft. He excelled at mathematics, science and geography. When his father retired from his London job, the whole family settled in Aberdeen. John became an active member of the Aberdeen Amateur Radio Society.



John’s PhD topic concerned the analysis of atomic motions in crystals derived from observations of diffuse X-ray scattering. This introduced him to a lifelong interest in diffuse scattering and the information that could be extracted from it. His initial calculations were performed in Algol 60 on an Elliott 803 computer, starting with the 1-phonon scattering in sodium chloride (NaCl). Later, he also used a triple-axis spectrometer at the PLUTO reactor at Harwell to study neutron scattering from huge sodium bromide (NaBr) crystals, which had been grown by ‘Dai’ Jones at Aberdeen. Together with Dr John Pirie and others, he developed the equipment necessary for a comprehensive study of energy-dispersive diffuse X-ray scattering using synchrotron radiation at the Daresbury Laboratory. For over 30 years he was a regular attendee at the triennial International Union of Crystallography Congresses. He published 16 papers in *Acta Crystallographica* and the *Journal of Applied Crystallography*, all related to diffuse scattering. His 1995 paper, “The analytical calculation of absorption in multifaceted crystals”, has over 1000 citations (and counting!) on researchgate.net.

John retired from the University of Aberdeen in 2007, but continued his lifelong love of physics and the history of science. He continued to curate the nationally recognised Historical Scientific Instruments Collection at Marischal College, ran the Aberdeen Mechanical Society and was an active member of the Scientific Instruments Society (of which he had been a founder member, in 1983).

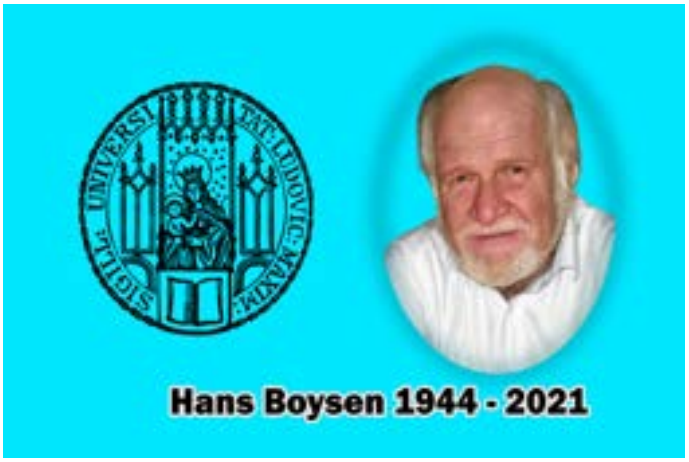
In his latter days, John bravely fought two cancers. He will be greatly missed by his family and friends, and by all those whom he taught at Aberdeen over a period of 50 years.

Moreton Moore is Emeritus Professor of Physics at Royal Holloway University of London, UK.

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OBITUARY

# Hans Boysen (1944–2021)




Wolfgang Schmahl, SoHyun Park, Anatoliy Senyshyn, Markus Hölzel, Martin Meven, Peter Gille, Guntram Jordan, Friedrich Frey, Wolfgang Moritz, Natalija van Well

On 19 March 2021, a few weeks before his 77th birthday, Dr Hans Boysen passed away. He was an important pioneer of crystallographic research on disordered structures and ferroics with neutrons in Germany.

Born in Karlsbad in 1944, Dr Boysen attended school in Flintbek and Kiel, where he also began to study physics. After completing his intermediate diploma, he moved to the Ludwig Maximilians University in Munich (LMU), where he graduated with a diploma in physics. He then did his doctorate under Professor Heinz Jagodzinski at the Institute of Crystallography and Mineralogy of the LMU and stayed there as a lecturer and research scientist until his well-deserved retirement.

Having worked on crystallographic investigations with ionizing radiation sources, he was already involved in the development and operation of neutron diffractometers at the first research reactor in Munich (FRM), the so-called “atomic egg” of Garching. Later, at the Heinz Maier-Leibnitz Research Neutron Source (FRM II), Hans Boysen made a lasting impact on crystallographic research. He was particularly committed to the planning, construction and use of the SPODI powder diffractometer at FRM II. His fundamental methodological contributions here included the implementation of focusing optics with monochromators and the efficient, high-resolution use of Debye–Scherrer cones. Besides, he actively advised on the design of the RESI single-crystal diffractometer at FRM II. Dr Boysen was also successful in developing sample environments, for instance to collect diffraction data at extremely high temperatures above 2000°C.

Dr Boysen's contributions to structural research in geosciences and materials sciences focused on the analysis of the thermal evolution of order–disorder phenomena and structural phase transformations using X-ray and neutron diffraction. Among these, highly noteworthy examples are the characterization of disordered structures of minerals and functional materials, such as ferroelectrics, and oxygen- and lithium-ionic conductors. His scientific records with the evaluation methods are of great current interest. His works form a lasting foundation on which we stand today.

Hans Boysen's commitment went far beyond pure research. Because of his broad expertise, he was active in national and international committees. He was also intensively involved in the training and further education of young people, and supervised numerous student, diploma and doctoral theses.

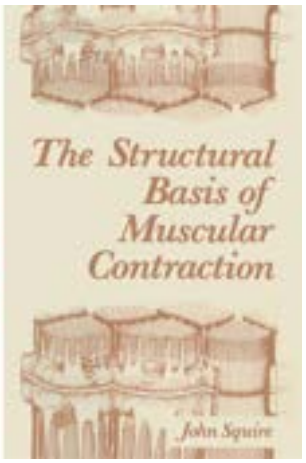
For his outstanding achievements in the development of scattering methods for structural research and in the field of crystallography on disordered systems, Hans Boysen, together with his long-standing colleague Professor Dr Friedrich Frey, received the Will Kleber commemorative coin of the German Society for Crystallography (DGK) in 2010.

Discussions with Dr Boysen were highly appreciated for his endless curiosity and interests in natural sciences. He was beloved for his profound and sympathetic nature by his colleagues and all his students. We miss him sorely.

OBITUARY

# Remembering John Squire (1945–2021): His achievements and legacy






Ed Morris, Pradeep Luther

It was with great sadness that we learnt of the death of John Squire, Professor of Structural Biophysics in the University of London, UK, and Visiting Professor at Imperial College, London, UK.

Professor John Michael Squire (BSc, PhD, CPhys, FInstP, CBiol, FSB) was internationally renowned for his research on the structural basis of muscle contraction. He joined Imperial College in 1972 where he established the Biopolymer Group within the Department of Metallurgy and Materials Science. He subsequently moved to the newly formed Biophysics Section in the Department of Physics in 1984, becoming a Reader and then Professor in 1995. In 1999 he became Head of the Biological Structure and Function Section within the newly established Division of Biomedical Sciences located in the Sir Alexander Fleming Building. After his official retirement in 2006 he maintained a strong and active interest in research through his ongoing affiliations with Imperial College and the University of Bristol, publishing his most recent paper in 2020.

John passed away on 31 January 2021, a victim of the deadly SARS-CoV-2 virus which has been causing havoc around the world since the start of 2020. John is survived by his wife, Melanie, their four daughters and ten grandchildren.

Over a research career of 51 years John established himself as a leader in the field of muscle research. Early in his career he introduced many of the key concepts in the field including the steric blocking model of thin filament regulation (with David Parry) and a general model for assembly of myosin filaments from the constituent myosin molecules (published in 1971, but only fully verified in recent years with the aid of new technology). Subsequently, using a combination of X-ray fibre diffraction and electron microscopy, he identified the particular structural features of bony fish muscle which make this an especially suitable system with which to observe the individual molecular steps by which muscles contract. Over a number of years, he proceeded to exploit this system to build up a detailed structural model of the mechanism of muscle contraction which he termed Muscle the Movie.

John’s research on muscle contraction and related areas such as the blood vessel glycocalyx formed the basis of a substantial body of more than 250 publications. Alongside his primary research papers he also wrote many reviews and book chapters. Of particular note is his 700-page book: *The Structural Basis of Muscle Contraction*, which was published in 1981 but still remains one of the best and most comprehensive introductions to the field.

John was also well known for his leadership within the scientific community. He founded and chaired the Collaborative Computational Project 13 (CCP13) which developed and supported software for the analysis

of X-ray fibre diffraction data. He set up and organised scientific meetings including the Imperial Muscle Initiative meetings and, together with David Parry, a series of international workshops on Coiled-Coils, Collagen and Co-proteins which were held every four years in Alpbach, Austria. John was a member of the British Crystallography Association (BCA), and a member of the BCA Council and Editor of *Crystallography News* from 1987 to 1995.

Throughout his time at Imperial College, John ran a thriving research group drawn from a wide range of nationalities and cultures. Within the group he fostered a supportive environment frequently leading to enduring friendships. The group and family members were regularly invited round to John and Melanie's house in Egham to a series of parties in their beautiful garden at which somehow the sun always seemed to shine. Many of his former students and postdocs who went on to build their own careers in science continued to work with him on collaborative projects. Additionally, he maintained a substantial network of collaborations with scientists all over the world. That so many were happy to continue to do science with John in this way serves as a testament both to the esteem in which he was held scientifically and to the fact that he was a pleasure to work with.

Ed Morris has recently retired from the Institute of Cancer Research, London, UK, and Pradeep Luther is at Imperial College London, UK.

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OBITUARY

# John C. H. Spence (1946–2021)



Henry N. Chapman, S. Samar Hasnain, Uwe Weierstall

John C. H. Spence ForMemRS, Regent's Professor and the Richard Snell Professor of Physics at Arizona State University (ASU), who continuously blazed thrilling new trails in the science and methodology of electron and X-ray imaging, died on 28 June 2021. John's career spanned developments in high-resolution electron microscopies to make the first observations of dislocation kinks at the atomic scale and the bonds in metal oxides and GaAs, to projection imaging using electrons emitted from a single-atom field-emission tip, to some of the first demonstrations of lensless X-ray imaging, and culminating in the spectacular new field of serial femtosecond crystallography using X-ray free-electron lasers. He was fascinated by the phase problem in diffraction and crystallography, with a close intellectual connection to Lawrence Bragg who made first attempts to utilize changes in unit-cell dimensions to map out the underlying molecular transform. The brightness inherent to undulator devices at synchrotron radiation facilities and, later, to X-ray free-electron lasers, provided coherent beams and fertile ground for fresh new ideas and methods in imaging and diffraction. John's sweeping knowledge of Fourier optics and microscopy theory, coherent micro-diffraction, dynamical diffraction, solid-state and quantum physics, as well as an inventiveness for instrumentation harking back to the age of enlightenment (some of his heroes were Lord Rayleigh, Humphry Davy and James Clerk Maxwell) brought forth a stream of new instruments and methods. John was a prodigious writer of papers, books, emails, lists and post-it notes. He shared ideas freely and widely through such writings and through his enthusiastic talks and interactions with colleagues and strangers all around the world.

John was born in Canberra, Australia. His father was a decorated Royal Australian Air Force (RAAF) pilot who was killed in action in the Korean War when John was four years old. His mother, a strong personality, imbued him with a keen sense of fairness, empathy, humanism and kindness that set him apart over the course of his entire academic career. He carried out his PhD at Melbourne University with Alan Spargo on electron energy loss spectroscopy (EELS). Together with CSIRO, Melbourne University was an early bastion of electron microscopy. In a postdoctoral fellowship at Oxford University with Sir Peter Hirsch, David Cockayne and Mike Whelan, John advanced dynamical diffraction theory and the pursuit of imaging atoms in the TEM, using channelling and lensing effects caused by columns of atoms in a crystal. He arrived in the USA in 1976 to join the group of John Cowley, a former Melbourne professor and founder of the Center for High Resolution Electron Microscopy at Arizona State University. There, he worked on the theory of the scanning transmission electron microscope (STEM), continued EELS in this microscope configuration, and further developed ideas to use the electron wavefield propagating through a crystal as a form of lens or as a way to locate dopant atoms by measuring X-ray fluorescence as a function of the phase of standing wave. He used high-resolution TEM to image defects in semiconductors, solving the famous question of the 'shuffle or glide' mechanism of dislocation cores in silicon. In 1995, together with Harry Kolar he was able to image moving dislocation kinks along partial dislocations by

taking advantage of forbidden f.c.c. reflections. He also made the first observations of *Pendellösung* radiation and coherent *Bremsstrahlung* due to electrons passing through the periodic potentials of crystals, a forerunner of his latest work at the BioDesign Institute, together with Bill Graves, on developing a compact XFEL.

In the 1980s, as an assistant professor and together with Mike O'Keefe and Jian-Min Zuo, John continued developing convergent-beam electron diffraction in the STEM to quantitatively and accurately map out charge density distributions. This culminated in the direct observation of atomic *d*-orbitals of copper oxide. With Mike O'Keefe in 2005, he also was the first to apply the charge flipping algorithm to experimental data and solve a real structure. At this time, John also started to investigate various mathematical approaches to directly solve the many-beam dynamical diffraction inversion problem for determining unknown crystal structures. He kept returning to this quest for the rest of his life and was finally able to publish an elegant and complete solution based upon an iterative method of projections together with Jeffrey Donatelli. In a talk he gave on the topic in May 2021, John remarked on how satisfied he was to have completed this work, saying it was what he was most proud of. But in addition to these theoretical underpinnings, John was quite the tinkerer and had a love of instrumentation (and vintage cars). One major achievement coming out of John's lab was the first CCD camera for electron microscopy in 1986, later fully developed by Gatan. In the early 1990s, he was able to obtain low energy electron projection and reflection holograms using a single atom electron field emission source, with Weida Qian and later Uwe Weierstall. Around that time John became interested in and investigated the possibility of imaging biological specimens at electron energies below the carbon *K*-shell to reduce radiation damage. With Theo Vecchione, he worked on designing a coherent photofield electron source for fast imaging. Enthralled by the invention of the scanning tunnelling microscope, he placed one inside a TEM holder, together with William Lo, to make simultaneous STM and reflection mode TEM images. He then combined, together with Uwe Weierstall, an STM with a time-of-flight spectrometer for identifying atomic species on surfaces. He often quoted Humphry Davy, 'Nothing tends so much to the advancement of knowledge as the application of a new instrument' and loved the ingenuity of our scientific forebears (his joy jumps off the pages of his recent book 'Lightspeed' on the history of the measurement of the speed of light).

In addition to his developments of electron microscopy for materials, John was the driving force to obtain an NSF grant to acquire a cryo-EM at ASU, which is now installed in the Southwestern Center for Aberration Corrected Electron Microscopy. For his achievements in Electron Microscopy, John received the Distinguished Scientist Award of the Microscopy Society of America in 2006, the Buerger Medal of the American Crystallographic Society in 2012, the J. M. Cowley Medal of the International Federation of Societies of Microscopy in 2014, the Burton Medal of Microscopy Society of America (1980) and a Humboldt Senior Scientist Award in 1991. John became a Fellow of the Royal Society (Foreign Member) in 2015, and a Corresponding (Foreign) Member of the Australian Academy of Science in 2016. He was also a Fellow of the American Association for the Advancement of Science, of the National Academy of Inventors, the American Physical Society, the Microscopy Society of America, of the Royal Microscopy Society, of the Institute of Physics (UK), and an Overseas fellow of Churchill College Cambridge, UK.

A sabbatical at Lawrence Berkeley Laboratory in 1997–1998 and a decade of summers spent there in research and on scientific advisory committees brought about a new chapter in John's interests. The demonstration of direct phasing of a coherent X-ray diffraction pattern of a non-periodic object by John Miao, David Sayre and others in 1999, sparked John's interest. He initiated a long-running series of international workshops on coherence and phase retrieval where he brought together the communities of convergent-beam electron diffraction, electron holography, electron ptychography, the budding X-ray diffractive imaging, and leading experts in non-convex optimization problems. John began investigating the opportunities and limits of the approach with Malcolm Howells (Henry Chapman later joining). They carried out the first demonstration of lensless imaging in three dimensions, using soft X-rays, and explored limitations due to radiation damage. A question was whether the approach could realize atomic imaging of macromolecules without needing to crystallize them. While the idea of out-running radiation

damage using would-be free-electron laser pulses had just been published by Janos Hajdu's group, John had many other ideas which he pursued with characteristic vigour, such as diffracting with a continuous X-ray or electron source from a stream of molecules aligned in a polarized laser field, and resurrecting an old idea, due to Kam, of calculating intensity correlations from snapshot coherent diffraction patterns of an ensemble of identical but randomly oriented molecules. Correlations of photons scattered from the same molecule would accumulate whereas those from different molecules would wash out. The short exposure times would freeze out any blurring due to rigid-body rotations. John coined the phrase 'diffraction before destruction' for outrunning the Coulomb explosion with intense femtosecond-duration pulses and joined the large collaboration, along with Janos Hajdu, Ilme Schlichting, Petra Fromme and Henry Chapman and many others, to carry out first experiments at the Linac Coherent Light Source, the first X-ray FEL. The success of the method which was later termed serial femtosecond crystallography (in homage to John's earlier idea of creating orientational order with a laser) relied on many factors – one of the most important being liquid microjets for crystal delivery, that John perfected with Bruce Doak, Dan Deponte and Uwe Weierstall. The method became the most widely used technique at XFELs, providing radiation-damage-free structures of macromolecules at room temperature and with temporal resolution. This was in no small part due to the effort of many collaborators in the NSF Science and Technology Center 'BioXFEL' which John founded and of which he was Director of Science, and which now consists of 13 partner institutions. He was always open to new ideas and generous with his time, encouraging new projects and researchers wishing to make use of the new XFEL technology. Thus he paved the way of utilizing serial crystallographic approaches to obtain structures free from radiation-induced chemistry (and associated structural changes), and structural enzymology via developments such as mix-and-inject systems. Together with Janos Hajdu and Henry Chapman, John was awarded the 2021 Gregori Aminoff Prize of the Royal Swedish Academy of Sciences for their contributions to FEL-based structural biology. Sadly, John did not live to attend the ceremony which was postponed due to the current pandemic.

John was a prolific writer and his monographs *High Resolution Electron Microscopy* and *Electron Microdiffraction* (with Jian-Min Zuo) are indispensable classics for electron microscopists. His emails, like his conversations, were full of irreverent wit, blinding insights, advice and encouragements, and often hard to keep up with. His writings showed in-depth understanding of the topic but clear understanding of the factors that contributed to the success of the latest advancement. One such example is his first editorial, entitled 'X-ray lasers and crystallography', as the Main Editor (Physics and FEL Science and Technology) of **IUCrJ** where he wrote 'As so often happens in the history of science, the breakthrough eventually occurred at the interface of several fields – synchrotron science (and especially their insertion devices), laser physics, and work on microwave tubes for radar, emerging from the second world war.' 'All of this culminated recently in the construction of the first hard-X-ray laser, the US Department of Energy's Linac Coherent Light Source (LCLS), at their SLAC laboratory near Stanford. The first X-ray lasing occurred in that two-mile long tunnel on April 21, 2009, at about 2 kV, in an all-or-nothing moment of intense excitement, as theoretical predictions proved spot-on.' He went on to acknowledge foresight of DOE 'The gamble taken by the DOE in committing to the $\$600 \times 10^6$ construction of the LCLS around 2004 was laudable, in these days when low-risk incremental science seems the only way to attract funding against high odds. Will it lase? Will it be useful? The past five years have seen their vision vindicated with breakthrough applications in many fields, from materials science and atomic and molecular physics to condensed matter physics and biology.'

He had a high sense of community service which was reflected in many arenas including his service to the International Union of Crystallography. In addition to his position as the Main Editor (Physics and FEL Science and Technology) of **IUCrJ** since the journal's launch, John was Co-editor of *Acta Cryst. A* from 1989 to 1999 and served on the IUCr Commissions on Electron Diffraction (now Electron Crystallography) (1996–2002; Chair 2002–2005) and Charge, Spin and Momentum Densities (now Quantum Crystallography) (1999–2008). When in 2012, after one of us (SSH), as Editor-in-chief of the IUCr journals, approached John to lead one of the sections (Physics and FEL Science and Technology) at the AsCA meeting at Adelaide outlining the vision and ambition for **IUCrJ**, he immediately saw its importance. Despite enormous demands on his time he agreed and started coming up with ideas how to

make **IUCrJ** a natural home for FEL Science and Technology. The launch of the **IUCrJ** was announced on 5 December 2012 with five main editors (E. N. Baker, C. R. A. Catlow, G. R. Desiraju, S. Larsen, J. C. H. Spence). In addition to assembling a fantastic team of Coeditors for his section and encouraging key authors to submit high quality papers, John led from the front as is reflected in an important contribution, entitled '*Ab-initio* phasing using nanocrystal shape transforms with incomplete unit cells' that appeared in the Inaugural issue.

John had many other interests included sailing, vintage cars and music. He was passionate about playing the piano, guitar and flute, and performed with other ASU faculty and postdocs in several bands over the years. His most recent ensemble 'Who Knew' wowed attendees of many conferences – they were a special feature of the BioXFEL conferences.

John will be remembered for his wit, generosity, openness, sense of duties to the wider community and above all for his tremendous scientific contributions across the sciences, reflected in some of the books and papers given below.

John is survived by his wife, Margaret, sister, Penny, son, Andrew with his wife Rebecka and two grandchildren, and by three stepsons, James, Daniel and Peter Stanzler and their families.

## Acknowledgements

We thank Margaret Spence for clarifications and input.

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This obituary was originally published in *IUCrJ* (2021). **8**, 705–708.

OBITUARY

# Tibor Sandor Koritsánszky (1954–2021)



Anatoliy Volkov

Dr Tibor Sandor Koritsánszky, Professor of Chemistry at Middle Tennessee State University (MTSU; Murfreesboro, TN, USA), one of the most prominent scientists in the field of quantum crystallography, a member of the editorial board of the *Journal of Mathematical Chemistry* and a leader of the IUCr *XD* project, passed away on 2 June 2021. Professor Koritsánszky was a well-known international scholar collaborating with numerous research groups across the world. His research, involving quantum-chemical modeling of molecular electron densities and high-resolution X-ray and neutron diffraction data, has significantly contributed to recent technical and methodological developments in quantum crystallography. He authored several book chapters and *ca* 120 peer-reviewed scientific papers, and gave more than 30 invited talks at international conferences.

Tibor earned his Master of Science degree in Physical Chemistry in 1979 from Eötvös Loránd University in Budapest, Hungary. The next several years he spent as a research associate and/or a visiting research scientist with several leading groups in Europe and the United States, including the Hungarian Academy of Sciences in Budapest; the Department of Chemistry at the State University of New York at Buffalo, USA (with Professor Philip Coppens); Institute for Mineralogy and Crystallography, University of Bern, Switzerland (with Professor Hans-Beat Bürgi); Institute of Chemistry, University of Uppsala, Sweden; Max-Planck Institute, Mülheim an der Ruhr, Germany; and the Department of Organic Chemistry, Technical University of Berlin, Germany.

In 1992, Tibor defended his PhD thesis “Experimental Charge Density Determination” under the supervision of Professor Peter Luger at the Institute for Crystallography, Free University of Berlin, Germany, where he remained for the next few years as a research associate, and where in 1996 he was promoted to Assistant Professor. In 1994, he was selected to supervise and lead a scientific project established by the IUCr Commission on Charge, Spin and Momentum Densities to create an integrated computer package (now known as *XD*, https://www.chem.gla.ac.uk/~louis/xd-home/) for multipole refinement and analysis of charge densities from X-ray diffraction data. Since then, the program has been developed into a standard interpretive tool used by ~200 research institutes and universities from *ca* 30 countries. Dr Koritsánszky was a member of the IUCr Commission on Charge, Spin and Momentum Densities (2011–2014), a member of the IUCr Commission on Crystallographic Computing (1996–2002) and served as a member of organizing and/or program committees for numerous international conferences, meetings and schools.

In 1998, he joined the Department of Chemistry at the University of the Witwatersrand in Johannesburg, South Africa, at the rank of Associate Professor. In 2000, he was appointed a Research Professor, Head of the Jan Boeyens (1934–2015) Structural Chemistry Laboratory and Director of the Molecular Design Research Center there. The very same year, Professor Koritsánszky and Professor Coppens published

their highly acclaimed “Chemical applications of X-ray charge-density analysis” review, which has been cited more than 700 times.

Before being hired by the Department of Chemistry at MTSU in 2002 at the rank of Professor, Tibor re-joined Philip Coppens for a year at the University at Buffalo as Research Professor. At MTSU, he was also a member of the Computational Sciences PhD program and taught a diverse array of classes ranging from general education courses in Physical Science to doctoral-level courses in Computational Science. His lectures and laboratory assignments were always very well prepared and well thought through, which made it easy for the students to follow the logic of the material. Professor Koritsánszky (or “Dr Tibor”, as many students called him) taught all his classes at the appropriate level, never sacrificing integrity and rigor. He was always available and willing to help students, and treated students’ failures as his own, always looking for a way to improve the delivery and clarity of the presented material. Being able to find time for both teaching and research, Professor Koritsánszky collaborated extensively with scientists at the Spallation Neutron Source at Oak Ridge National Laboratory (ORNL). There, he led the multi-university (MTSU, North Carolina State University, Hauptman-Woodward Medical Research Institute and ORNL) project IMAGINE to obtain a state-of-the-art single-crystal neutron diffractometer at ORNL’s High Flux Isotope Reactor (HFIR). Neutrons, unlike X-rays, are ideally suited to reliably pinpoint the locations of the hydrogen atoms, especially in macromolecular structures. Moreover, the high intensity of the HFIR’s neutrons allows the study of much smaller crystal samples than was previously possible. Tibor was also an indispensable strengthening member of the German Research Foundation (DFG) priority program 1178 “Experimental charge density as the key to understanding chemical interactions” (initiated and coordinated by Tibor’s life-long colleagues from Germany: Professor Dietmar Stalke, University of Gottingen; Professor Wolfgang Scherer, University of Augsburg; and Professor Luger, Free University of Berlin), which would not have been possible without his fundamental contributions. In the last few years, Professor Koritsánszky and his former Computational Science PhD student, Dr J. Robert Michael (now at the National Center for Computational Sciences, ORNL), have been using computational tools to explore the thermal motion effects on the electron density distribution in molecular crystals, and the degree of validity of the Hansen–Coppens/Stewart pseudoatom model that has been widely used in the experimental X-ray based electron density studies.

Throughout his entire research career, Tibor was highly respected in scientific circles. His work was always scientifically careful, extremely elegant and mathematically rigorous. He had a rare gift in being able to almost instantaneously understand a broad spectrum of challenging scientific problems. Many consider Tibor to be one of the pillars of the quantum crystallography community, whose contributions to the field will continue to have an impact in the foreseeable future.

Despite his busy teaching schedule and a demanding research career, Tibor was always able to find time for his family, whether it was hiking or boating, or just relaxing at home with his wife, Maria, their sons David and Adam, and their daughter, Luca. He was a man who loved simplicity – the joy of sharing a beer on the deck with his friends, telling stories and making people laugh. It is not surprising that most people who met Tibor were almost immediately captivated by his personality and became his friends. Tibor was admired and loved by everyone around him for his outstanding intellect, his quiet wisdom, his dry wit and his kind spirit. The tragic loss has created a void in the hearts of the members of his family, friends and colleagues that (many fear) will not be easily (if ever) filled.

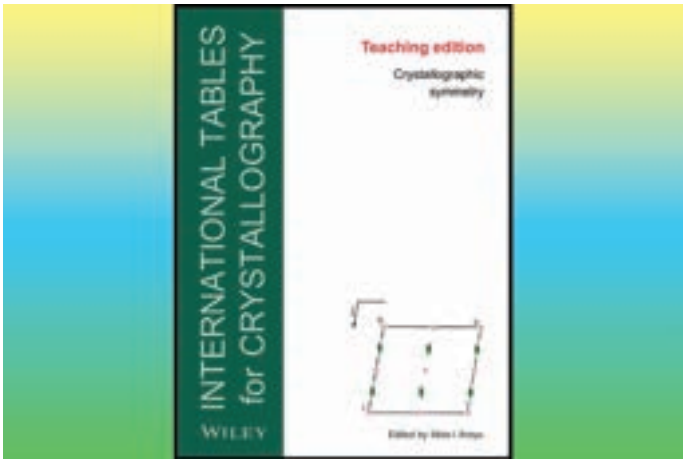
Tibor will be sorely missed, but his personal and scientific legacy will endure!

A Life Tributes page has been created for Tibor at https://www.jenningsandayers.com/obituaries/Tibor-Koritsanszky/ where everyone who knew Tibor is welcome to share their memories of him.

This obituary was written by Anatoliy Volkov, on behalf of and with contributions from the family, friends and colleagues of Tibor Koritsánszky.

BOOK REVIEW

# Teaching Edition of International Tables for Crystallography: Crystallographic symmetry




Massimo Nespolo

**Edited by Mois I. Aroyo. IUCr/Wiley, 2021. Softcover, pp. xii + 236. ISBN 978-0-470-97422-3. Price GBP 29.99.**

The *Brief Teaching Edition of Volume A* of *International Tables for Crystallography* was first published in 1985 and was meant to serve as a light, easy-to-carry sample of the content of Volume A, containing the diagrams of all of the 17 plane groups as well as 24 space groups covering the seven three-dimensional crystal systems. The pages explaining the symbols used in the diagrams as well as a small subset of the theoretical parts were also included. The usefulness of this edition was essentially in its reduced weight: if one had the complete Volume A, then it would add nothing to what was already in your possession.

Things changed radically this year, with the publication of the new *Teaching Edition* (the adjective 'Brief' no longer applies), which is no longer a simple selection of some pages from the main volumes (note the plural: the content of three volumes is now presented: A, A1 and E, and even some content related to Volume B is included!) but contains an extensive theoretical part specifically written for teachers of crystallographers, which is however perfectly suited for self-study as well. With this complete reworking of the *Teaching Edition* we definitely now have a long-overdue complement to the main volumes.

The new *Teaching Edition* spans 11 chapters divided into two parts: *Introduction to crystallographic symmetry* (seven chapters) and *Crystallographic symmetry data* (four chapters). The first part gives the theoretical background needed to understand crystallographic symmetry, *i.e.* the 'tools' that every crystallographer uses on a daily basis without necessarily mastering them. The presentation is extremely pedagogical and does not require in the reader any prerequisite that goes beyond what an average undergraduate student in any scientific curriculum usually gets (simple matrix algebra is however necessary and, sadly enough, it is no longer present in all curricula). The second part is a step-by-step guide to the use of Volumes A, A1 and E. We discuss below in some detail the content of each chapter.

## Part 1. Introduction to crystallographic symmetry

Divided into seven chapters spanning 105 pages, this first part provides the necessary understanding about point, line, plane, space and superiodic groups, with the rigour and precision that one hardly ever finds elsewhere.

Chapter 1.1 (nine pages) is *A general introduction to groups*. Besides the basic concepts of group theory, available in many textbooks, fundamental concepts like normal subgroups, factor groups and normalizers (too often considered 'beyond the scope' despite the fact that they are essential to understand relations among groups of paramount importance for *e.g.* phase transitions) are introduced as well. That all this wealth of information is collected in just nine pages without becoming cryptic for the average reader is definitely impressive.

Chapter 1.2 (seven pages) deals more specifically with *Crystallographic symmetry*, with particular attention to analysis of the matrix treatment of symmetry operations. The fundamental distinction between *geometric element* and *symmetry element*, ignored by almost all textbooks, is duly emphasized.

Chapter 1.3 (15 pages) is *A general introduction to space groups*. Here the reader finds all they need to understand Bravais lattices, the choice of the unit cells to represent them, their metric properties and the classification of space groups. Certainly surprising is to see the crystal systems relegated to the last section (Section 1.3.4.4), among *Other classifications of space groups*. For a mathematician, this category is probably less fundamental than Bravais classes, presented separately, while for a crystallographer it is *the* most used classification.

Chapter 1.4 (25 pages) deals with *Space groups and their descriptions*. A detailed explanation of how Hermann–Mauguin symbols are constructed and concretely used is followed by the presentation of the coordinate triplets, Wyckoff positions and space-group diagrams. The last part is dedicated to sections and projections, whose importance for locating atoms from diffraction data and in the construction of subperiodic groups cannot be overestimated.

Chapter 1.5 (18 pages) is about *Transformations of coordinate systems*. An updated version of the classical text and tables about the relations between the various types of unit cells is followed by a section about transformations among the various settings of space groups, with the synoptic tables of short, full and extended Hermann–Mauguin symbols up to the orthorhombic crystal system.

Chapter 1.6 (14 pages) is an *Introduction to the theory and practice of space-group determination* and is more related to Volume B of *International Tables*. Here the reader learns how to extract symmetry information from the diffraction pattern, with some basic ideas about electron microscopy, in particular convergent beam electron diffraction (although this section is too short to provide more than an appetizer). Four examples of derivation of the space group from the analysis of the diffraction pattern are worked out with enough details to provide the reader with a clear idea of how the procedure works. Particularly welcome is the reintroduction of the diffraction symbol, whose disappearance from Volume A I had criticized in my review (Nespolo, 2017). Its name has been changed from *extinction symbol* in the previous editions to *diffraction symbol*, which is definitely more appropriate, extinction being a physical phenomenon completely different to the one leading to the reflection conditions. Note however that the diffraction symbol presented here is a shortened version of the homonymous symbol introduced by Buerger (1942), which also contained the Laue class, and is closer to the aspect introduced by Donnay & Harker (1940), where asterisks were used instead of hyphens. The symbols used in *International Tables* are a hybrid of the two and this is the reason why the term *extinction symbol* was used in the previous editions.

Chapter 1.7 (17 pages) is devoted to *Applications of crystallographic symmetry: space-group symmetry relations, subperiodic groups and magnetic symmetry,* and is an introduction to the content of Volumes A1 and E that will certainly be perceived as enlightening by the reader who is unfamiliar with these volumes. These topics are of paramount importance for every scientist who goes beyond the 'pressbutton/ feed-software' blind process, yet they are still today relegated to 'advanced topics' and absent from most textbooks. This chapter will hopefully be the occasion for their democratization.

## Part 2. Crystallographic symmetry data

Divided into four chapters spanning 127 pages, this part presents a selection of plane, space and subperiodic group diagrams, enriched by step-by-step user guides.

The first three chapters show how to extract all the necessary information from Volume A (Chapter 2.1: 25 pages of text and 79 pages of symmetry tables), Volume A1 (Chapter 2.2: seven pages of text and five pages of symmetry tables) and Volume E (Chapter 2.3: two pages of text and six pages of symmetry tables).

The last chapter, Chapter 2.4 (two pages) introduces *The Symmetry Database*, which provides real added value for subscribers to the online series of volumes with respect to the print editions. Indeed, subscribers now have access not only to the electronic (HTML, PDF) versions of the *Tables* but also to an interactive database which is extremely helpful in the daily work of crystallographers and contains a wealth of information that could not be printed for obvious reason of space.

Because perfection does not belong to this world, a few improvements and corrections, although definitely minor, should be considered for the next printing.

Doubt may rise in the mind of the reader when comparing the following two statements about the rhombohedral lattice.

'For this lattice, the primitive basis $\mathbf{L}_R$ consisting of three shortest non-coplanar vectors which are permuted by the threefold rotation is also regarded as a conventional basis' (p. 21); and 'For the space groups within one crystal family the same coordinate system is usually used, which is called the *conventional coordinate system* (for this crystal family)' (p. 31).

To avoid any misunderstanding it would perhaps be better to emphasize that including the rhombohedral coordinate system among the conventional ones (p. 21) is an exception of practical use with respect to the general rule (p. 31).

A glitch occurs at p. 53, when describing an example of space-group diagram we read that 'the geometric (should be *symmetry*) element is a diagonal glide plane'.

The transformation of Miller indices under a change of basis (p. 68) is presented in the section 'reflection conditions', but these actually concern Laue indices. While it is true that Miller and Laue indices do transform in the same way, considering the widespread confusion between these two concepts (Nespolo, 2015) a precise distinction would be welcome.

The pleonasm 'holohedry of the lattice' (p. 78) could be avoided. Also, the shortcut 'chirality' for 'chirality sense' (pp. 78 and 86) could have been avoided by adopting the more precise term *handedness* ('same handedness' or 'opposite handedness' is self-evident, whereas 'same chirality' or 'opposite chirality' is puzzling: chirality is the property of being chiral and its opposite is achirality).

The term 'general reflections' (pp. 81 and 85) used in the examples following the section where the classification of reflection conditions (general versus special; integral versus zonal versus serial) might lead to some confusion in the reader about the difference between *general reflections* and *general reflection conditions*. A statement drawing the reader's attention could be useful when introducing the examples.

The citation Glazer *et al.* (2013) occurring twice at p. 225 is actually from 2014 (the reference is correct, the citation is not).

In conclusion, the *Teaching Edition* of *International Tables for Crystallography* is not, and it was not meant to be, a standalone textbook. True beginners who know nothing about crystallographic groups and have never opened *International Tables* will certainly learn a lot from this book, but will nevertheless need to go for a more basic textbook in order to get an introduction to the subject from scratch. But anybody who has used *International Tables*, possibly scratching their head over diagrams, trees and

coordinate triplets, will find here all the necessary information, provided step-by-step, to finally get the most out of the treasure trove of information collected in *International Tables*. With a more than affordable price, you have no excuse not to place your order as soon as you reach the period (meaning full stop, not translation!) at the end of this sentence.

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This article was originally published in Nespolo, M. (2021). *Acta Cryst.* A**77**, 506–508.

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