INTERNATIONAL UNION OF CRYSTALLOGRAPHY

IUCr Newsletter

Volume 29 · Issue 2

Collected archival edition
Original article text and available illustrations

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LETTER FROM THE PRESIDENT

An extraordinary year, an extraordinary Congress!



Sven Lidin

It is really an ordinary Congress, but has been postponed from 2020 and instead of being bookended by the previous and the coming triennia, it is preceded by a quadrennium and followed by a biennium. It is all very confusing. It is confusing to me, it is confusing to the IUCr Executive Committee (EC) and it has been confusing to all our Commissions who have all had to find work-arounds for the changed conditions for our work.

During the month of June 2021 the EC held virtual meetings with all the IUCr Commissions. This is normally a major task to organize when everyone is on-site for the General Assembly, but trust me, it is no less of a task to organize when everyone is at home and sitting in their own time zones. The time slots when delegates from Australia, Asia, Africa, Europe and the Americas all can meet comfortably are limited. They are in fact so limited that it is impossible, and only through the flexibility of everyone were we able to make this work to a fashion.

I am deeply indebted to the EC and the Commission Chairs for their devotion to make this happen and to our Executive Secretary, Alex Ashcroft, for organizing and keeping everyone informed, alert and happy, regardless of the local time.

We have also held the Finance Committee meeting ahead of time in order to take care of business and minimize the necessary meeting time in conjunction with the upcoming Congress.

The Congress and General Assembly will be run on European time and for the sake of all participants from other time zones, we will try to keep the formal meetings to a minimum. It is a challenge.

I want to make my last bulletin as President of the IUCr one of hope and enthusiasm for the future, and I think how all of you have faced these very challenges is a source of inspiration. With so much dedication from so many people the IUCr stands stronger than ever.

The Prague Congress will be unique in our history, and the local organizers, headed by the ever enthusiastic and hopeful Radek Kuzel, are doing the impossible – foreseeing the unforseeable, planning for the unplannable and adapting to rapid change.

I hope that as many of you as possible can join us in Prague, in real life or virtually. Please check the website for updates and possibilities for you. It is of course particularly important that speakers and delegates for the General Assembly familiarize themselves with the procedures for this hybrid meeting: IUCr 2020 – 25th IUCr Congress (iucr25.org).

I wish to end this bulletin as I started four years ago by thanking you all for the trust you have put in me by electing me and for all your hard work and enthusiasm that has kept our union alive through these difficult times. I hope and believe that the Prague Congress marks the beginning of the end of the COVID pandemic and that we may meet as we used to in the near future to reforge the bonds that form our union.

EDITORIAL

Editorial



Centre image by permission of Thomas Hooper (Stainlessvision).

Mike Glazer

What passes for summer is now upon us, and thoughts are turning to the possibility of being able to enjoy a holiday in foreign parts. Here in the UK, the latest plan is to remove most of the COVID restrictions by 19 July despite warnings from many scientists. I think we are part of an experiment. We shall see.

One of the (many) problems of getting older is the frequency with which one sees old friends passing on. Recently, I was shocked to hear of the death of John Spence (we hope to have an obituary in the next issue). You may recall that John wrote a fascinating article for us last year.

Another well-known crystallographer who has passed is Sidney Abrahams. A stalwart of the IUCr, Sidney was for several years Editor-in-chief of our journals. At the time, I was Editor of *J. Appl. Cryst.* and so attended many journal commission meetings chaired by Sidney. We also had common research interests in the field of relating crystal structures to physical properties. Also in this issue, we report the deaths of Joseph Yariv (born Poland, died Israel), Sow-Hsin Chen (born Taiwan, died USA), Jerry Jasinski (USA), Ekkehart Tillmanns (born Germany, died Austria), Nikolay I. Leonyuk (Russia) and Graciela M. C. Punte (Argentina).

On pleasanter matters, I was delighted when English Heritage erected a blue plaque in honour of Kathleen Lonsdale. These are rare recognitions of places where famous individuals have lived or worked, and those of you who have been to England may have seen these on buildings.

There has recently been a great deal of discussion on the meaning of the word “crystal.” The problem arose when it was discovered that the old rules about periodic arrays of atoms and molecules (as taught in most textbooks) to define a crystal needed some amendment after Nature threw a curveball several years ago, with the discovery of quasicrystals by Dan Shechtman (Nobel Prize in Chemistry, 2011). It turns out that defining a crystal requires much more careful consideration. Recently, Carol Brock set up a discussion by the IUCr Nomenclature Commission to consider this problem and, following this, was able to update the definition in the Online Dictionary of Crystallography in order to reconcile the old and new definitions. It would be interesting to hear what you, the reader, think about this. Simply send me your comments directly, and we can then publish them as a letter.

Two great Russian scientists also figure in this issue. Yuri Slovokhotov and Alex Yanofsky tell us about Yuri Timofeevich Struchkov, who was a renowned crystallographer, widely admired throughout the world for the huge range of his research. He and his group were prolific in publishing their work, and indeed

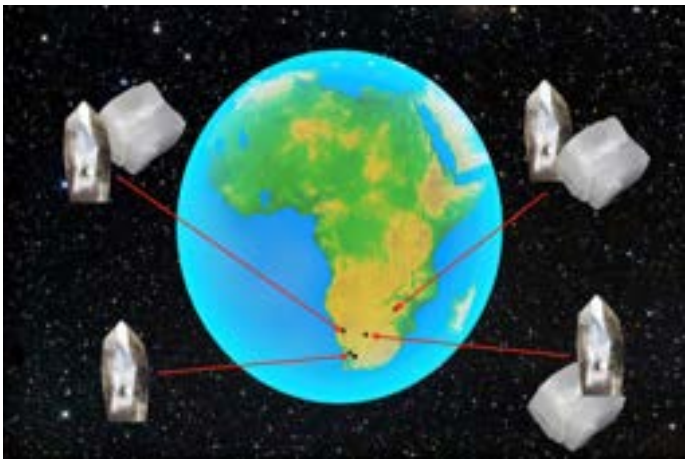
Struchkov’s name was on hundreds of papers. In 1993 he was elected as Vice-President of the IUCr.

Istvan and Balazs Hargittai have written about one of the most influential Russian crystallographers of his time, namely Aleksandr Isaakovich Kitaigorodsky, who was Struchkov’s PhD supervisor. I have a personal connection to him in that when I was doing my PhD research beginning in 1965, my topic was the crystallography of mixed organic crystals. Kitaigorodsky was the only other person in the world working on this at the time, and so I was an avid reader of his papers and books, using my rudimentary knowledge of Russian plus the help of a dictionary. Now, this leads me to an anecdote. I had always wanted to meet the great man, and in 1966 I was able to attend the IUCr Congress in Moscow. I took the opportunity to visit Kitaigorodsky’s Institute, but unfortunately, he was not there then. I did get to meet some of his staff members and see the equipment that I had read about in his publications. The next opportunity came in 1975 at the IUCr Congress in Amsterdam. Kitaigorodsky had been allowed to leave the Soviet Union to come to the conference for the first time in many years. He was supposed to have a poster on his work, and so in anticipation, I went to look at it. I was most disappointed to see that the poster board was blank and no sign of my hero. So I then set about tracking him down and eventually found him sitting on a chair out in a corridor giving court to several of his acolytes who surrounded him. I went up with some trepidation and introduced myself to him. I said, “Professor Kitaigorodsky. I was sorry to see that you had not put up your poster. I was so looking forward to discussing it with you.” He laughed and then replied very slowly, “Well now ... if I go to the poster board ... then I would have to put my poster there ... and then people will come to see my poster ... and then they will ask me questions about it ... and then I will have to answer their questions ... . Better not to put the poster there.”!

Finally, very recently, the biological crystallography community has received a most important boost to their abilities to solve biological crystal structures. The development of artificial intelligence in the prediction of protein folding has caused considerable excitement, especially as the organisations involved have now made this freely available to everyone. I am grateful to John Helliwell who has written about this fantastic tool for the *Newsletter* here.

FEATURE ARTICLE

Ancient crystal collectors



Juan Manuel García-Ruiz

In a paper published in *Nature* in March 2021, Jayne Wilkins and co-workers reported the discovery of non-utilitarian calcite crystals collected by *Homo sapiens* during the late Pleistocene, 105,000 years ago (Wilkins *et al.*, 2021). The crystals, along with burnt ostrich eggshells, were found in the excavations of a rock shelter located in Ga-Mohana Hill, at the edge of the Kalahari Desert (southern Africa) that at that time was a considerably wetter savanna environment. Unlike previous deposits, the shelter is sited 600 km inland, suggesting a non-coastal but pan-African origin of behavioral innovations among *Homo sapiens*. The discovery confirms once again the enigmatic attraction that crystals provoke in hominins.



The cache of calcite crystals deliberately collected by *Homo sapiens* 105,000 years ago in the Kalahari desert, then a wetter savanna. Scale bar in cm. Courtesy of Dr Jayne Wilkins.

The collected crystals are semi-translucent white rhombohedrons with regular, well-formed facets up to 3.2 cm in length (see Figure). Calcite crystals with a rhombohedral cleavage shape do not occur naturally in the shelter walls or ceiling. Therefore, according to the anthropologists, they were intentionally collected, most probably from a dolomitic limestone exposure about 2.5 km from the shelter. Calcite crystals are too soft (of value 3 on the Mohs hardness scale), and they do not have a conchoidal fracture but a perfect rhombohedral cleavage. Therefore, they cannot be used for the production of tools. Because of their mechanical properties, their size and the lack of reworking, the authors claim that these crystals represent a small cache of deliberately collected non-utilitarian objects. In other words: more than 100,000 years ago, *Homo sapiens*, living far from the coast, had developed a mind capable of complex symbolic behavior.

There are two main reasons why crystals could be attractive to our ancestors: their optical properties and their unique morphology. The Kalahari's calcite crystals are not transparent, so we can discard the notion that the hominids were amazed by observing double birefringence. They are not bright but instead are matt whitish. Therefore, the attractiveness of these crystals must have been in their morphology, their polyhedral, Euclidean forms being so different from the fractal and curved shapes of all the natural objects of the savanna. Although the age at which the Kalahari crystals were collected may seem very old to us, the truth is that crystals were the first objects that hominids collected, including *Homo erectus*, far before the creation of consciousness. There is much evidence of this (Bednarik, 2003; Harrod, 2014). As discussed in an earlier edition of the *IUCr Newsletter*, twenty quartz crystals were found in the famous cave at Zhoukoudian alongside the remains of *Homo erectus* that date back 700,000 years (Pei, 1931). Complete quartz prismatic crystals were found in a stratum from the Lower Acheulian (between 300,000 and 150,000 years) at the archaeological site of Singi Talav in India (d'Errico *et al.*, 1989). Interestingly, these Indian crystals belonged to different outcrops, which implies an extraordinary level of pattern recognition. The Wonderwerk Cave, also in the Kalahari Desert, may represent the earliest known collected quartz crystals in southern Africa, but need further evidence for their significance and antiquity (see Wilkins *et al.*, 2021, supplementary information). Crystals of quartz younger than those of calcite collected at Ga-Mohana Hill have also been reported in other archaeological sites in Africa and Eurasia. Remarkably, there are no quartz crystals in the 105,000-year-old deposit, but many of them have been found in the overlying levels that date to 31,000 years old and 15,000 years old (Wilkins, personal communication).

None of those crystals were used as tools, nor worked on, nor do they even have perforations or signs of use as jewels. They were objects that the hominins considered valuable in themselves. In summary, 700,000 years ago (perhaps even earlier if other discoveries are confirmed), the brain of *Homo erectus* felt so attracted by the shapes of quartz crystals that they decided to treasure them (Bednarik, 2011).

The question is unavoidable: why did those hominins, who had not yet a developed consciousness, feel drawn to those quartz and calcite crystals? Most probably, the explanation comes down to their singularity (García-Ruiz, 2018). When *Homo erectus* tried to understand the world with their preconscious brains, the first thing to do was to find patterns, to separate what is similar from what is different. Everything they saw in the savanna was curved or ramified. They observed the trees, the bushes, the furrows carved by water, the streams, the clouds, the mountains, the animals and also their fellows. There were no straight lines, no object formed by flat surfaces, no polyhedral shapes. Everything that Nature has created on the Earth's face is the product of continuous branching and curvature, of the iteration that leads to self-similarity. Everything, except crystals. When *Homo erectus* discovered quartz, pyrite, calcite or fluorite crystals, they must have noticed that these polyhedral objects were utterly different from all other objects around them.

This unique geometry of crystals is likely why hominins so desired them. This is why they are considered key pieces when seeking for early symbolic behavior. The implications of this “crystal fascination” are various and important. Do our brains like order because we were attracted by crystals, or did our

ancestors collect crystals because their brains were already prepared to search for order? Were crystals the catalysts for the neuronal processes that led to the ability to abstract and to use abstraction for understanding the world? Did crystals lead the first art-style towards abstraction and geometry? Did crystals play a role in creating a big beyond? Are they in the origin of religions and teleology? New discoveries of crystal collectors in African and Eurasian archeological and paleontological sites, such as the excellent one reported by Wilkins and her team, are needed to understand the relation of different species of hominins with idiomorphic crystals. But a deep meaning of that relation and its contribution to shaping our culture will only be achieved after future neurological investigations with children and apes.

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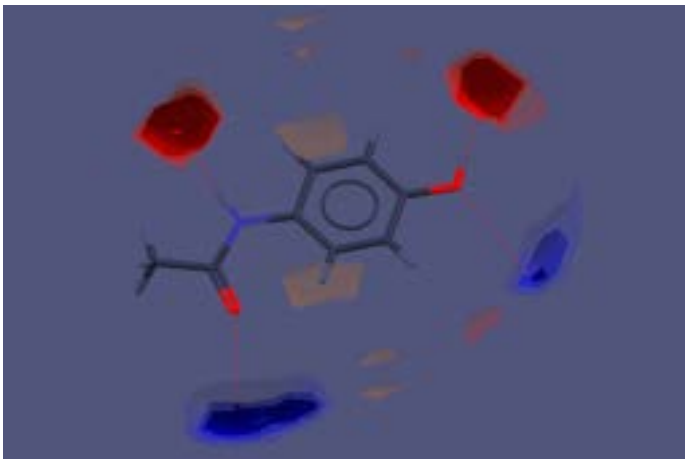
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FEATURE ARTICLE

Digital design of pharmaceutical solid forms



Cheryl L. Doherty

The pharmaceutical industry delivers new drugs after a lengthy research and development process, often 10 years or even more. Whereas drug molecules are chosen for their biological activity, the physical properties of the active pharmaceutical ingredient (API) have a significant impact on the success of the medicine. This is because the internal arrangement of the molecules and inter- and intramolecular interactions in the solid state affect the behaviour of the drug in many ways such as by influencing the solubility, dissolution rate, chemical and physical stability, and tabletability. Together these solid-state properties contribute significantly to determining the safety, manufacturability and bioavailability of the eventual drug.

The solid-state properties are locked in when the selection of the solid form and associated crystallisation route are determined, making these key milestones in the drug development process. Viable options for development may include single-component free drugs, salts, hydrates, co-crystals and occasionally even solvates. Pharmaceutical materials scientists evaluate the accessible solid forms for each API and develop one that exhibits the physical and mechanical properties most suitable to produce a successful drug product. It is well known that many APIs also spontaneously crystallise in multiple arrangements to form polymorphs and for the same reasons these different polymorphs of an API will also have different physical properties. Typically the solid form that is most stable under conditions relevant to the manufacturing and storage of the drug product is considered the most acceptable form for development. It is standard practice in industry to perform experimental solid-form screening to identify the thermodynamically stable and relevant metastable polymorphs initially during the pre-formulation phase of drug development, with more comprehensive screens being carried out later on.

Whereas the best chance of finding the most stable solid form comes from designing the widest experimental screen possible, it is not feasible to explore every possible experimental condition in a reasonable timescale. There can also be practical challenges to this approach including poor solubility, ubiquitous solvate formation, or the slowing/switching off of form changes due to the presence of impurities. Therefore, despite best efforts, unexpected new polymorphs can still appear, even in well-screened systems and there have been multiple examples of drug product recalls involving a change in the API solid form. Ritonavir[1] and rotigotine[2] are two notable examples that show that the late discovery of a new stable polymorph can result in significant challenges to providing a reliably safe and efficacious drug product. In each case the stable form was not identified prior to launch despite extensive experimental screening. This shows it is crucial to understand the potential solid-form landscape for each candidate and to use that information to do the right set of experiments that will allow the identification

of the stable form for every case. Control of the solid form is of such importance that solid-form screening is a regulatory requirement for new drugs with the robustness of the solid form to meet safety, efficacy and manufacturing requirements being covered in ICH regulatory guidelines.[3,4]

For these reasons the selection of an appropriate pharmaceutical solid form for development, previously based wholly on experimental output, is now commonly enhanced with ‘digital design’ by using computational structural tools in parallel. A range of tools is now available and these tools may be applied individually or in combination to inform the selection decision. For statistically based methods, which compare the crystal structure of a new API against observations in published crystal structures, access to a large dataset that is highly relevant to the molecules under study is considered to be essential. The extent to which this is true for the publicly available Crystal Structure Database (CSD) has been demonstrated in a detailed comparison with two proprietary pharmaceutical databases.[5] Information derived from the CSD has now been used on several pharmaceutically relevant compounds to make successful knowledge-based predictions of polymorphism using the hydrogen-bond propensity analysis. This model carries out a statistical analysis of hydrogen-bonding patterns for a given structure, identifies potential patterns and compares these to those in known crystal structures in order to rationalise the physical form stability. The observed thermodynamic stability ranking for polymorphs of both lamotrigine[6] and crizotinib[7] have been successfully rationalised using this approach.

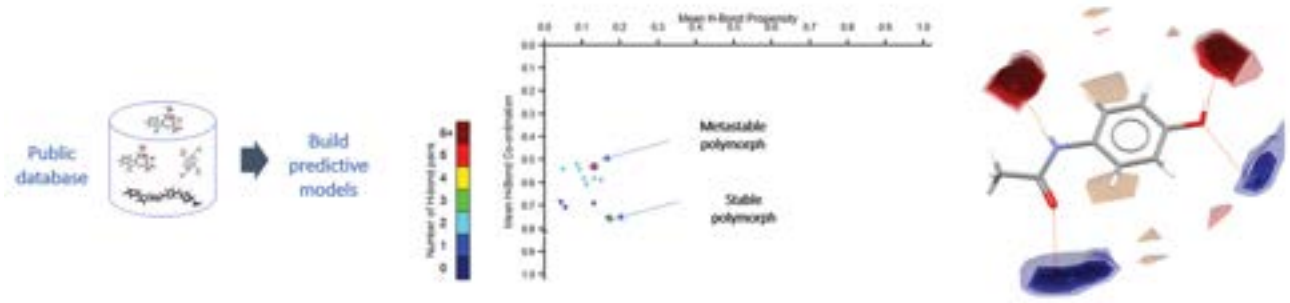


Figure 1. Crystal structure databases can be used to make predictive models to identify stable solid forms using (middle) hydrogen-bond propensity and (right) full interaction maps.

Full interaction maps are a further tool that create a visualisation of the common geometries for intermolecular interactions in the form of a colour density plot. The data are drawn from observed geometries from a wide selection of pre-defined functional groups that are combined into a single tailored visualisation, specific to the molecule and conformation. Full interaction maps have also been successfully used to assess polymorph stability in pharmaceutical candidates, including the anti-bacterial drug suloprenem.[8]

The use of these tools requires a single-crystal structure to be obtained, involving the preparation of single crystals of a suitable size and quality. This can be accomplished early in the development process from a relatively small amount of pure API and a little – or sometimes a lot – of patience. Advances in diffraction technology allow ever smaller crystals to be examined, by X-ray diffraction and more recently using electron diffraction techniques. Once the structure has been obtained these knowledge-based analyses allow an early assessment of the likelihood of polymorphism, identifying high-risk candidates and enabling risk mitigation strategies (such as extra screening) to be initiated.

Crystal structure data are, however, not always readily available, especially at very early stages in the drug discovery process. It is also valuable to consider all the possible forms in a potential solid-form landscape, even those that have not yet been obtained experimentally. For this reason the *ab-initio* generation of solid-state structures by computational methods starting from only the molecular structure has been explored with increasing interest across the pharmaceutical industry.[9] The great improvements in the ability to accurately predict crystal structures has been tracked by a series of blind tests hosted by the Cambridge Crystallographic Data Centre (CCDC) over the last 20 years. The results of these blind tests demonstrate the rapid improvements in both accuracy and in the increasingly API-like

nature of the test candidates. The most recent of these in 2016 showed the successful predictions for salts and polymorphic systems taken from real, modern candidates in the pharmaceutical industry.

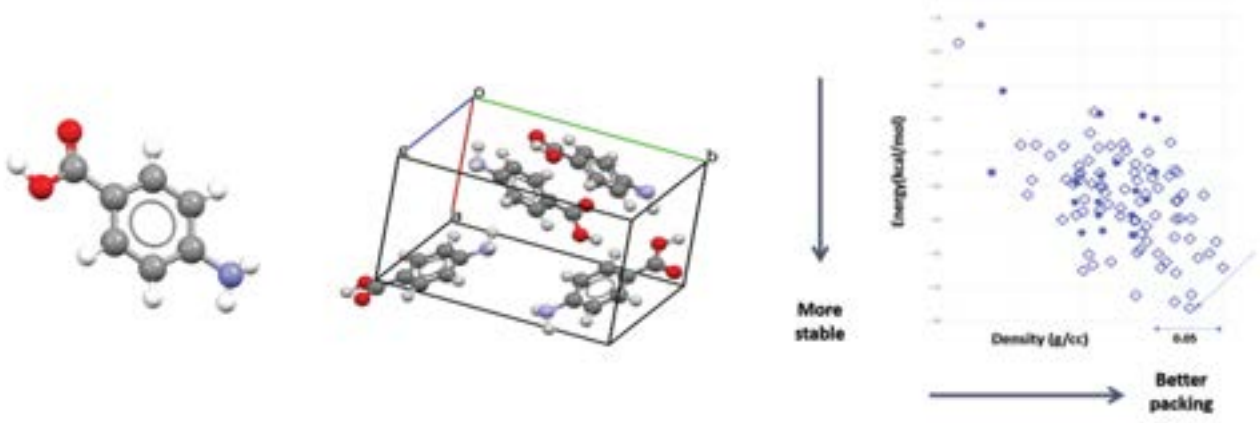


Figure 2. Crystal structure prediction methods calculate the possible conformation and packing possibilities for an API, identifying the most energetically stable form possible.

It is possible to guide the solid-form screening work to be carried out by comparing the computationally predicted solid-form landscape with the experimentally known solid forms for a new candidate. Where the experimentally obtained form matches the predicted arrangement and is energetically distinct, the project can be progressed rapidly with high confidence in the stability of the polymorph. If not, focussed experiments targeting the predicted stable form would be initiated to mitigate the risk that the most stable form has not been found. The application of these tools and technologies have maximum impact in fast moving therapeutic areas, allowing candidates to progress quickly into the clinic with a stable and appropriate solid form. This has been seen to be successful in the rapid acceleration of an oncology candidate to commercial launch by applying CSP for crizotinib[7] and the use of cloud-based methodology as a proactive “virtual polymorph screen” on oxabispidine.[10]

It is clear that the addition of structurally based computational tools to solid-form development has enhanced the traditional experimentally driven selection process. Faster calculations, in some cases using cloud-based methods, allow these techniques to highlight risk even at early stages of development, leading to more informed experimental design. By implementing these tools we can even allow structural modifications to be tailored in order to improve the physical properties of the solid form prior to commencing studies in the clinic. The advent of digital design in the pharmaceutical solid state forms the heart of a computationally enhanced design process that can enable the solid-form landscape of a new API to be fully understood and allowing rapid product design.

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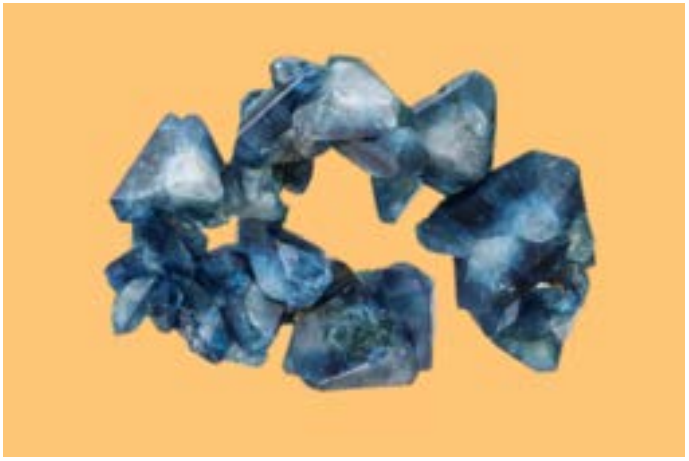
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FEATURE ARTICLE

Museum crystallography and community interactions



Benitoite, California’s state gemstone. This benitoite ‘wreath’ is one of NHMLA's more famous specimens. Photo credit: NHMLA.

Aaron J. Celestian

Museums are not just warehouses of objects. While the Natural History Museum of Los Angeles County (NHMLA) does have many objects (>35 million and counting) in its collections, and the gems, minerals, rocks and meteorite collections specifically at NHMLA house about 175,000 specimens, our larger goal is to serve our communities, both the scientific and broader public. The museum is a repository for extinct materials (*e.g.* from depleted mines, excavated land to make way for buildings, changed environs) and newly discovered crystals, either by natural processes or synthesized in the lab. The museum is a trusted resource, where new knowledge can be generated and interacted with, in an accessible and safe space.

One theme of research in the mineral sciences department at NHMLA involves interactions between humans and the minerals in the environment. This can be thought of in three ways: (1) human influence on the landscape that alters solid Earth chemistry in a permanent way, (2) anthropogenic changes to the Earth system that affect climate and biological processes, and (3) mineralogical and industrial processes that directly affect human health.

One way to measure how fast a planet is changing is to measure its mineral diversity over time. Today, there are over 5700 minerals (mindat.org) that have been described with about 100 new minerals each year, but the Earth started with roughly 250 minerals (Hazen *et al.*, 2008). As the Earth changed (*e.g.* plate tectonics, the Great Oxidation Event, biomineralization), the chemistry of Earth also evolved. These major events are marked by changes in mineral diversity in the rock record. Human-environment interactions have produced over 200 minerals that would not have otherwise occurred (Hazen *et al.*, 2017), and many of these are now curated in NHMLA’s permanent collections. Two examples of these minerals are rowleyite (Kampf *et al.*, 2017) (Fig. 1) and phoxite (Kampf *et al.*, 2019), which are new structures recently discovered from the Rowley Mine. This mine allowed bats to take residence, and chemicals in the bat feces reacted with the mine wall to produce rowleyite (the most porous framework mineral) and phoxite (a porous phosphate oxalate framework structure). Work at NHMLA uses *in situ* diffraction to monitor crystal growth so that we can better understand the biogeochemical conditions of formation and the mechanisms of crystallization. Both new minerals have potential applications in carbon capture and agriculture, respectively, which would further increase the footprint of human-environment influence. The rapid expansion of the mineral kingdom that is co-evolving with human activity is one global marker of changes to Earth chemistry. It thus is thought of as an indicator of the start of the

proposed Anthropocene Epoch.

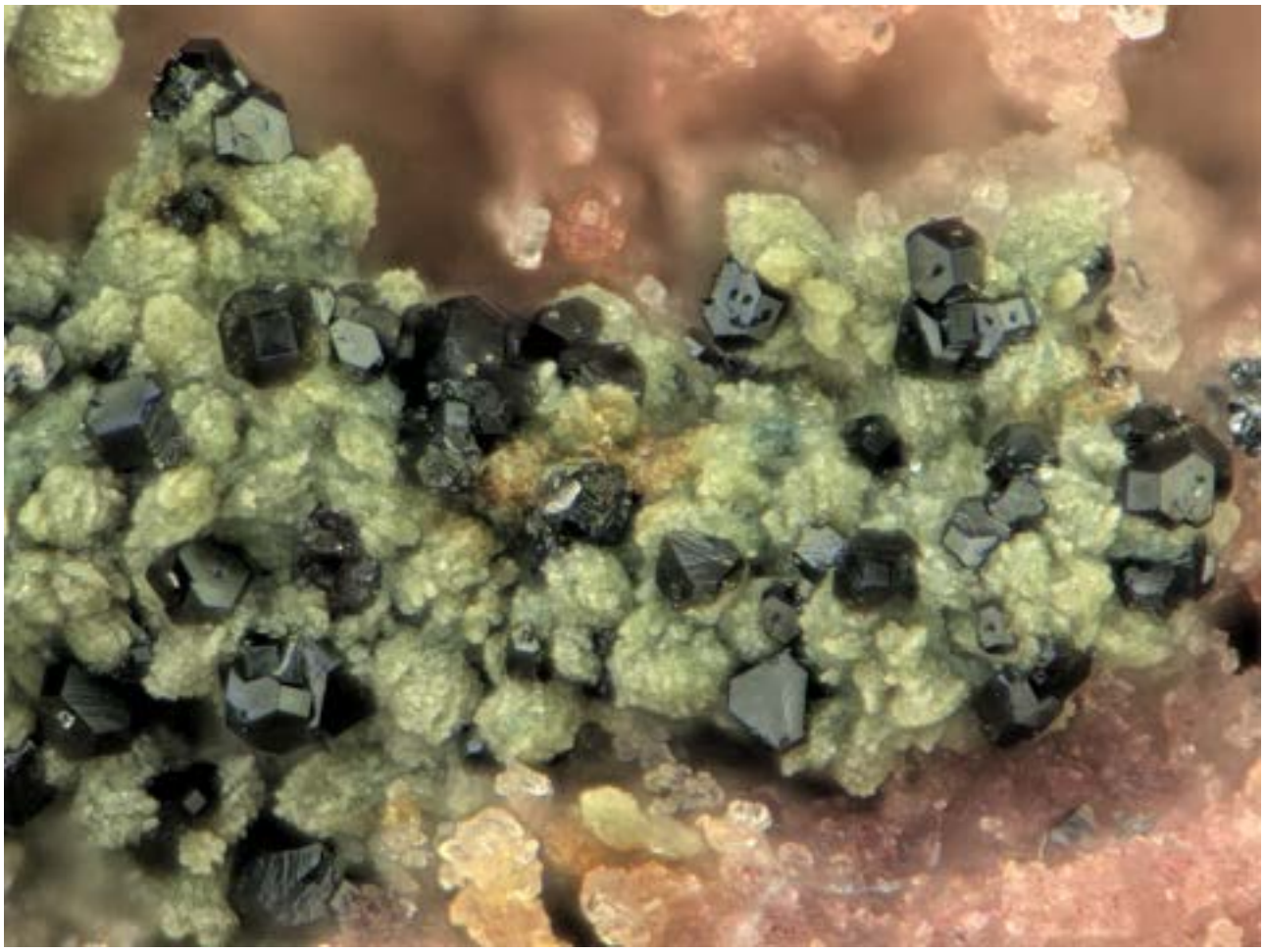


Fig 1. Rowleyite crystals (black) with mottramite (green) on quartz from the Rowley Mine, Arizona, USA. The field of view is approximately 0.5 mm.

In today's oceans, marine pH changes resulting from carbon dioxide emissions to the atmosphere affect the balance of minerals present. NHMLA collaborates with the University of Southern California and CalTech to determine carbonate and sulfate minerals (*e.g.* calcite, aragonite, barite) present in the marine water column and compares that with the seafloor sediment record (Dong *et al.*, 2019). Not only are naturally occurring materials preserved in the geologic record, but new anthropogenic materials are incorporated as inclusions within crystals that will be preserved for geologic time. NHMLA has found the structurally disordered Magnelí phases (Fig. 2, sub-stoichiometric titanium oxides, Ti_xO_{2x-1} for x = 4 to 10) that form at coal-burning power plants (Yang *et al.*, 2017) in a wide range of environments, from the bottom of the ocean to the tops of Everest (collaboration with American Alpine Climbing Association) to evaporite minerals in hypersaline lakes (Celestian *et al.*, 2021). The biological effects of Magnelí phases are an area of ongoing study, but they are structurally stable, globally distributed and may have served as a trace mineral for the onset of the Industrial Revolution.

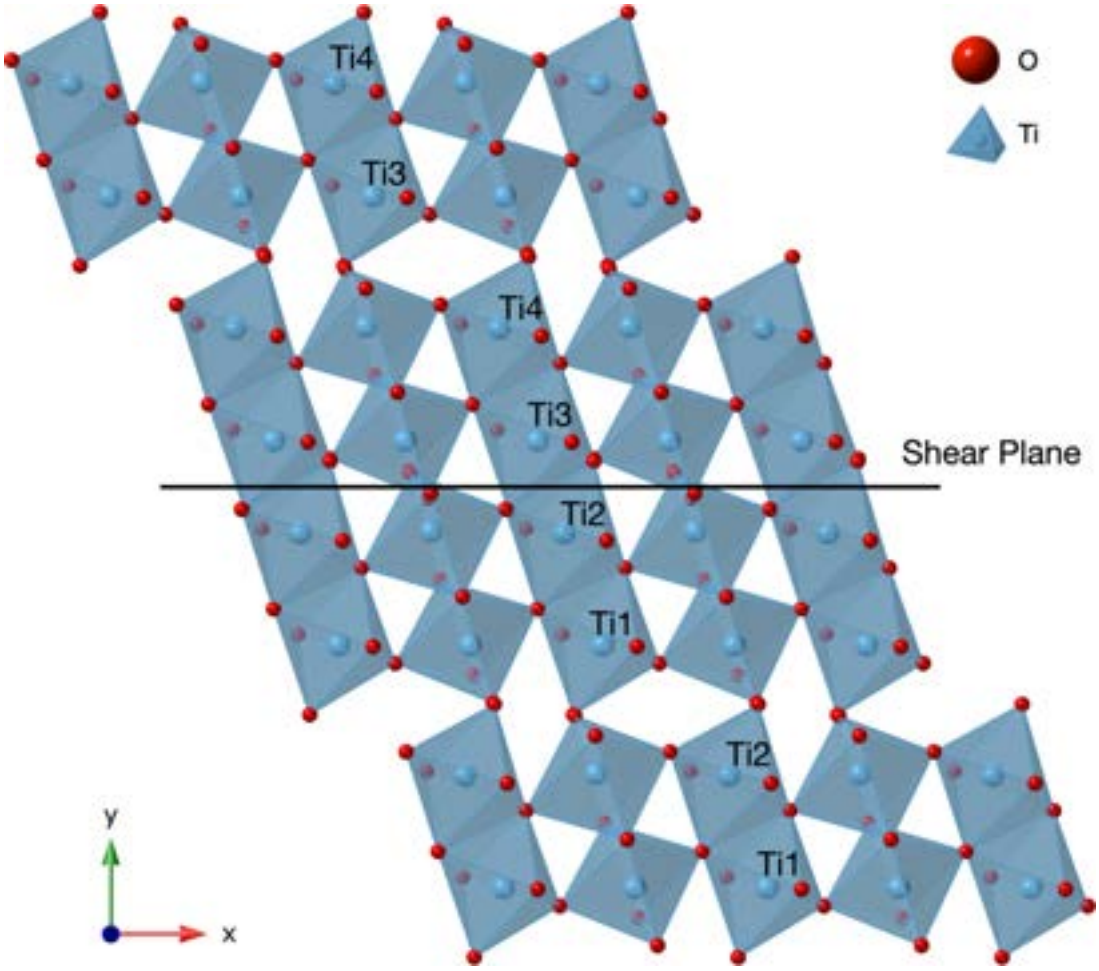


Fig. 2. A structure model of Ti_4O_7. These materials start as natural anatase or rutile, and are then heated under high-temperature anoxic conditions, which creates oxygen defects that are “filled” by systematic shear planes.

Ongoing and pervasive extraction and application of heavy metals have impacted human health. There are hundreds of examples of humans being affected by heavy-metal poisoning events. One example close to NHMLA is the environmental disaster of South Los Angeles at the former Exide lead-acid battery recycling facility (Johnston & Hricko, 2017). Funded in part by the Getty Foundation’s Pacific Standard Time 2024 (Vankin, 2021), Self-Help Graphics and NHMLA are developing a process to sequester Pb. This work will involve designing new crystals and modifying naturally occurring microporous minerals to selectively absorb Pb from soils in community gardens and yards where Pb levels can exceed 500 ppm. This project will not replace the responsibilities of government clean-up efforts but should provide a means for community members to feel safer in their own homes and gardens. The success of the project will depend on community trust garnered through effective educational initiatives regarding crystallography and crystal chemistry to demonstrate material effectiveness and stability. The resulting Pacific Standard Time 2024 art exhibition will deploy a temporary remediation method along with scientific communication through art.

Museums utilize crystallography for both basic research and applied initiatives to expand knowledge of minerals and their structural dynamics. As a publicly supported non-profit institution, NHMLA’s mission is to serve our community. Crystallography allows us to learn more about how humans influence and are impacted by minerals and enables advancement of that mission.

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SPECIAL REPORT

Reaction to announcement of AlphaFold Database



John R. Helliwell

At 4 p.m. on 22 July 2021 the bulletin board of the CCP4 (Collaborative Computational Project, Number 4, for macromolecular crystallography) conveyed, what I would call, a dramatic announcement from EMBL–EBI:

"*DeepMind and EMBL's European Bioinformatics Institute (EMBL-EBI) have partnered, initially for a 2-year period, to make hundreds of thousands (and eventually many millions) of AlphaFold structure predictions freely available to the community through a new data resource, AlphaFold DataBase (AlphaFold DB). AlphaFold is an Artificial Intelligence (AI) system developed by DeepMind that predicts a protein's three-dimensional (3D) structure from its amino-acid sequence. The initial release of the resource provides structure predictions for most of the proteins in the human proteome as well as for the proteomes of 20 other species of significant biological or medical interest.*"

[The relevant academic references are Jumper *et al.* (2021) and Tunyasuvunakool *et al.* (2021).]

The day before, in its Wednesday release of new experimentally derived Protein Data Bank (PDB)-archived structures, 214 new ones were made available, added to the ~180,000 in the archive. It was from all these that a core set of a few tens of thousands of experimental structures provided the learning set for DeepMind, *AlphaFold*2, to embark on its predictions of protein folds. That startling success was the basis for my article in the *IUCr Newsletter* last year (Helliwell, 2020), including a historical look back on the protein folding problem over many decades, as well as offering my congratulations to the DeepMind team on their research breakthrough.

I immediately alerted the *IUCr Newsletter* Editor, Mike Glazer, about the new EMBL–EBI announcement. Both he and I thought that this needed a description, inevitably brief, given a deadline for this article of four days later. Obviously to report on precisely what all this meant, I should do some direct incursions into the new AlphaFold Database (AlphaFold DB). I had already taken to heart this short extract from the EMBL scientists (Cusack *et al*., 2021) that came with the announcement:

"*While AlphaFold DB will, in general, accelerate structural biology research, it will likely also induce a shift in emphasis from initial structural determination to the study of the more mechanistic and functional aspects of protein structures. Although this in turn may lead to an objective re-evaluation of the large-scale structural biology infrastructures devoted to structure determination (e.g. synchrotron X-ray crystallography beamlines), it is likely that for the foreseeable future they will be essential to validate and thus fully harness the potential of structure prediction, and to enable structural investigations for which no reliable predictions can be made at this time (structure of nucleic acids and large complexes,*

ligand and fragment screens, investigations of dynamics, etc.)."

The above paragraph's perspective was quite similar to my own (Helliwell, 2020), apart from the bit about the need for an "*objective re-evaluation of the large-scale structural biology infrastructures devoted to structure determination (e.g. synchrotron X-ray crystallography beamlines).*" As a pioneer of the development of synchrotron radiation beamlines and their use in crystallography, I obviously have some affection for such infrastructures, and so this is me saying I have a conflict of interest in scrutinising such a statement. Whether these beamlines are to be eventually replaced, under the auspices of the AlphaFold DB, I was in turn rather struck by what I presume is the Google lawyers' view of an AlphaFold DB structure:

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(the prefacing of each line of the above with "REMARK" is the style of a PDB coordinate file).

Anyway, whatever the lawyers might imagine, I set about doing some tests. I chose my Oxford University DPhil project (1974 to 1977): "X-ray studies concerning the structure of sheep liver 6-phosphogluconate dehydrogenase (6PGDH)", available on request from the Bodleian Library, University of Oxford. My research and those of a few further PhD students, under the expert supervision of my supervisor Dr Margaret Adams, led to the PDB deposition 2pgd (Adams, Helliwell & Bugg, 1977; Adams *et al.*, 1991). AlphaFold DB offered several predicted 6PGDHs, none for the sheep (*Ovis aries*) liver enzyme. But I could choose the human enzyme predicted structure, with its closely similar amino-acid sequence (Fig. 1).

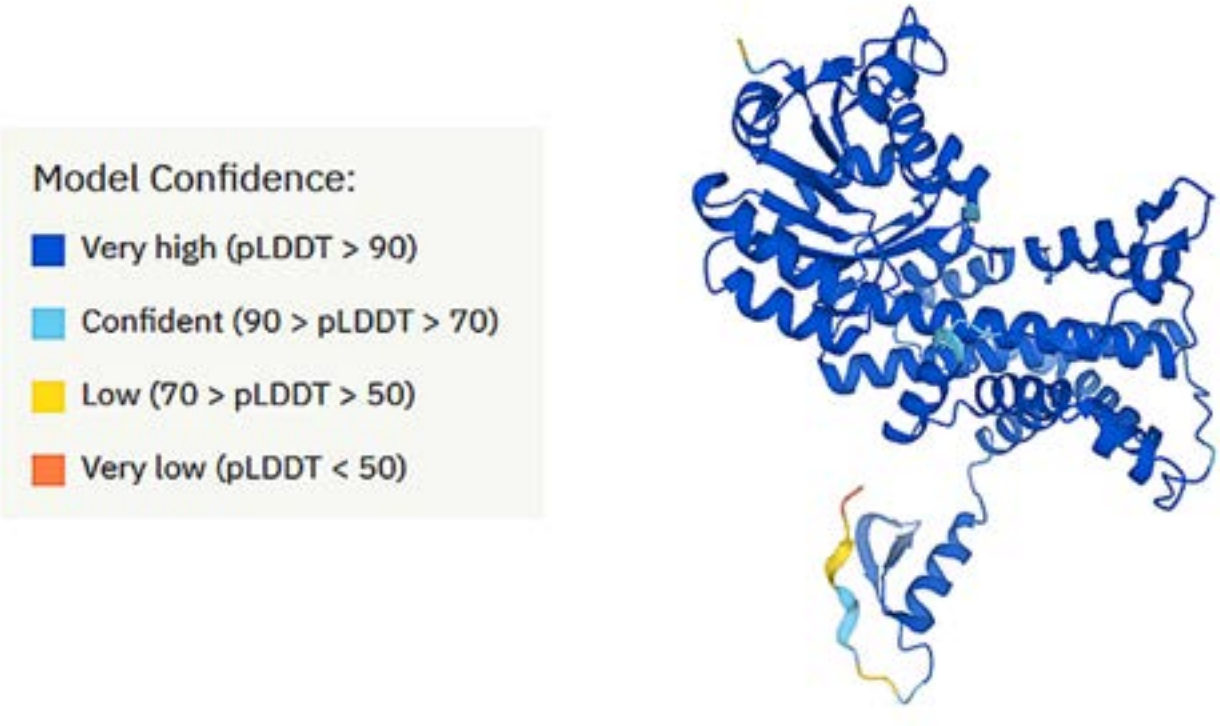


Figure 1. The AlphaFold DB human 6PGDH. This shows the ribbon diagram for the predicted 3D structure. Note the "Model Confidence" colour coding at left, *i.e.* "Very high" throughout apart from a short stretch of amino acids at the bottom of this screenshot. This "Model Confidence" is described as "*AlphaFold produces a per-residue confidence score (pLDDT) between 0 and 100. Some regions below 50 pLDDT may be unstructured in isolation.*"

I used this AlphaFold DB human 6PGDH 3D structure to perform molecular replacement (MR) with the *Phaser* MR program (McCoy *et al.*, 2007) to solve the sheep liver 6PGDH X-ray diffraction data's structure. The program ran swiftly with the concluding message "*EXIT STATUS: SUCCESS CPU Time: 0 days 0 hrs 2 mins 39.18 secs (159.18 secs)*". As Fig. 2 shows, the placement of the AlphaFold DB model by *Phaser* MR into the sheep liver 6PGDH unit cell gave the (F_{obs} – F_{calc}) electron density with clear evidence for one of our experimental crystallization procedure sulfate ions. That is, we had used high-concentration ammonium sulfate for crystallization, whose diffraction signal was in the F_{obs} (observed structure factor) list. The AlphaFold DB model was, of course, an *in silico* model, in effect in a vacuum, with no ligands and no bound waters. In the F_{obs} – F_{calc} difference map there were also clear indications of the amino-acid substitutions of histidine for glutamine at positions 56 and 213 of the sheep *versus* the human sequence (not shown). So, in terms of my DPhil, the time I had taken searching for isomorphous replacement heavy-atom derivatives for phasing would have been saved; difficult to estimate exactly, but let me say one year. But note that I also spent a considerable time on the functional crystallographic studies (substrate binding experiments and so on) and which formed a significant part of my thesis.

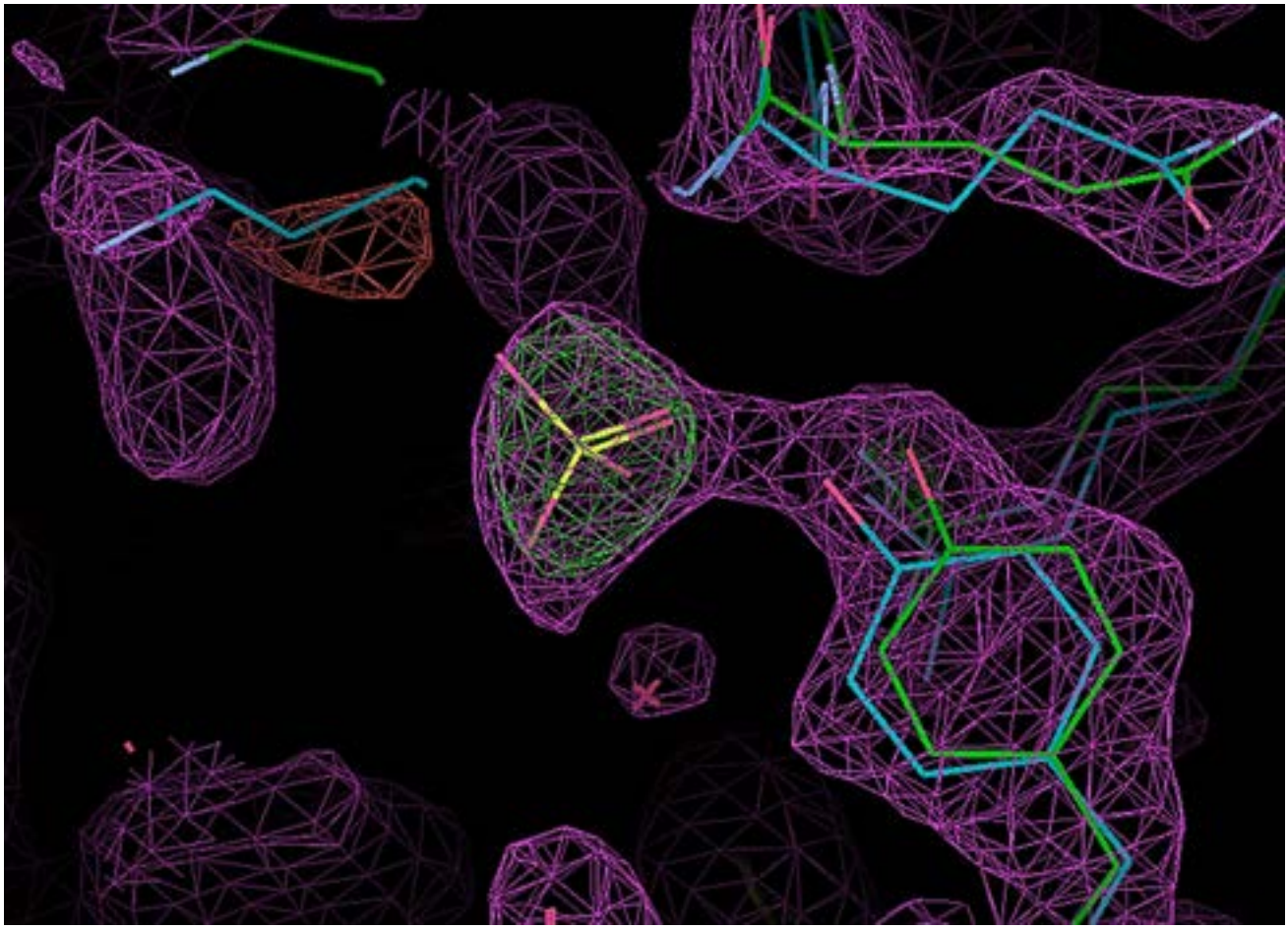


Figure 2. The (F_{obs} – F_{calc}) (contoured at 3σ, in green) and the ($2F_{obs}$ – F_{calc}) (contoured at 1.2 r.m.s., in magenta) electron-density maps from the *Phaser* MR run shows the sulfate ion present in the sheep liver 6PGDH crystal, PDB code 2pgd. The F_{obs} are the sheep X-ray diffraction structure-factor data for 2pgd (green model for protein, yellow for the sulfur in the sulfate ion, oxygens in red), the F_{calc} are based on the correctly placed human 6PGDH AlphaFold DB model (in blue, oxygens in red). This figure was prepared using *Coot* (Emsley & Cowtan, 2004).

I was alerted by my Twitter friend @aemiele "structural biophysicist based in Lyon" that the web server in Seattle https://robetta.bakerlab.org/ had much to commend it as an alternative to AlphaFold DB, also published a few days ago (Baek *et al*., 2021). I duly registered for Robetta and was able to submit the sheep liver 6PGDH amino-acid sequence. I also noted a similar view to the Google lawyers by the University of Washington lawyers:

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At the time of submitting this article to the *IUCr Newsletter*, I have not yet received an email with Robetta's predicted sheep liver 6PGDH 3D structure. I would note though that there was a sizeable queue of submitted jobs awaiting their turn.

I would conclude this short article by saying that I think this is a game-changer in protein structural science; it will speed up many experimental studies. Again I congratulate all concerned. Besides a speeding up it may even ease a project past a complete impasse of crystal structure determination, because of the unavailability of a suitable MR model or isomorphous or anomalous dispersion phasing tools, by providing a predicted structure as the phasing start. It will, I imagine, also stimulate new project directions.

There are some further points for interesting discussion. Firstly, the vast majority of the training set of experimental structures are cryostructures, not room-temperature let alone 37°C (physiological temperature for mammals) structures. Secondly, the predicted structures have no bound waters. For substrate or inhibitor binding these waters are either displaced or are the hydrogen bonders through which a ligand molecule attaches itself to a protein. Thirdly, there is an elaborate range of validation processes that the crystallographic community has evolved (see *e.g.* https://ecm2019.org/satellites/data-science-skills-in-publishing/), including the work of the PDB Validation Task Forces, over many years. The discussions for and against the need for rigorous evaluation of predicted structures *versus* their internal 'accuracy estimates' will probably continue. My view (Helliwell, 2020) remains that, although *AlphaFold* does give its own uncertainty estimate (Fig. 1), rigorous assessment requires it be measured against an experimentally determined protein structure. The new aspect, with AlphaFold DB, is that where there is a predicted structure alone, I suggest that one should proceed with the lawyers' views, quoted above, in one's mind.

Meanwhile, the last word I think should be given to the PDBe in its tweet:

"@PDBeurope The structure predictions on the AlphaFold DB website include those that already have experimentally determined structures in the PDB. In these cases, the AlphaFold pages display links to our #PDBeKB protein pages to compare existing data."

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Editor's note: See the *Acta Cryst. D* article on *AlphaFold*2.

IUCR JOURNALS NEWS

Modern approaches and tools for teaching crystallography – call for papers for special issue



Graciela Díaz de Delgado, Sean Parkin

Acta Crystallographica Section E: Crystallographic Communications is the journal of choice for the publication of high-quality structure determinations of compounds of a very diverse nature. Many of these studies involve the use of advanced concepts to solve difficult problems such as twinning, disorder, modulated structures *etc*. Reports on the determination of challenging structures can provide a good opportunity to show young crystallographers and newcomers to crystallography how to approach these difficult problems. The global health crisis caused by the COVID-19 pandemic has had a negative impact in our field as many laboratories have had to close for long periods of time. However, scientists have continued working remotely, teaching online courses and tutoring students using different electronic meeting platforms.

Following the long teaching tradition of the IUCr, we would like to encourage you to submit manuscripts to *Acta Cryst. E* that can be used as educational materials so that the younger generation may benefit from the knowledge and expertise of experienced crystallographers.

In this new environment, we would particularly like to receive articles based on experiences of teaching crystallography online and of how best to use some of the facilities available to access diffraction equipment remotely. Articles giving practical advice on using crystallographic software to address different aspects of structure determination, refinement, graphics, analysis *etc*. would also be welcome. Besides the main text of the manuscript, we encourage the submission of supporting information containing comprehensive practical examples, instructions, software input files, reflection data and results. Contributions on new tools for teaching the fundamentals of crystallography would also be of interest. Examples of articles with a strong teaching component that have been published recently in our journal are available here.

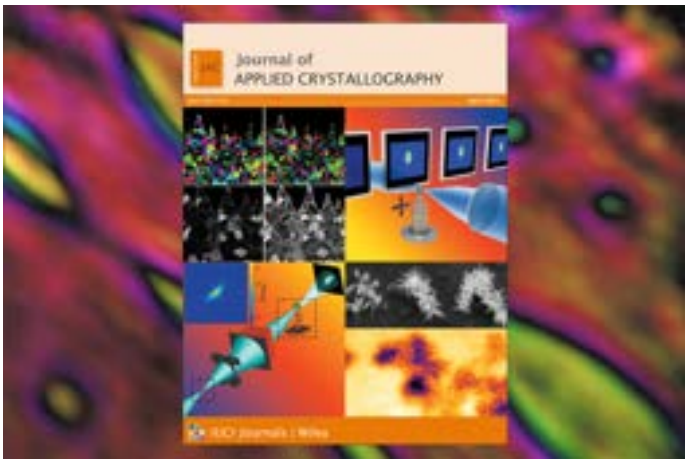
All manuscripts for this special issue should be submitted by 30 June 2022. They will be subject to the normal peer-review process.

We look forward to your submissions!

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IUCR JOURNALS NEWS

Special issue on Ptychography: software and technical developments



Stefano Marchesini, David Shapiro, Filipe R. N. C. Maia

Ptychography is an X-ray technique that achieves resolutions of one billionth of a metre, can reach a macroscopic field of view, and has the capability to retrieve chemical or magnetic contrast, among other features. It includes variants such as ptycho-tomography and spectro-ptychography, which are some of the most computationally intensive techniques being developed at X-ray light sources around the world.

In the past few years ptychography has grown into a diverse landscape of methods to address ever more complex systems. These techniques involve beautiful mathematics, from harmonic analysis and algebraic geometry to complex numerical analysis.

The Ptycho Developers 2019 workshop held at Berkeley Laboratory on 3–5 June 2019 was attended by 60 participants from five continents. The goal of the workshop was to bring together developers and practitioners of ptychography to assess the current landscape of available algorithms and software, investigate commonalities, and discuss a range of topics, from required theoretical and algorithmic advancements and unmet needs to practical issues of implementation and deployment. Other topics included workflows, beamlines, optics and high-performance computing solutions.

Several ideas presented at the Berkeley workshop were later turned into manuscripts. These manuscripts formed the seed that grew into this virtual special issue, being complemented by diverse contributions from around the world.

This special issue provides us with an opportunity to take a glimpse of some of the developments addressed in the workshop. The 14 articles it contains cover a broad variety of subjects but can be roughly divided into the following groups:

(1) A new speckle-based ptychography variant is introduced by Morgan, Murray, Prasciolu *et al.* (2020), aimed at wavefront metrology. This is accompanied by two further manuscripts, headed by the same author: one of them (Morgan, Murray, Quiney *et al.*, 2020) describing *speckle-tracking*, an open-source software package for the analysis of data from the newly developed X-ray speckle-tracking ptychography variant, and the other (Morgan, Quiney *et al.*, 2020) showcasing its first application, aiding the fabrication of wedged multi-layer Laue lenses by providing a way to accurately characterize them.

(2) Du *et al.* (2020) address the dose requirements of multiple X-ray microscopy techniques with detailed numerical simulations and conclude that it is the photon fluence, and not the experimental geometry, that limits the resolution of a properly conducted measurement.

(3) Two manuscripts highlight a broad range of direct applications of ptychography. De Caro *et al.* (2020) use it to image some peculiarly shaped nanocrystals in a polymer matrix. Sala *et al.* (2020) show us how ptychography can be used with a range of sources and a variety of purposes by imaging the wavefront of an X-ray free-electron laser.

(4) Significant advances in instrumentation are presented by three manuscripts. Khant *et al*. (2020) show us that NanoMAX is ready for ptycho-tomographic measurements, taking advantage of the high coherent flux delivered by the diffraction-limited storage ring of MAX IV. Schropp *et al*. (2020) introduce us to PtyNAMi, which stands for ptychographic nano-analytical microscope, and chronicle how the P06 beamline at PETRA III has been converted into a state-of-the-art hard X-ray ptychography instrument. Pound *et al.* (2020) tell us how ptychography can be carried out parasitically at an X-ray free-electron laser, demonstrating it at the Coherent X-ray Imaging endstation of the Linac Coherent Light Source. Beamtime at such facilities is even more constrained than at synchrotrons due to their serial nature, so the ability to carry out multiple experiments simultaneously will be very valuable.

(5) Two manuscripts by Chang and co-workers (Chang, Marcus & Marchesini, 2020; Chang, Rong *et al*., 2020) tackle the theory and algorithms for spectral ptychography and dichroic ptychography.

(6) Lastly, there were several contributions showcasing new computer codes and software, as was to be expected from a developers' conference. Baraldi *et al.* (2020) describe highly optimized software tools for fast ptychographic reconstructions, particularly suited to the high-speed fly scans that have been developed at Sirius. Wakonig *et al.* (2020) introduce *PtychoShelves*, a high-performance modular framework for ptychographic reconstructions developed at the Paul Scherrer Institute, but which has been tested at a variety of light sources including home sources and electron sources. Finally, Favre-Nicolin *et al.* (2020) announced an updated version of *PyNX*, a highly flexible operator-based toolkit for coherent X-ray imaging related tasks, from data simulation to analysis. The new version includes a complete overhaul of the ptychography-related code as well as increased use of GPUs for better performance.

There is no doubt that the Ptycho Developers 2019 workshop helped push forward the field and we hope to see future editions of the workshop as soon as we are able to gather together again. We very much look forward to more developments to broaden the scope and increase the speed, power and user-friendliness of ptychography.

The virtual special issue is available at https://journals.iucr.org/special_issues/2021/ptycho.

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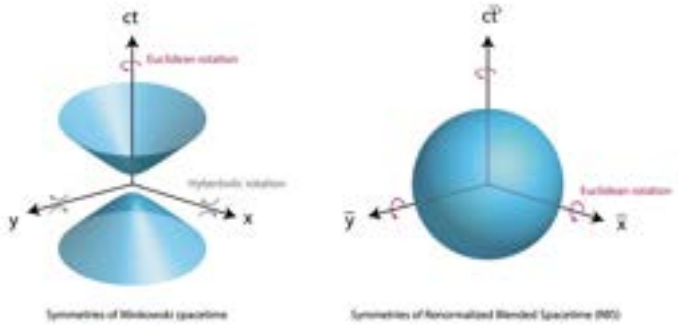
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This article was originally published in *J. Appl. Cryst.* (2021). **54**, 384–385.

RESEARCH NEWS

Spacetime crystals proposed by placing space and time on an equal footing



Conventional 3D Minkowski spacetime (two space axes, *x*, and *y*, and one time axis, *ct*, depicted in the left panel) is hyperbolic. Events in the past and future at a fixed spacetime distance from the origin form two sheets of the hyperbola as shown. A new reformulation of special relativity by Gopalan, called the renormalized blended spacetime (RBS), transforms the Minkowski hyperbolic sheets into a Euclidean sphere (right panel). This topological transformation allows one to express the symmetries of the Minkowski spacetime, namely a combination of Euclidean and hyperbolic rotations, all as pure Euclidean rotations. The relativistic physics content in both formulations is equivalent. (Figure courtesy of Hari Padmanabhan, The Pennsylvania State University, USA.)

A scientist studying crystal structures has developed a new mathematical formula that may solve a decades-old problem in understanding spacetime, the fabric of the universe proposed in Einstein's theories of relativity.

"Relativity tells us space and time can mix to form a single entity called spacetime, which is four-dimensional: three space axes and one time axis," said Venkatraman Gopalan, professor of materials science and engineering and physics at The Pennsylvania State University, USA. "However, something about the time axis sticks out like sore thumb."

For calculations to work within relativity, scientists must insert a negative sign on time values that they do not have to place on space values. Physicists have learned to work with the negative values, but it means that spacetime cannot be dealt with using traditional Euclidean geometry and instead must be viewed with the more complex hyperbolic geometry.

Gopalan developed a two-step mathematical approach that allows the differences between space and time to be blurred, removing the negative sign problem and serving as a bridge between the two geometries.

"For more than 100 years, there has been an effort to put space and time on the same footing," Gopalan said. "But that has really not happened because of this minus sign. This research removes that problem at least in special relativity. Space and time are truly on the same footing in this work." The paper, published in the July 2021 issue of *Acta Cryst. A*, is accompanied by a commentary in which physicists Martin Bojowald (Penn State) and Avadh Saxena (Los Alamos National Laboratory, NM, USA) write that Gopalan's approach may hold the key to unifying quantum mechanics and gravity, two foundational fields of physics that are yet to be fully unified.

“Gopalan’s idea of general relativistic spacetime crystals and how to obtain them is both powerful and broad,” said Bojowald. “This research, in part, presents a new approach to a problem in physics that has remained unresolved for decades.”

In addition to providing a new approach to relate spacetime to traditional geometry, the research has implications for developing new structures with exotic properties, known as spacetime crystals.

Crystals contain repeating arrangement of atoms, and in recent years scientists have explored the concept of time crystals, in which the state of a material changes and repeats in time as well, like a dance. However, time is disconnected from space in those formulations. The method developed by Gopalan would allow for a new class of spacetime crystals to be explored, where space and time can mix.

“These possibilities could usher in an entirely new class of metamaterials with exotic properties otherwise not available in nature, besides understanding the fundamental attributes of a number of dynamical systems,” said Saxena.

Gopalan’s method involves blending two separate observations of the same event. Blending occurs when two observers exchange time coordinates but keep their own space coordinates. With an additional mathematical step called renormalization, this leads to "renormalized blended spacetime."

“Let’s say I am on the ground and you are flying on the space station, and we both observe an event like a comet fly by,” Gopalan said. “You make your measurement of when and where you saw it, and I make mine of the same event, and then we compare notes. I then adopt your time measurement as my own, but I retain my original space measurement of the comet. You in turn adopt my time measurement as your own, but retain your own space measurement of the comet. From a mathematical point of view, if we do this blending of our measurements, the annoying minus sign goes away.”

This article is based on a news story appearing in Penn State News.

EDITORIAL

Crystal engineering in IUCrJ: from 'the' crystal structure to 'a' crystal structure



Gautam R. Desiraju

Crystal engineering continues to grow at an astonishing pace and one even discerns the appearance of offshoots that are beginning to develop as subjects in their own right, in the same way that crystal engineering itself developed as an offshoot of supramolecular chemistry in the 1990s. **IUCrJ** has successfully captured some of these latest developments in the 30 odd papers published in the journal in 2020 and early 2021. These papers encapsulate the idea of 'structure' as applied to molecular organic and metal–organic crystals. What is 'structure'? How does 'structure' relate to property? As one becomes more adept in designing structures, does one develop a parallel felicity in designing properties?

Until the end of the 1980s, the focus in small molecule crystallography was the determination of the unknown crystal structure of a molecular solid, which was typically obtained as a stable, well formed crystal that diffracted well and wherein the solution and refinement of the structure, in other words the (*x*, *y*, *z*) of all the constituent atoms and their atomic displacement parameters (ADPs), constituted the entirety of the exercise. The story, as it were, ended with obtaining these (*x*, *y*, *z*) and ADP values and given the emphasis then on the molecular structure of an organic compound, the exercise was deemed to be over when crystallography confirmed or, in some cases, corrected the molecular structure as obtained by chemical and spectroscopic methods. This was 'the' crystal structure (Clegg, 2021).

Starting with the 1990s, a new story started, one that began rather than ended with the (*x*, *y*, *z*) of all the atoms in the unit cell, and thus began the modern version of the subject of crystal engineering, a term that was coined in the mid-1950s but used in a limited and restricted sense until the 1990s. Why does a molecule crystallize the way it does? Why does a molecule not crystallize in some other way? Why do some molecules form good crystals and others not? Why do the habits of molecular crystals show so much variability? And most important, why do some compounds crystallize with more than one crystal structure – polymorphism?

Polymorphism (Broadhurst *et al.*, 2020; Shi *et al.*, 2020) was the beginning of a new theme that conceives of a structure determined by crystallography as just that: a structure. Any given pure chemical compound can have more than one stable crystal structure. Gradually, this idea of a one-to-many correspondence between molecular and crystal structure extended to a compound having metastable and transient crystal forms as well, and finally *in silico* forms that were still to be isolated experimentally. This breakdown of the one-to-one correspondence between molecule and crystal also occurred in the reverse direction. Different molecules could have what is essentially the same crystal structure, isomorphism (Ranjan *et al.*, 2020), and so an *in silico* structure of a given compound, which might not have been realized experimentally, might well be seen experimentally in a closely similar analog. For

example, chloro groups could be replaced by methyl or bromo groups, or even by a nitro group; an ethene linkage could be replaced by a sulfur atom; an (*R*)-*sec*-butyl group could be replaced by an (*S*)-*sec*-butyl group, and so on. In the end, things that were known earlier, were rediscovered in a new context, a not uncommon occurrence in science. And so, by using the device of solid solution formation (Verma *et al.*, 2020), one could engineer the crystal structure of a given compound into a computer-generated structure through solid solution formation with a congener that displays the desired structure experimentally in its pure state. Effectively, the idea could be extended: a two-component crystal could have the crystal structure of a single-component crystal. All these 'structures' of a single organic compound effectively constitute the structural landscape. Any structure determined in the laboratory with crystallography is a data point in this landscape. A compound is associated not with one unique crystal structure but with many. Any of these is 'a' crystal structure in this landscape. And thus, crystal engineering facilitated this conceptual shift in thinking from *the crystal structure* to *a crystal structure*.

One of the papers published in **IUCrJ** in 2020 puts this in a colourful way. Talking about olanzapine, Reutzel-Edens & Bhardwaj (2020) refer to their report as 'painting what is a partial picture of the amazingly complex crystal chemistry of this important drug molecule'. Yes, a landscape needs to be painted and what one needs in the palette is a combination of high throughput crystallization and high throughput crystallography. What we need are databases not of crystal structures but of crystal landscapes. Crystal structure prediction (CSP) should graduate into developing a library of landscapes. *In silico* structures and virtual supramolecular synthons are already a reality in the armoury of the crystal engineer and so the subject advances.

IUCrJ published 30 papers in the Crystal Engineering and Chemistry section in 2020 out of the total of 165 in this section since the inception of the journal in 2014. **IUCrJ** published 130 papers in all sections last year as compared to the 650 that the journal has published in its entirety since its inception in 2014. So, the chemistry component in 2020 is just about its running average across all years, but it must be noted that we have more sections now than we did in 2014. The papers in our section continue to contribute their share to reaching an impact factor of 5.401 in 2019, which is an impressive increase over the previous year.

In keeping with the theme of 'structure' a number of papers dealt with cocrystals both in the pharmaceutical context (Fatima *et al.*, 2020; Ranjan *et al.*, 2020) and in a more general sense (Rajkumar & Desiraju, 2021). Other areas addressed included charge density (Chodkiewicz *et al.*, 2020; Sanjuan-Szklarz *et al.*, 2020; Duverger-Nédellec *et al.*, 2020), phase transformations (Smets *et al.*, 2020; Rekis *et al.*, 2021), crystals obtained under exotic conditions (Buganski & Bindi, 2021; Hu *et al.*, 2020; Németh, 2020), and metal-organic frameworks (MOFs) (Yadava *et al.*, 2020; Park *et al.*, 2020) apart from core papers on supramolecular synthons and synthetic strategy.

I would like to see more papers in this section in 2021–2022 as the journal gradually moves into the latter part of its first decade. Publishing in **IUCrJ** is prestigious even as the relevance of open access continues to strengthen and grow. The Co-editors in this section have contributed significantly especially with the trials and tribulations of the pandemic that we seem to be getting through. Publication times have not been seriously affected and I owe all of them a big thank you.

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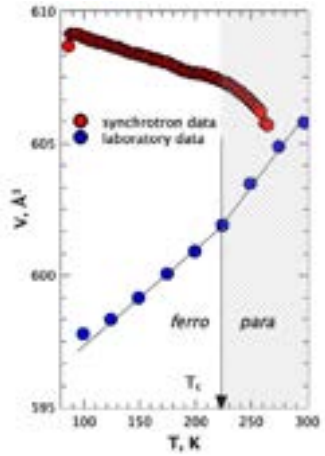
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This article was originally published in *IUCrJ* (2021). **8**, 148–149.

COMMENTARY

Impact of synchrotron X-ray radiation damage on a molecular ferroelectric crystal



Ines E. Collings

With the increasing use of third- and fourth-generation synchrotrons, the small-molecule crystallographic community has recently begun to assess the radiation damage impact of high intensity beams on samples. This is especially important for *in situ* studies, where the material of interest is investigated as a function of various external conditions and measured multiple times. If the sample is changing not only as a function of applied stimulus, *e.g.* temperature or pressure, but also with the duration of X-ray irradiation, how can these two effects be separated? Furthermore, if the sample does exhibit radiation damage, will this reduce or enhance material functionality; in other words, could we begin to use radiation to fine-tune properties on the micron to nanoscale?

In the article by Bogdanov *et al.* (2021) published in the June 2021 issue of *Acta Cryst. B*, the authors tackle these questions with their study on the molecular ferroelectric crystal glycinium phosphite (GPI), which has the chemical formula $NH_3CH_2COOH{\cdot}H_2PO_3$. At the European Synchrotron Radiation Facility (ESRF), they performed variable-temperature single-crystal diffraction measurements in fine-temperature steps to fully characterize the order parameters and strains involved in the ferroelectric transition of GPI. Since the ferroelectric behaviour is associated with an ordering of light hydrogen atoms and rearrangement of hydrogen bonds, single-crystal diffraction is favoured over the faster powder diffraction measurements. However, a deterioration of the data quality is observed upon cooling, manifested by a suppression of scattering at high angles and an increase of atomic displacement parameters for all atoms. Since this suggests the possibility of radiation damage, the authors complemented their synchrotron study with a similar experiment using a laboratory diffractometer. Most striking is the completely different thermal expansion response of GPI upon cooling. In the laboratory experiment, a volume contraction of 1.2% is observed upon cooling to 100 K, in contrast to the volume expansion of 0.4% recorded in the synchrotron study. Thus, two effects are acting on the GPI crystal in the synchrotron study: the first is the influence of cooling, and the second, more dominant, impact is that of the radiation damage. Even though the effects of radiation damage are reduced at low temperatures (Goeta & Howard, 2004), the GPI crystal exhibits a steady increase in volume that is correlated to the duration of synchrotron X-ray irradiation.

The X-ray-induced volume expansion in GPI is thought to be related to the formation of PO_3^{2-} radical defects (Vanishri *et al.*, 2007). However, such structural defects are hard to identify using X-ray crystallography since defective-GPI and GPI would only differ in the occupation of hydrogen atoms. Indeed, despite the discrepancy in the unit-cell dimensions, the structural models show very similar

atomic positions in both synchrotron and laboratory data sets. Moreover, Bogdanov *et al.* (2021) show that the paraelectric to ferroelectric transition temperature of GPI at 225 K remains unchanged for both GPI crystals measured at the synchrotron and in the laboratory. Further studies are needed to assess whether defective molecular ferroelectrics, with enlarged volumes from X-ray irradiation, would give rise to new functionality.

Whether we seek to avoid or utilize radiation damage in our structural research, what is clear is that a greater understanding of its impact and ways to control it are needed. Already, we are heading in this direction, with a greater number of systematic studies on radiation damage (Christensen *et al.*, 2019). However, the diversity in small-molecular systems makes it very challenging to propose general guidelines for radiation impact, as is documented in the field of macromolecular crystallography (Garman & Weik, 2017; Garman & Weik, 2019). While synchrotron beamtimes will likely remain dedicated to understanding the material and its functionality, additional good practices can be implemented to monitor the role that radiation can play. This can include several re-measurements on the same sample position and conditions after a given time of irradiation, in addition to measurements on different sample positions that were not exposed to the beam, as was performed in the investigation by Coates *et al.* (2021). Another strategy, as highlighted by Bogdanov et al. (2021), is the use of complementary X-ray laboratory or neutron data, where the radiation damage is substantially less than that from synchrotron sources, to disentangle the effects of radiation damage and material behaviour.

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This article was originally published in *Acta Cryst.* (2021). B**77**, 307–308.

COMMENTARY

New node on the block for organic solid-state chemists: rtct-tetrakis(pyridin-4-yl)cyclobutane

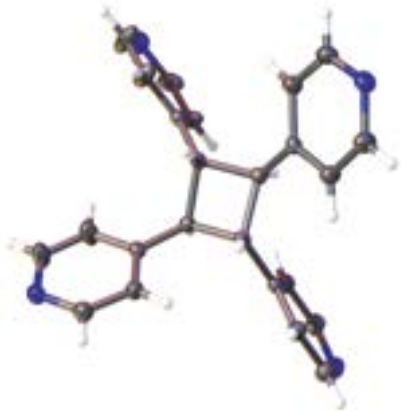


Figure 1. Rendering of the asymmetric unit of **rtct-TPCB**. Anisotropic displacement ellipsoids have been set at the 50% probability level.

Eric W. Reinheimer

As chemists and crystallographers, growing a crystal and obtaining the structure of a synthesized molecule is often considered the dénouement of a sometimes long and often arduous process. To peer into the atomic world and view the three-dimensional model of a molecule that might determine future research projects is the greatest reward for these interminable hours in the lab. While the molecules and the conclusions may outlast your graduate, postdoctoral, or professional career, are all syntheses and applications truly complete with that crystal? Is that the end or can more be done?

Seminal work by Schmidt and co-workers in 1971 signaled the advent of *organic solid-state syntheses* and demonstrated that even within a single crystal additional synthetic work was possible. In their report, Schmidt reported that *trans*-cinnamic acid and its derivatives were capable of undergoing a [2 + 2] photodimerization to generate functionalized cyclobutanes *post*-crystallization (Schmidt, 1971). As a direct result of this observation, the authors ultimately proposed the 'Topochemical Postulate', which hypothesized that olefin-containing molecules within 4.2 Å of one another can undergo a photodimerization in the solid state.

While cinnamic acid crystallizes in such a way that the olefin is rendered photoactive, a far greater number of olefin-containing molecules as a consequence of their crystal packing are photostable and are unable to undergo a photodimerization reaction. Pioneering research in crystal engineering has demonstrated that in order to perpetuate the onset of photoreactivity in these latter instances, the use of templates is required to align the reactant molecules in order to photoreact (Gan *et al.*, 2018). Numerous supramolecular interactions, such as hydrogen and halogen bonding, argentophilic and aurophilic interactions, along with others, have proven to be reliable in the formation of reactive solids containing olefins that are photostable as a single-component solid. This templated approach to solid-state synthesis has yielded novel molecules (i.e. cyclophanes and ladderanes) whose yield is prohibitively low when prepared through a standard solution-based approach. The progress of the solid-state syntheses can be followed by ^{1}H NMR as the disappearance of olefinic H atoms coupled with the concomitant appearance of H atoms from cyclobutane moieties signals the onset of photoproduct formation (Campillo-Alvarado *et al.*, 2019; Chanthapally *et al.*, 2014; MacGillivray *et al.*, 2000; Pahari *et al.*, 2019;

Sezer *et al.*, 2017; Volodin *et al.*, 2018).

Even though the solid-state syntheses of functionalized cyclobutanes have yielded some very aesthetically appealing molecules and crystal structures, further synthetic efforts utilizing these systems can be undertaken with the rational design of highlyconnected networks. Careful consideration of both the nodes and linkers is essential given their direct influence on the topology of the network (Jiang et al., 2018). Functionalized cyclobutane molecules generated using the [2 + 2] photodimerization reaction have been successfully incorporated as nodes within these connected networks. A survey of the literature has demonstrated that the most studied photoproduct within these networks is the *rctt*-isomer of tetrakis(pyridin-4-yl)cyclobutane (***rctt*-TPCB**), which has functioned as a node in both metal-based and molecular networks (Li *et al.*, 2014; Blake *et al.*, 1997; Oburn *et al.*, 2020; Baldrighi *et al.*, 2010). While the *rctt*-isomer of tetrakis(pyridin-4-yl)cyclobutane is the most-often utilized, a second isomer (***rtct*-TPCB**) is known and considered to be more thermodynamically stable. The H_2SO_4-catalyzed procedure to prepare the *rtct*-isomer from its *rctt* homologue in dimethyl sulfoxide (DMSO) was first outlined in 2010 (Peedikakkal *et al*., 2010); however, no single-crystal structure of ***rtct*-TPCB** or a structure demonstrating that it could, like ***rctt*-TPCB**, be incorporated into a purely molecular network was reported.

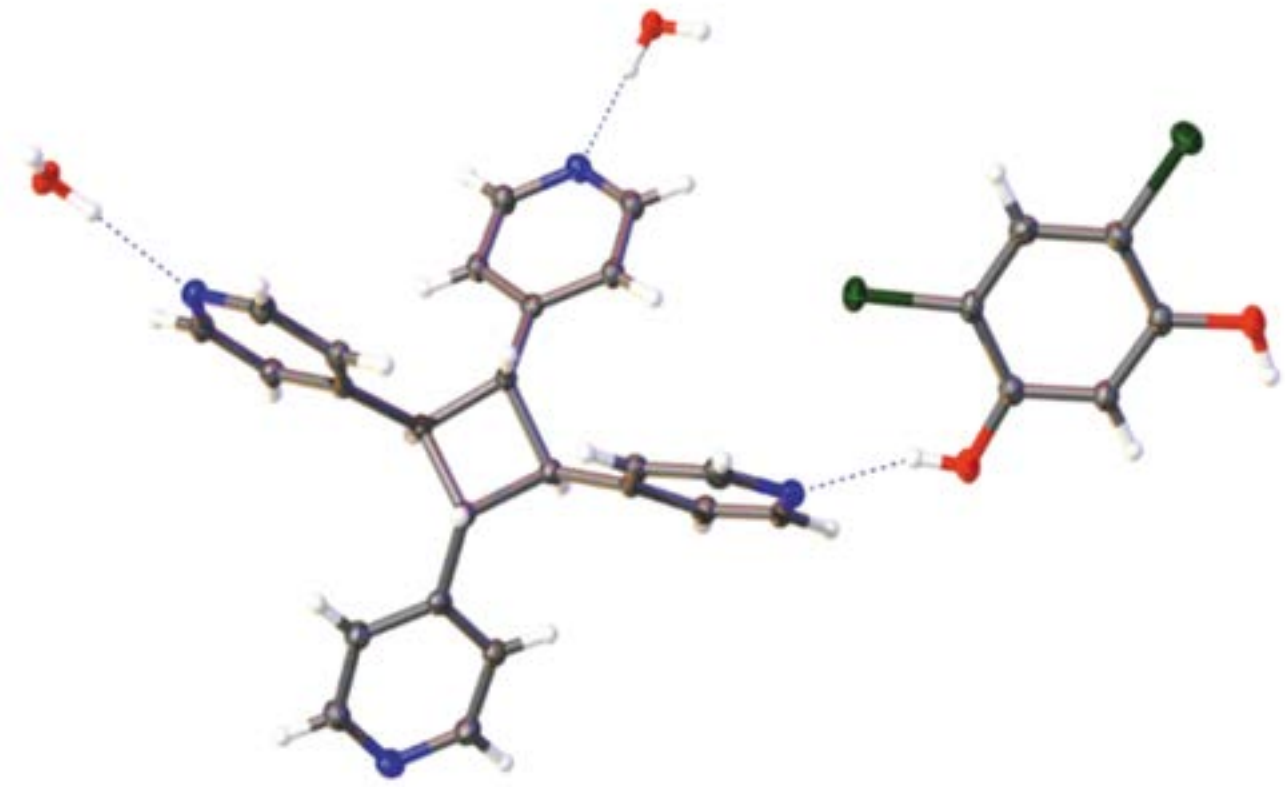


Figure 2. The functionalized cyclobutane **rtct-TPCB** functioning as a hydrogen-bond-accepting three-connecting node by engaging in O—H...N hydrogen bonding. Anisotropic displacement ellipsoids have been set at the 50% probability level.

Thanks to the recent report by the Groeneman research group, the lack of a structure for the ***rtct*-TPCB** isomer (Fig. 1), as well as structural proof that it can function as a node in a molecular network, has been clarified for the solid-state and crystal engineering communities (Santana *et al.*, 2021). Within the extended structure, ***rtct*-TPCB** functions as a three-connecting node by accepting three O—H...N hydrogen bonds with included water molecules and 4,6-dichlororesorcinol (Fig. 2). This type of packing arrangement for the node is required to form the ornate honeycomb topology (Fig. 3). Ultimately, it was found that the honeycomb network formed a three-dimensional network through the onset of C—H...N hydrogen bonding between a H atom on the cyclobutane and the final pyridyl N atom on a neighboring ***rtct*-TPCB**.

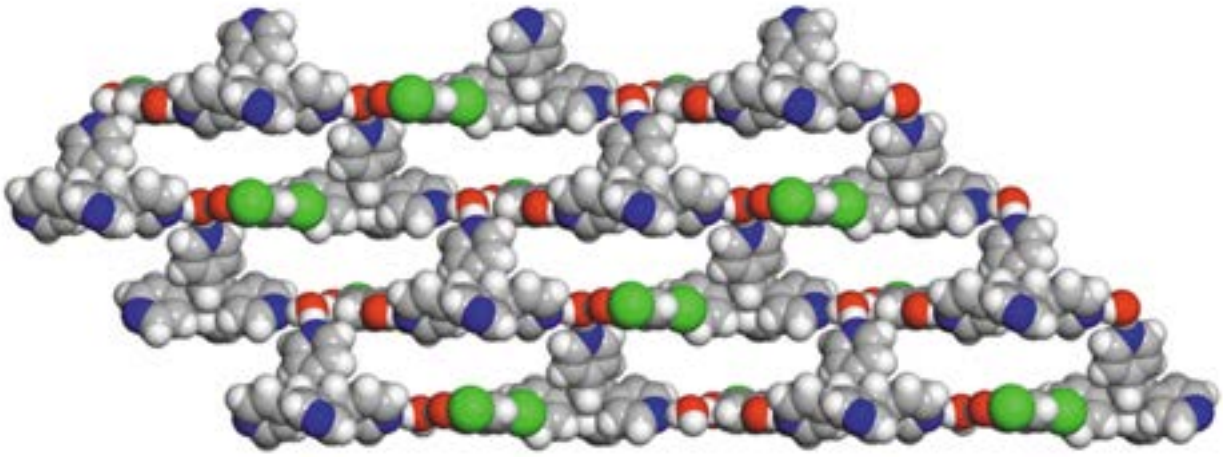


Figure 3. Extended view of the honeycomb network illustrating the observed channels.

Due to this highlighted work, we now know the symmetry and unit-cell parameters of the functionalized cyclobutane **rtct-TPCB**, as well as a multicomponent solid containing it. What other networks and topologies are possible? With the facile, acid-catalyzed procedure to generate other regioisomers, it is exciting to envision additional regioisomers of other functionalized cyclobutanes yet to be reported. The authors are to be applauded for finally addressing the paucity of information on this under-utilized isomer and shedding some light on it.

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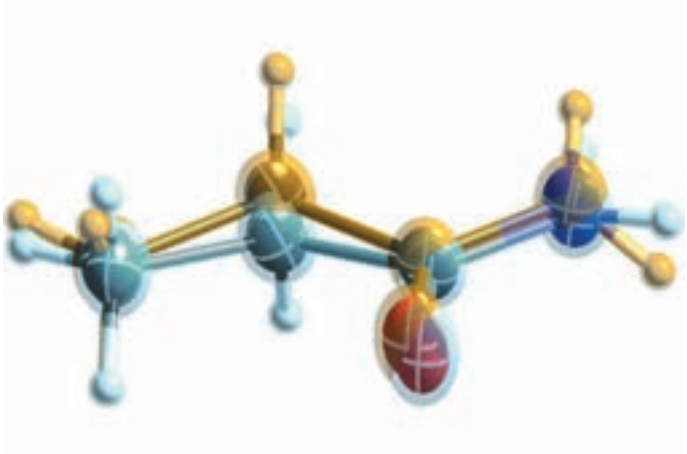
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This article was originally published in *Acta Cryst*. (2021). C**77**, 123–124.

COMMENTARY

Towards a better understanding and improved refinement of disordered crystal structures



Peter Müller

Single-crystal X-ray diffraction, the gold-standard of structure elucidation, has become well established, even mainstream, and in every respect a mature method. Most challenges of routine structure determination can be considered overcome and even mostly automatized. Modern diffractometer software determines unit cells and designs data collection strategies with minimal user input. Similarly, the phase problem is solved and space groups are determined fairly automatically by software, and most routine structure refinements can be performed with a few clicks of the mouse in just minutes. Even twinning has lost its terror through the power of computing, perhaps with the exception of reticular-merohedral twins. The one remaining issue in routine crystal structure determination is the refinement of disorder. An appreciable amount of crystal structures show at least some disorder, if only in the solvent part, and crystallographers regularly spend most of their time tackling disorders, some of which can take days to parameterize properly.

A new approach towards describing, understanding and modelling disorders has been offered by Birger Dittrich in the March 2021 issue of **IUCrJ** (Dittrich, 2021). Dittrich introduces the useful term 'archetype structure', which he defines as each distinguishable conformation of the molecule(s) in a crystal structure. A structure with just one simple two-component disorder gives rise to two archetype structures, one consisting of all ordered atoms plus the atoms of one disorder component, the other archetype consists of all ordered atoms plus the atoms of the other disorder component. A structure with more disorders gives rise to a larger number of archetype structures: two independent two-component disorders lead to four archetype structures, three independent two-component disorders to eight, and so forth. The crystal structure is then the superposition of all archetype structures.

Dittrich's method takes each archetype structure and individually optimizes it computationally by means of a 'molecule-in-cluster' method (Dittrich, Chan *et al.*, 2020), where the geometry of a molecule undergoes an energy minimization while taking into consideration a cluster of surrounding molecules as arranged in the crystal packing. From the optimized geometries, Dittrich derives restraints that he uses to assist a conventional least-squares refinement, the results of which can significantly improve the fit of the molecular model to the diffraction data. Constraints are automatically generated for the displacement parameters of proximate atoms to avoid over-parameterization.

The geometry restraints derived from the optimization step are, necessarily, direct restraints where set target values are specified for interatomic distances and angles. This is in contrast to relative restraints

(also known as similarity restraints) where parameters within a structure are related to one another rather than to specific target values. Direct restraints are generally based on 'outside information', that is from methods unrelated to the actual diffraction experiment and the crystal structure at hand. Therefore, direct restraints are sometimes considered less elegant and relative restraints are typically preferred for the refinement of disorders (Müller, 2009). In the case of Dittrich's method, however, the target values are not taken from some table found in a textbook, but are derived computationally, based on the actual crystal structure, which largely negates the arguments against direct and for relative restraints.

With larger structures and especially with structures suffering several independent disorders, Dittrich's molecular models can become bulky. Describing an ordered atom as the overlay of two, four or even more identical positions with individual occupancies adding to 100% requires the use of constraints to allow convergence of the refinement. This is not a problem, however, since constraints, by their very nature, eliminate refined parameters and there is no practical difference between an atom described with three positional and nine thermal parameters, and the same atom split into several sites that are constrained to exhibit identical coordinates and anisotropic displacement parameters.

In addition to showing a way of improving the fit of disordered molecular models to the diffraction data, Dittrich's paper also contributes to the general understanding of the disorder phenomenon. For example his four common-sense requirements for disorders are helpful: disorder can only occur if (*a*) the disorder components overlap well (*i.e.* they fit inside the same 'shrink-wrap envelope'), (*b*) the charge distribution is similar for all components (*i.e.* the electrical potential on the surface of the envelope is roughly the same for all components), (*c*) inter- and intramolecular contacts (such as, for example, hydrogen bonds) made by all components are similar, and (*d*) the conformational energies of the individual disorder components are not too different from one another. In addition, Dittrich places disorder in context with molecular conformation, space group symmetry and diffuse scattering, which links many aspects of crystal structure determination in a way that has practical implementations, thus transcending mere intellectual exercise.

Finally, Dittrich's method allows for an interesting way of distinguishing static from dynamic disorder. Theoretically, the former is introduced during crystallization and cannot be influenced by the temperature during the diffraction experiment while the latter corresponds to actual motion in the crystal, which can be 'frozen out' or at least reduced at lower temperatures. The above-mentioned energy requirement, combined with the energy differences derived from the individual molecule-in-cluster optimizations, can help in classifying a disorder as static or dynamic and even have implications for understanding and possibly predicting polymorphism, which is of vital importance in the world of pharmacology (Dittrich, Sever & Lübben, 2020).

It remains to be seen how and when this method will be implemented by programmers and accepted by the crystallographic community. In many cases, a carefully and expertly executed conventional modelling of disorders using mostly similarity restraints for geometry and anisotropic displacement parameters is not significantly inferior to the results of Dittrich's method; however, the molecule-in-cluster approach offers a new and relatively easy to use tool that will likely be considered a standard method before too long.

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This article was originally published in *IUCrJ*, **8**, 150–151.

COMMENTARY

Increasing complexity: structural equivalence and combinatorial approaches to preparation of high order cocrystals

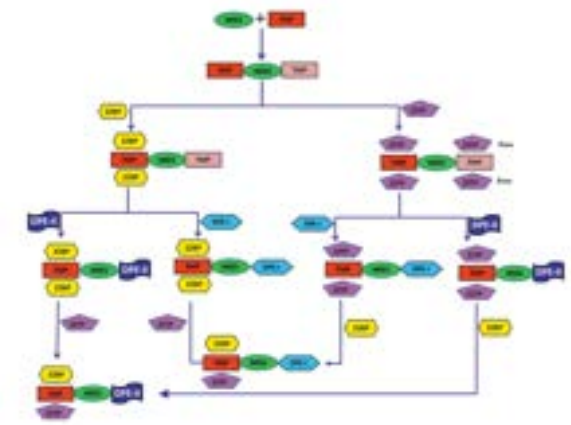


Figure 1. Schematic for the construction of five-component cocrystals. Reproduced from Rajkumar & Desiraju (2021).

Susan A. Bourne

Ever since Gerhard Schmidt (1971) proposed that a better understanding of packing principles of organic molecules would lead to predictable reactivity in their crystalline state, the topic of crystal engineering has offered the intriguing prospect of controllable functional design in the solid state. Revitalized in the 1980s by, among others, Desiraju (1989), crystal engineering has undergone explosive growth in the early years of the 21st century. This is evident in the growing number of papers on the topic in **IUCrJ** (Desiraju, 2020).

From early work in the design of molecular crystal structures (MacGillivray *et al.*, 2000; Bhogala *et al.*, 2005) crystal engineering has grown and branched to include polymorphism (Broadhurst *et al.*, 2020), mechanical properties (Karki *et al.*, 2009) and improved properties of pharmaceutically or biologically active solids (Mannava *et al.*, 2021). The latter is often achieved through the formation of multi-component crystals including cocrystals. Cocrystallization has become so familiar a concept that some authors now refer to ‘cocrystal engineering’ (for example Kumar *et al.*, 2020; Ye *et al.*, 2020).

While many have explored this area, and have produced binary cocrystals for multiple applications, higher dimensional cocrystals remain relatively rare. But the challenge is an exciting one, particularly for the design of multicomponent crystals combining several active pharmaceutical ingredients (APIs). Multidrug cocrystals can be expected to produce a synergistic therapeutic effect, while crystal engineering of the molecular packing and dominant non-covalent interactions may allow the modulation of physicochemical properties to improve drug formulation and delivery. To date, the only approaches to succeed in preparing ternary API solids have been the ‘drug–bridge–drug’ strategy developed by Liu *et al.* (2018) and the ionic cocrystallization method (Mazzei *et al.*, 2019; Song *et al.*, 2020). However, the former is limited by the difficulty of identifying a suitable bridge, while ionic cocrystallization involves coordination bonds to a metal ion ‘glue’ holding the APIs together.

Increasing the number of components in a crystal vastly increases the complexity of the synthesis. An understanding of the conditions required for the reliable and reproducible formation of higher order multicomponent crystals remains elusive. This is the challenge addressed in an article in the March 2021 issue of **IUCrJ** (Rajkumar & Desiraju, 2021). Earlier work by one of the authors suggested a crystal

engineering strategy to achieve quaternary and quinary cocrystals, based on the structural inequivalence of some of the chemical constituents in the cocrystals (Mir *et al.*, 2016). This postulates that, if a given molecule is found in two different structural environments in a lower order cocrystal structure, these features can be exploited to increase the number of components in a new solid structure. This approach has been used to obtain ternary and quaternary cocrystals. To extend to higher order cocrystals, the authors then invoke a combinatorial approach, using the persistence of a molecule in different solid forms to select the best interactions from a range of possibilities.

The strategy is illustrated by the design and synthesis of quaternary and quinary cocrystals of model components 2-nitroresorcinol (NRES), tetramethylpyrazine (TMP), either 2,2'-bipyridine (22BP) or 2,2'-bithiophene (22TBP), and 1,2-di(4-pyridyl)ethane (DPE). A flowchart approach (see Fig. 1) is used to identify the structural inequivalences which can be exploited to extend the chain, as opposed to those that are ‘dead ends’ in the synthetic sequence. The proof of concept is shown by the synthesis of several five-component solid solution cocrystals obtained by mechanochemical combination of a quaternary cocrystal with an additional component.

Aside from the intrinsic beauty of the resulting structures, this is an elegant example of a retrosynthetic approach being applied to systems built on noncovalent interactions. While hydrogen bonding is the dominant interaction in the examples reported here, the approach lends itself to use with other forms of supramolecular interactions such as halogen bonding, coordination bonding and π–π interactions.

An algorithmic approach, such as that described here, is a positive step in the direction of developing an understanding of non-covalent interactions and their influence on synthesis of multicomponent crystals. This is essential for the advancement of crystal engineering into the new era of true design of functional materials.

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This article was originally published in *IUCrJ* (2021) **8**, 152–153.

IUCR ACTIVITIES

New Teaching edition of International Tables for Crystallography: Crystallographic symmetry



The Brief Teaching Edition of Volume A of *International Tables for Crystallography* (*IT*) was first published in 1985. Edited by Theo Hahn, it found its way into many a classroom or laboratory as a convenient and inexpensive tool for researchers and students. Now in its sixth edition, the publication has a new Editor, Mois I. Aroyo, and – although still a practical size – has dropped the "Brief" from its title. The following description of the contents is extracted from the Editor's Preface.

This Teaching Edition (hereafter referred to as the TE) is the successor to the *Brief Teaching Edition of Volume A* (which was last revised in 2005), although new material, substantial revisions and reorganisation of the text has resulted in a book that is quite different in structure and content to its predecessor. It focuses on the particular topic of symmetry, owing to its fundamental role in crystallography, and provides a unified and coherent introduction to the symmetry information found in three volumes of *International Tables for Crystallography*: the basic crystallographic data for the plane and space groups in Volume A (*IT* A), the symmetry relations between space groups treated in Volume A1 (*IT* A1) and the subperiodic-group data found in Volume E (*IT* E). It also introduces the Symmetry Database, which forms part of the online version of *International Tables for Crystallography* at https://it.iucr.org.

This Teaching Edition is designed for graduate (and postgraduate) students, and young researchers who have some awareness of the basics of symmetry and diffraction and who need to use crystallographic symmetry methods in their work. The TE can thus serve as an interface between elementary crystallography textbooks and the texts in *IT* A, A1 and E. Sufficient up-to-date and accessible references to further specialized sources are provided for those who need to go deeper into the subject, and to textbooks and basic crystallographic literature that could be helpful for those who need a course in basic crystallography or introduction to the mathematics that is required. The fruitful combination of tables for practical use and a didactic introduction to symmetry makes this TE a handy tool with which researchers and students can familiarize themselves with the use of crystallographic symmetry and its practical applications.

Part 1 of the TE is based on the material of the introductory part of the sixth edition of *IT* A, with additional explanations and illustrative examples to provide the reader with practical experience in the use of crystallographic symmetry data. The fundamental concepts of group theory, focusing on those properties that are of particular importance for crystallography, are introduced in the first chapter. The matrix formalism, which provides efficient instruments for the analytical description of crystallographic

symmetry, is the subject of Chapter 1.2. Chapter 1.3 offers an introduction to the structure of space groups and their various classification schemes, focusing on the classifications into space-group types, geometric crystal classes and Bravais types of lattices. Chapter 1.4 deals with various crystallographic terms and the symbols used to present the symmetry data in the space-group tables of *IT* A. The purpose of Chapter 1.5 is to provide the mathematical tools needed to work out how crystallographic data are transformed if the coordinate system is changed. Chapter 1.6 offers a detailed presentation of methods for determining the symmetry of single-domain crystals from diffraction data. The tables of general reflection conditions shown in this chapter also include 'diffraction symbols' (previously known as 'extinction symbols'), and four examples of space-group determination from real intensity data are analysed. Chapter 1.7 deals with applications of crystallographic symmetry, including symmetry relations between space groups, as treated in *IT* A1, and subperiodic groups, as covered in *IT* E. Possible applications of the subperiodic groups in the description of crystal systems with rod and layer symmetry are discussed. Finally, magnetic groups, including magnetic subperiodic groups and magnetic space groups, are briefly introduced.

The material of Part 2 focuses on the presentation of the tables of crystallographic symmetry data found in Volumes A, A1 and E, including descriptions of the symbols and terms found in the tables and guides for their use. Representative sets of tables of varying complexity are included that will be useful for teaching about crystallographic symmetry. The guide to the space-group tables of Volume A is very similar to that given in Chapter 2.1 of *IT* A and is complemented by the tables for 7 of the plane groups and 35 of the space groups. In Chapter 2.2, the data for the maximal subgroups and minimal supergroups in *IT* A1 are described and illustrated using the examples of $P3_112$ (151) and $P3_121$ (152). The guide to the arrangement of the data specifying relations between Wyckoff positions in *IT* A1 uses the tables of $P6_3/mmc$ (194) and *Cmcm* (63) as examples. The subperiodic-group tables in *IT* E are described in Chapter 2.3 and are illustrated by the tables of the rod group $\not{p}mc2_1$ (No. 17), and of the layer groups $p2_1/b11$ (No. 17) and *pbam* (No. 44). Finally, Chapter 2.4 gives a brief description of the Symmetry Database, which provides online access to data for the crystallographic space and point groups that extend and enhance the symmetry information given in the printed editions of *IT* A and A1.

Commentary by Carolyn P. Brock, Editor-in-chief, *International Tables*

The first edition of the *Brief teaching edition of Volume A*: *Space-group symmetry* (the BTE) in 1985 contained complete descriptions of the 17 plane groups, 24 selected space-group examples, and those basic text sections of Volume A necessary for the understanding and handling of space groups (Parts 1, 2, 3 and 5). By simply copying parts of Vol. A the cost of the BTE was kept low for the benefit of students. Subsequent editions were unchanged except for revisions made to Vol. A (*e.g.* the change of group *Cmca* to *Cmce*).

While the low cost and small size of the *BTE* appealed to students and their teachers, many found the expertise needed to understand its text daunting. When Mois Aroyo became Editor of Vol. A he began to rethink the BTE, a process that lasted at least 13 years. The new *Teaching Edition* of 2021 (the TE) is still brief and low-cost, but it differs so much from the BTE that it is a new effort rather than a revision.

While working on the TE Mois participated as a lecturer/tutor in dozens of schools organized by the IUCr Commission on Mathematical and Theoretical Crystallography, where he gained an excellent understanding of students' needs. He then recruited some new authors and exercised a firm, but always very tactful, editorial hand. The result is a wonderful book that assumes much less prior knowledge and also introduces students to topics (*e.g.* magnetic groups, subperiodic groups, sub- and supergroup relationships, space-group determination by electron diffraction) that show crystallographic symmetry to be a field that is still under development. The TE also introduces the *Symmetry Database*, an electronic resource that is part of *International Tables*. A few pages of the TE are printed in color because the illustrations would have been difficult to understand in grayscale.

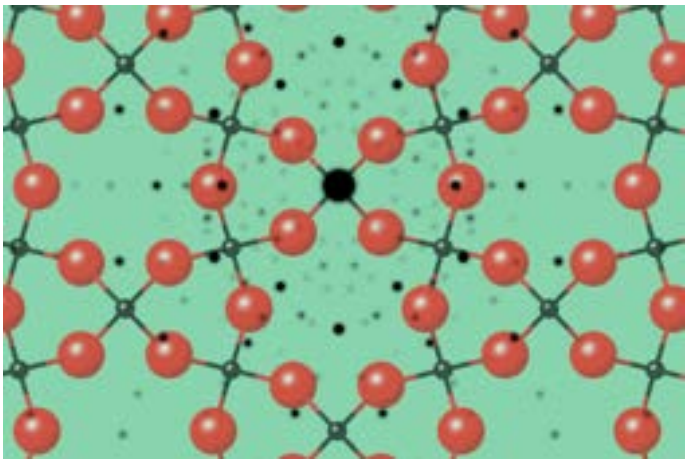
While no actual diagram of a magnetic space group is included in the TE, Danny Litvin’s very large compilation of the magnetic space groups (ISBN 978-0-9553602-2-0; 106 MB) is freely available *via* the IUCr website.

It is difficult to thank Mois Aroyo adequately for his many years of work on this very successful project.

The 250-page paperback (ISBN 978-0-470-97422-3) is available from Wiley at a cost of 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.

IUCR ACTIVITIES

Change to the definition of "crystal" in the IUCr Online Dictionary of Crystallography



Carolyn P. Brock

Definition
(The following applies to solids that, when illuminated by radiation having a wavelength on the order of Å, generate a three-dimensional diffraction pattern primarily characterized by discrete peaks. The entry for subperiodic crystal discusses crystals for which the diffraction pattern is instead primarily characterized by intensity maxima that are layers or rods.)

Historically, crystals were first defined in terms of their external morphologies, with the angular and symmetry relationships between their faces leading, in *ca* 1800, to the Law of Rational Indices. Over a century later the information from X-ray diffraction resulted in a definition based on the three-dimensional translational periodicity inferred from the periodicity of the array of Bragg peaks in the diffraction patterns of most crystals. More recently crystals have been discovered for which the array of Bragg peaks is not periodic in three dimensions so that the repeating atomic arrangement lacks translational periodicity in at least one direction. Currently there are two alternative (but theoretically equivalent) definitions of a crystal; both are based on the central idea of spatial order. One focusses on direct (or real) space, the other on reciprocal (or diffraction) space. The latter is more compact and elegant; the former can be easier to visualize.

Direct-space definition
A solid is a crystal if its atoms, ions and/or molecules form, on average, a long-range **ordered** arrangement.

In most crystals the arrangement is a periodic array that is governed by the rules of translational symmetry. In aperiodic crystals (incommensurate and quasicrystals) the arrangement is not periodic in three dimensions but is nevertheless still fully ordered, where the ordering follows particular mathematical rules.

Reciprocal-space definition
A material is a crystal if it has **essentially** a sharp diffraction pattern.

A solid is a crystal if it has **essentially** a sharp diffraction pattern. The word **essentially** means that most of the intensity of the diffraction is concentrated in relatively sharp **Bragg peaks**, besides the always present diffuse scattering. In all cases, the positions of the diffraction peaks can be expressed by

$$\mathbf{H} = \sum_{i=1}^{n} h_i \mathbf{a}_i^* \quad (n \geq 3)$$

Here $\mathbf{a}^*_i$ and h_i are the basis vectors of the reciprocal lattice and integer coefficients respectively and the number n is the minimum for which the positions of the peaks can be described with integer coefficient h_i.

The conventional crystals are a special class, though very large, for which $n = 3$.

[The adjective "known" reflects the fact that it is possible to envisage mathematically-based models that lie outside these definitions. See *e.g.* Grimm (2015).]

See also
- *Acta Cryst.* (1992), A**48**, 928 where the definition of a crystal appears in the **Terms of reference** of the IUCr Commission on Aperiodic Crystals.

Reference
Grimm, U. (2015). *Acta Cryst*. B**71**, 258–274. *Aperiodic crystals and beyond*

The new definition as of March 2021.

History of the definition of a crystal in the Online Dictionary of Crystallography

The IUCr Online Dictionary of Crystallography (ODC) was conceived by André Authier in 2005 as a project of the Commission on Crystallographic Nomenclature (CCN). Authier was the Editor of the ODC until 2009, when he was succeeded by Gervais Chapuis. For more information about the ODC see an article published in the *IUCr Newsletter* in 2019 here. That *Newsletter* article also includes some discussion of the definition of a crystal.

A definition of the term *crystal* created by the Commission on Aperiodic Crystals (CAC) was added to the ODC in 2009. At the core of that definition are statements that a crystal is "a material which exhibits **essentially** a sharp diffraction pattern" and "that most of the intensity of the diffraction is concentrated in relatively sharp **Bragg peaks**." There is no mention of a crystal being an array of atoms or groups of atoms that are periodic in three dimensions. The 2009 definition is a revision of a definition first given in the CAC section of the IUCr's 1991 Executive Committee report [see *Acta Cryst.* (1992). A**48**, 928].

Reasons for broadening the definition of a crystal

At the very end of the 2014 IUCr Congress and General Assembly in Montreal, I became Chair of the CCN because Mike Glazer, who had agreed to take over that role from Authier, had just been elected Vice President of the Union. The next day, three crystallographers approached me about revising the definition of a crystal in the ODC. They explained that the current definition, while correct and elegant, was a problem for teachers of crystallography. The focus on reciprocal space often makes the definition difficult for non-experts to understand; the translational periodicity that characterizes most crystals is easier for them to grasp.

During the year before the 2017 Hyderabad Congress, Mike Glazer (the member of the IUCr Executive Committee who had been assigned to the CCN) and I drafted an expanded definition. The proposed revision included the current definition *verbatim* but added some information about how the definition had changed with time and gave an alternative definition focused on direct space and mentioning translational periodicity.

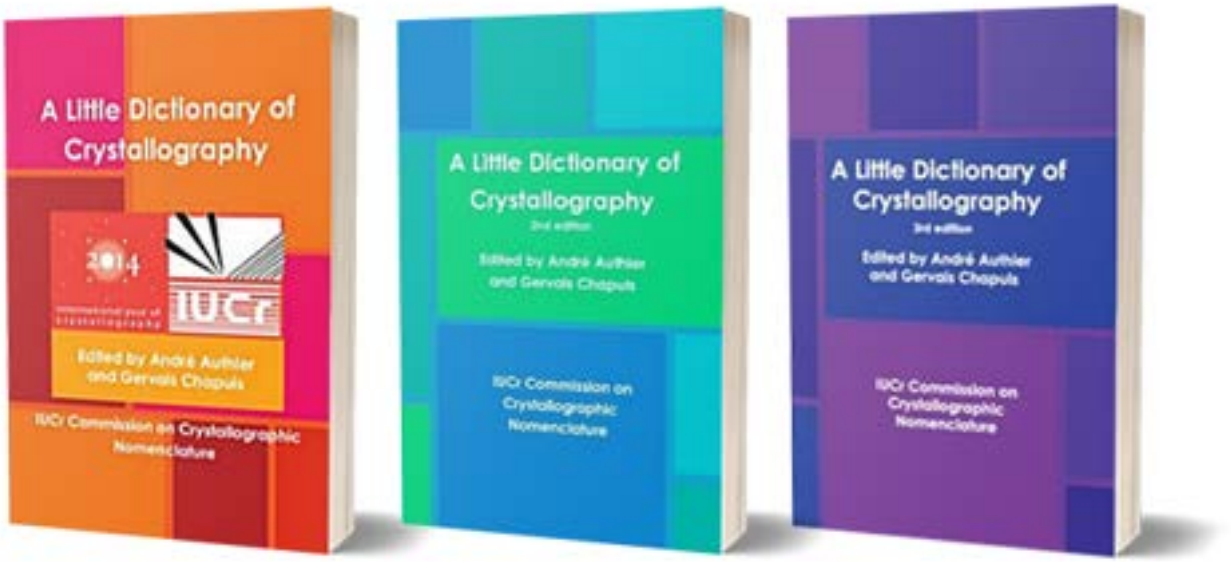
Because the ODC (https://dictionary.iucr.org/Main_Page) is run as a Wiki we could have simply uploaded the expanded definition, but we circulated it instead to a small number of crystallographers we thought would be interested. We discovered immediately that there would be serious objections to any change in the existing definition.

In 2020 Mike and I recruited Marjorie Senechal, a mathematician who was one of the original members of the CAC and was involved with developing the original 1992 definition. She suggested a small number of changes to our original proposal. Once those changes were made, she supported it.

The proposed definition was circulated to the 56 members of the CCN on 9 February 2021. The initial response led to a revised proposal being sent out on 23 February. Eventually 20 CCN members participated in the email discussion. When the 17 March deadline arrived, the 39 members of the CCN who voted (72% of those eligible) had all endorsed the proposal. The expanded definition was posted to the ODC website on 23 March.

Although the proposal was adopted, it was generally agreed that the proposal is an *interim* solution and should be considered further. Marjorie Senechal points out that the 1992 definition too was always intended to be an *interim* definition because the characteristics of atomic arrangements (other than the classically periodic ones) that give rise to sharp Bragg peaks were not known. She adds that while a great deal of work has been done on that problem, the answers found so far require more mathematical

background than should be expected of students consulting the ODC.



The new edition of the print version of the ODC (right) contains the latest definition of crystal. It can be obtained here.

Issues noted by CCN members during discussion of the proposal

The most basic problem noted during discussion of the proposal is that the purpose of the ODC is unclear. Is it the place where the crystallographic community puts its consensually-arrived-at precise definitions or a place where non-experts can find useful definitions? One member of the CCN wrote that

… the dictionary should be there to help and to facilitate, not (generally) to legislate and regulate.

but other CCN members have different views.

Another member pointed out that most definitions are compromises between generality and accuracy.

One physicist wrote:

I also think that if the objective is to make people less confused, then we should simply define ‘crystal’ as something with periodic long-range order. … On the other hand, if the objective is to make a precise definition of ‘crystal’ as anything with long-range-order, we can do that, but we should be more like mathematicians and define it precisely and not concern ourselves with whether the definition is intelligible to people or not.

It is understood by all that the definition of a crystal is intimately related to the definition of *order*, and especially *long-range order*, but defining order is not a simple task (see the *Newsletter* article referenced above).

One CCN member asked what “relatively sharp Bragg peaks” means. What about subperiodic crystals (especially polytypes and crystals that are mixtures of polytypes) in which the Bragg scattering is concentrated in rods? Another CCN member replied that crystallographers working with polytypes were consulted when the CAC definition was formulated.

No mention is made in the definition of the wavelength of the radiation that produces the diffraction pattern. If the wavelength is not similar to the spacings between scattering units there can be no diffraction pattern.

Definitions used in other fields and given in sources like Wikipedia may be at variance with the ODC definitions. To what extent should the IUCr be concerned? To what extent should IUCr journals require that authors use terminology accepted by the CCN? How is the answer to that question likely to affect the recruiting of new authors to IUCr journals?

There are many related terms for which definitions are needed in the ODC. Besides *order* (*long-*, *medium-* and *short-range order*) the list includes *ideally imperfect crystal*, *liquid crystal*, *metacrystal* and perhaps *paracrystal*. Also needed are definitions for *mosaic block* and for *disorder of the first and second kinds*. Definitions for *subperiodic crystal* and *stacking fault* were added to the ODC during the discussion of the proposal.

A structural biologist asked how things that look like crystals (transparent, plane faces) but do not diffract should be described (other than as “disappointing”).

The term *solid* appears in the real-space definition but *material* is used in diffraction-space definition. Is that inconsistency important?

Summary

While there are unresolved questions about the definition of a crystal, many members of the CCN wrote that a real-space definition mentioning periodicity is needed. Many also wrote that, while the proposed definition is imperfect, it seems to be the best that can be formulated now. In coming years there may be advances in how we think about structures that are ordered or disordered in one or more dimensions. The definition currently in the ODC will almost certainly be revised at some future time.

MEETING REPORT (IUCR SUPPORTED)

Sixth Conference of the Bangladesh Crystallographic Association



Altaf Hussain

The Executive Committee of the Bangladesh Crystallographic Association (BCA) decided in January 2020 to hold its sixth conference on 15–16 January 2021. Accordingly, the first flyer was circulated to educational and research organizations in Bangladesh and uploaded to the BCA website in the first week of February 2020. The Organizing Committee started applying to both national and international organizations for funds, and one such application was sent to the IUCr Calendar Committee in April 2020. Though the first case of COVID-19 in Bangladesh was identified in the second week of March 2020, we expected that the situation in January 2021 would allow for a real conference to take place as planned. However, the COVID pandemic situation worsened around the globe. The BCA then decided in October 2020 to hold its conference virtually on 15–16 January 2021. Accordingly, the second flyer and a poster were uploaded to the website, and circulated by normal post to different educational and research organizations in Bangladesh and electronically to many national, regional and international crystallographic societies. The BCA received responses, beyond its expectation, from the crystallographic communities of many countries confirming their participation in this two-day virtual conference.

The conference was attended by 148 faculties, scientists and graduate students from home and abroad. Of these 148 participants, 53 were female and 68 were young scientists (age below 35 years). A total of 113 abstracts were received; the programme and abstract book can be viewed here. To accommodate all these presentations, the inauguration session followed by three plenary lectures was held on the evening of 14 January 2021. Professor Sven Lidin, IUCr President, was the guest of honor at this session. He also delivered a plenary lecture during the conference. The conference included 12 technical sessions, and in those technical sessions 7 plenary lectures, 22 invited talks, 61 oral presentations and 23 posters were presented.

A postconference survey was arranged through a Google link. Many participants responded to this survey and mentioned that without a virtual format, the conference would probably not have had such a strong international flavor. Almost all the sessions were lively and completed on time with a good attendance though there was some buffering in internet connection, mainly from the participants' end. Prizes had been awarded for the best poster presentations, and it was suggested that organizers should present awards to the best oral presenters too.

The BCA gratefully acknowledges financial support from the IUCr and from two national organizations, and thanks all attendees for their kind participation. We are looking forward to cooperation and collaborations from crystallographic communities of the world in future events of the BCA.

Professor Altaf Hussain is President of the BCA and Chair of the Organizing Committee of the Sixth Conference of the BCA.

MEETING REPORT

Eleventh Belgian Crystallography Symposium BCS-11



Elise Pierson (Namur University, Belgium), winner of the IUCr Journals Poster Prize at BCS-11 for her poster "Deciphering the structural features of *Brucella melitensis* SerB, a single-ACT domain phosphoserine phosphatase".

Luc Van Meervelt

More than 100 participants took part in the Eleventh Belgian Crystallography Symposium BCS-11, which was held online on 17 March 2021 under the aegis of the Belgian National Committee for Crystallography. Due to the success of previous BCS meetings, the National Committee for Crystallography organizes this symposium at biennial intervals to promote contacts between Belgian scientists using diffraction and microscopy techniques for studies in the fields of condensed matter chemistry and physics, material sciences, earth sciences and life sciences. As one of the main objectives is to promote personal contacts, the symposium program includes flash and poster presentations in addition to plenary lectures.

During the opening lecture by Eriko Nango (Tohoku University, Japan), a nice overview was given of recent advances in recording a three-dimensional movie of the structural changes in bacteriorhodopsin at room temperature using time-resolved serial femtosecond crystallography at the SPring-8 Angstrom Compact Free Electron Laser SACLA. The minerals from alkaline rocks are the favourite study materials of Fabrice Dal Bo (University of Oslo, Norway). In comparison to most rocks, these materials are relatively enriched in alkali, halogens and high field strength elements. The complicated crystal chemistry of these minerals was illustrated by many examples such as eudialyte, steenstrupine-(Ce) and phosinaite-(Ce). The morning session was closed by nine flash presentations including three from our sponsors. During the lunch break a series of rooms were available for posters (24) and sponsors.



Speakers at BCS-11. From left to right: Eriko Nango, Fabrice Dal Bo, Martin Schmidt and Koen Janssens.

In the afternoon, new methods for structural investigations of nanocrystalline and amorphous organic compounds were introduced by Martin Schmidt (Goethe-Universität Frankfurt am Main, Germany). Many examples of the use of cross-correlation functions (FIDEL method) and the pair-distribution function (PDF) were given. Koen Janssens (University of Antwerp, Belgium) highlighted the use of chemical imaging tools such as macroscopic X-ray diffraction and X-ray fluorescence in the identification of pigmented materials such as lead white and ultramarine used in many 15th century paintings. These non-destructive techniques have been used during the restoration of the famous Ghent Altarpiece (Jan and Hubert Van Eyck).

At the end of the meeting, two prizes for the best flash presentations were awarded to Bram Muylemans (Leuven) and Thi Yen Hang Bui (Leuven) together with the IUCr poster prize to Elise Pierson (Namur) and the BCS-11 poster prize to Morgane Valentin (Liège). This BCS-11 meeting was sponsored by Bruker AXS, MiTeGen, Eldico Scientific, CGB-CBB and IUCr Journals.

NOTICE

Trust Science pledge



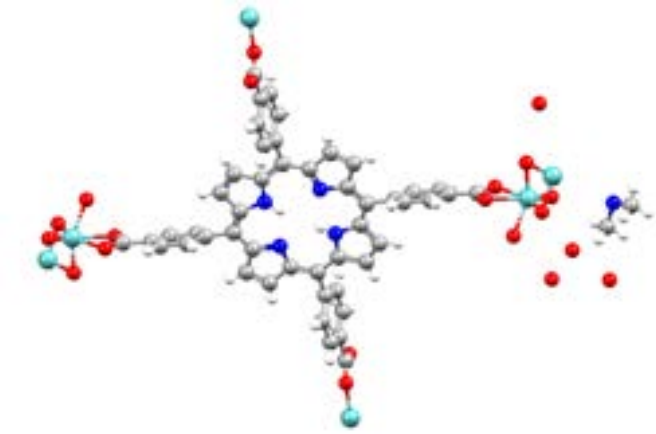
The International Day of Light (IDL) is held on 16 May every year, the anniversary of the first successful operation of the laser in 1960 by physicist and engineer, Theodore Maiman. First celebrated in 2018 by UNESCO, the IDL is a global initiative that provides an annual focal point for the continued appreciation of light and the role it plays in science, culture and art, education and sustainable development, and in fields as diverse as medicine, communications and energy. The broad theme of light allows many different sectors of society worldwide to participate in activities that demonstrates how science, technology, art and culture can help achieve the goals of UNESCO – education, equality and peace.

The message of IDL 2021 – Trust Science – encourages both scientists and the public alike to sign and support a declaration affirming their trust in science and the importance of public confidence in the scientific process. By signing the pledge to Trust Science, supporters of science including educators, scientists, engineers, healthcare professionals, Nobel Laureates, science enthusiasts, organisations, students and people worldwide are uniting to raise awareness of the importance of light science and technology in areas such as communications, sustainable development, climate action and healthcare.

To learn more, please click here.

NOTICE

MOF data set provides 10k structures free for academic research



The Cambridge Crystallographic Data Centre (CCDC) has made more than 10,000 metal–organic framework structures free to academics in the new CSD MOF Collection (Li *et al*., 2021).

"We have seen incredible growth in the number and variety of MOFs synthesised over the past 50 years, and now the variety of applications is growing too. We hope that access to this data collection will support research in this important field," said Seth Wiggin, Senior Scientific Editor at CCDC.

The CSD MOF Collection includes the crystal-structure data for 10,636 MOFs from the Cambridge Structural Database (CSD). In order to make the data more ready for computational calculations, the structures have been simplified to space group *P*1, and all guests have been stripped out to leave just the framework itself. The data are provided as single-block CIF files along with CSV indexes, allowing researchers to search and view the structures, or apply analytical or learning techniques to the data. The collection has been built to provide a simplified and focused data set for computational research, including just the 3D porous frameworks, rather than the complete collection of 107,980 MOFs in the full CSD with all the user-led filtering options available in the CSD Software Portfolio.

To build the collection, the CCDC worked with researchers at the Department of Chemical Engineering and Biotechnology, University of Cambridge, UK. Here the molecular mechanisms and applications surrounding porous materials like MOFs are researched, including their use in drug delivery systems, hydrogen storage and carbon capture.

"It's great to see experimentally derived structures from the CSD made available for free in this MOF collection," said David Fairen-Jimenez, who leads the Adsorption and Advanced Materials lab there. "The potential of MOFs is still being explored in a wide range of applications — and having this trusted data freely available will help to advance work further."

Indeed reports estimate that the global MOF market will grow by around 34% from 2021 to 2026, with big industry players like BASF and Strem Chemicals active in R&D. As this industry interest grows, fundamental research to understand properties, mechanisms and emerging applications of MOFs will be of increasing importance.

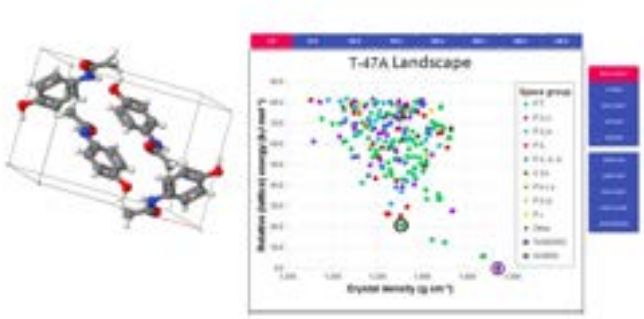
The CSD MOF Collection is available to download now from www.ccdc.cam.ac.uk.

Reference

Li, A., Bueno-Perez, R., Wang, S.-D., Wiggin, S., Ward, S. C., Wood, P. A. & Fairen-Jimenez, D. (2021). *Matter*, **4**(4), 1105–1107.

NOTICE

CCDC simplifies management of complex crystal structure prediction data



The Cambridge Crystallographic Data Centre (CCDC) announces the launch of *CSD-Theory* software for use in small-molecule pharmaceutical development. This latest addition to CCDC's software toolbox delivers easy storage, management, search and display of crystal structure prediction (CSP) results alongside experimental structures on a web-based platform and an API interface. The new software, developed in collaboration with six major pharmaceutical companies, will enable organisations to incorporate and share CSP knowledge earlier and more easily in small-molecule drug development — reducing risks and shortening the time to market.

Small-molecule therapeutics are regularly administered in crystalline, solid forms that undergo extensive testing to ensure stability, efficacy and safety. However, the 3D structure and packing of the crystalline form significantly impacts the properties of the therapeutic and, to further complicate matters, it is possible for a small molecule to have multiple crystal forms (known as polymorphs). These polymorphs have the same molecular structure, but different packing. Predicting alternative polymorphs is both very important and very complex; predictions can produce hundreds, if not thousands, of possible crystal structures. Varying polymorphs can be unstable in storage, ineffective or at worst unsafe, which can — and has — led to drugs being pulled from market, costing millions. For example, rotigotine, the active ingredient in transdermal patches for Parkinson's disease, was withdrawn for reformulation due to an unexpected polymorph in 2007.

The ability to reliably predict the occurrence of polymorphs *in silico* would enable pharmaceutical companies to identify and mitigate such risks earlier, as well as identify and progress suitable candidates faster with fewer issues. While CSP is already a key part of the drug development process for many companies, the science and tools are still evolving. Exploration of CSP data is a bottleneck, with data often confined to a handful of computational specialists.

As a registered charity, an active centre of research and a producer of leading informatics software, the work that the CCDC does in collaboration with academic institutions, pharmaceutical companies and external software providers is critical to advancing methods in structural science, developing new approaches and expanding the utility of existing tools.

The new *CSD-Theory* software provides a user-friendly, interactive web interface that simplifies the CSP data storage, management, search and display, enabling users of all levels to access and share critical information across teams. The additional API component allows CSP data to be stored ready for AI or ML analysis. Capable of handling the full spectrum of CSP-data types and providing a platform to easily share results, *CSD-Theory* is set to drive forward the data-first approach in pharmaceutical drug development.

As well as the new software, the CCDC has an extensive and growing suite of tools to aid molecule discovery and materials design —including the Cambridge Structural Database (CSD) — the world's repository for small-molecule organic and metal–organic crystal structures. The CCDC also runs collaborative initiatives to drive advances in CSP such as the CSP Consortium and CSP Blind Test.

Similar to the CASP protein structure predicting challenge, which saw tech giants such as Google push the boundaries of protein folding prediction in 2020, the CSP Blind Tests enable researchers to test novel methods for small-molecule CSP in a controlled, unbiased setting — facilitating the development of computational techniques to predict crystal structures. The chemical systems tested are unpublished crystal structures sourced by the CCDC, overseen by an external referee from crystallographers across academia and industry. Previous years have demonstrated that these Blind Tests translate to real-world results that have the potential to revolutionise the pharmaceutical industry.

The current, 7th CSP Blind Test began in October 2020 and participants have 18 months to make their predictions. This year has already seen a record number of participants — 42 people in 18 countries — making it the most popular test to date. This demonstrates the growing interest and need for fast and accurate CSP and the software tools such as *CSD-Theory* to effectively manage, use and share data, that in turn enables pharmaceutical companies to incorporate this critical information into small-molecule drug development.

For more information, please visit www.ccdc.cam.ac.uk

HISTORY OF CRYSTALLOGRAPHY

From Lucretius to Kitaigorodsky

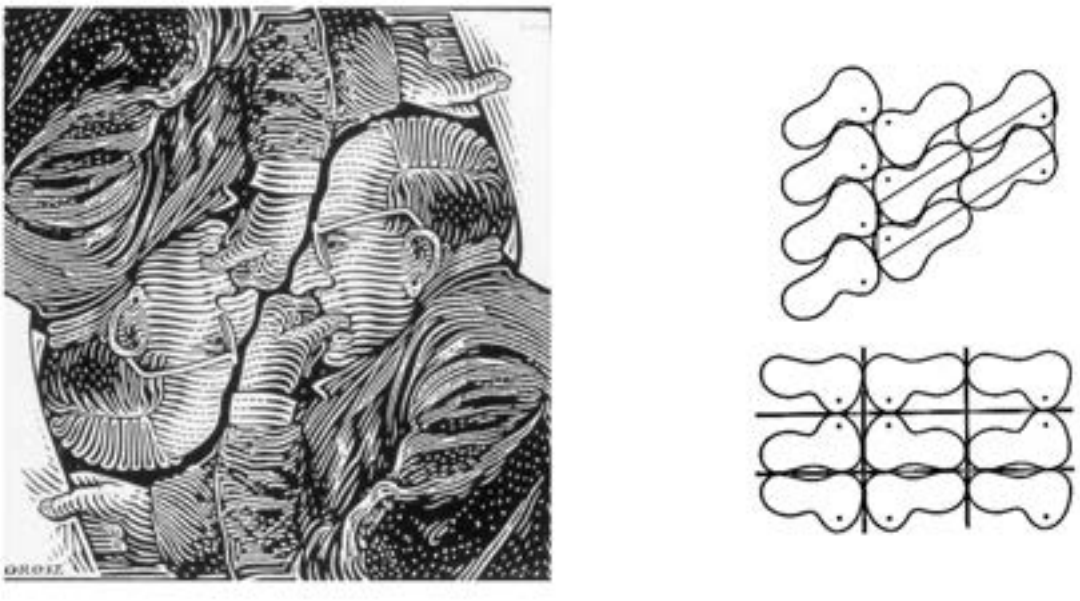


Istvan Hargittai, Balazs Hargittai



In 1940, Linus Pauling and Max Delbrück considered the most advantageous arrangements of molecules participating in biological processes [1]. They suggested that interaction between complementary parts would have precedence over interactions between identical parts. Also, intermolecular interactions such as van der Waals attraction and repulsion, electrostatic interactions, hydrogen-bond formation, *etc*., give stability to a system of two molecules with complementary rather than identical structures in juxtaposition. We see here a direct connection to molecular packing in molecular crystals. Aleksandr Isaakovich Kitaigorodsky (left) published a brief paper in 1945, “The Close-Packing of Molecules in Crystals of Organic Compounds,” which reads like an announcement of a program of a large research

project of understanding and predicting close packing of molecules in molecular crystals [2]. Our presentation of his ideas is qualitative, trying only to give a flavor of his approach, which was, though rudimentary, highly original. Soon, he came to the idea of testing arrangements of molecules represented by a general arbitrary shape for densest packing. He started by looking at the arrangements in a plane and concluded that lower symmetry packs better.

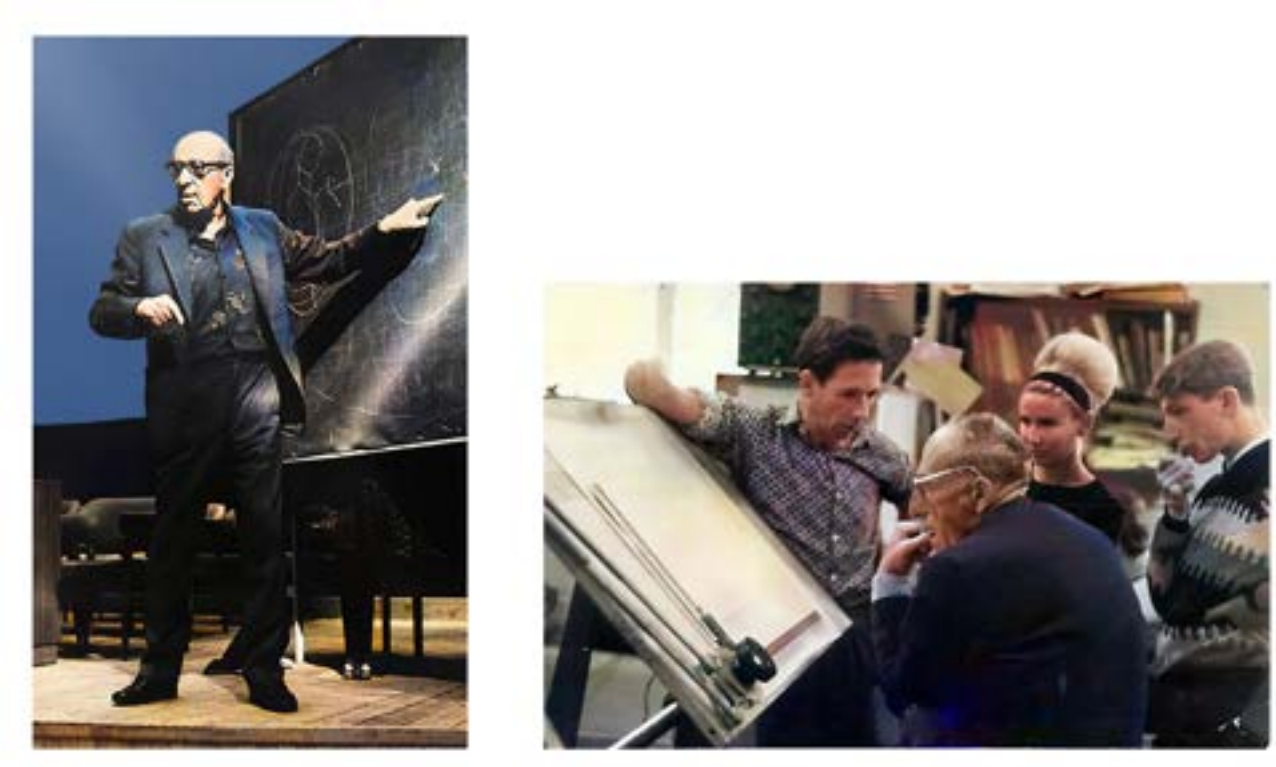


Left: “Complementary Kitaigorodsky” by Istvan Orosz (from [5]), and right: Two examples of packing arrangements of an arbitrary shape in the plane after Kitaigorodsky.

The title of the above artistic portrait by Istvan Orosz is “Complementary Kitaigorodsky” and the figure on the right shows two examples of packing arrangements of an arbitrary shape in the plane after Kitaigorodsky [3]. The upper arrangement has *p*2 plane group with twofold rotation, and the lower arrangement has *pmm* plane group with symmetry planes. In a complementary arrangement, the protrusion of one molecule complements the indentation of the other molecule. Hence such an arrangement provides denser packing than one possessing mirror planes. Lucretius over 2000 years ago proclaimed in his *De rerum natura* [4]:

“Things whose fabrics show opposites that match,
one concave where the other is convex,
and *vice versa* will form the closest union.”

Thus Kitaigorodsky’s observations were consistent with those by Lucretius. Kitaigorodsky then moved on to examine the three-dimensional case. For this, he constructed a device, which was as simple as it was ingenious. He called it “structure-seeker” and it consisted of a simple stand to which a wooden “model” of arbitrary shape could be fastened, and he tried to surround it with a number of identical “models,” examining the density of packing for each of the 230 three-dimensional space-group types. For the densest possible packing, he found six space-group types and even fewer for molecular shapes with symmetry centers. He succeeded in establishing an order of diminishing frequency of occurrence among the space groups. At the time of this empirical investigation, very few structures of organic crystals were known. Many years later, when there were hundreds of thousands of known crystal structures, his predictions proved to be justified, and only minor adjustments were necessary.



Left: Kitaigorodsky lecturing (courtesy of Irena Akhrem). Right: Kitaigorodsky among his disciples in the 1960s; from left to right: Erlen I. Fedin, Kitaigorodsky, Natalya V. Gorskaya and Ilya P. Amiton (courtesy of Jan Kandror).

Aleksandr I. Kitaigorodsky (1914–1985) was born in Moscow into a family of a well-known chemical engineer. He received an excellent education at home. He was a good pianist and a good dancer, he spoke French, had a good knowledge of German, and later he learned English. He graduated from the physics department of the Lomonosov Moscow State University in 1935, and he received his Candidate's degree (PhD-equivalent) in 1939. His dissertation was on the X-ray crystallography of amino acids. During World War II, he was in charge of a physics section of an armament plant. After the war, he worked at the Institute of Organic Chemistry of the Soviet Academy of Sciences, and earned his higher doctorate in 1946 for his work on the arrangement of molecules in crystals of organic compounds. From 1954, he worked in the newly established research Institute of Heterorganic Compounds of the Academy. He was the head of the Laboratory of X-ray Structure Analysis and Organic Crystal Chemistry. He was a most original thinker, and he often spoke his mind, which did not win him any favors with the authorities. This, and his having Jewish roots, prevented him from getting elected to the Science Academy and appointed to a university professorship at Lomonosov University, two promotions that he had fully deserved. He had many excellent disciples who have represented and spread his legacy in Russia and internationally.

For further reading about Aleksandr Kitaigorodsky, see also references [5], [6], and [7].

Literature

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[2] A. I. Kitaigorodskii (1945). 'The Close-Packing of Molecules in Crystals of Organic Compounds.' *J. Phys. (USSR)*, **9**, 351–352.

[3] A. I. Kitaigorodsky (1973). *Molecular Crystals and Molecules.* New York: Academic Press; Russian original, 1971.

[4] Lucretius, *The Nature of Things* [*De rerum natura*], 1st edition, translated by F. O. Copley. New York: W. W. Norton & Co., 1977. This quoted passage is from Book VI, lines 1084–1086, p. 72.

[5] I. Hargittai & M. Hargittai (2000). *In Our Own Image: Personal Symmetry in Discovery.* New York: Kluwer/Plenum, Chapter 6: “Aleksandr Kitaigorodskii,” pp. 112–142.

[6] I. Hargittai (2013). *Buried Glory: Portraits of Soviet Scientists.* New York: Oxford University Press, Chapter 11: “Aleksandr Kitaigorodskii — Soviet Maverick,” pp 251–266.

[7] E. V. Leonova, compiler (2011). A. I. Kitaigorodsky: *Scientist, Teacher, Friend* (in Russian, Moscow: ‘Moskvovedenie’). Е. В. Леонова, Составитель (2011). *А.И. Китайгородский — Ученый, учитель, друг* (Москва, ’Москвоведение’).

Istvan Hargittai is at the Budapest University of Technology and Economics, Hungary, and Balazs Hargittai is at Saint Francis University, Loretto, PA, USA.

HISTORY OF CRYSTALLOGRAPHY

The Lonsdale Blue Plaque

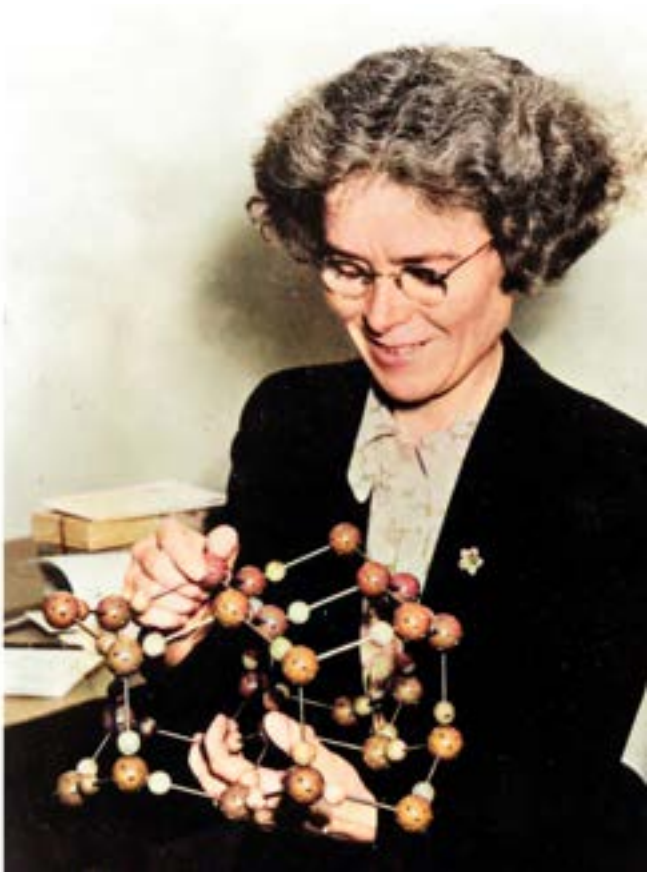


Kathleen Lonsdale in academic gown (courtesy of The Lonsdale Family) and her Blue Plaque (courtesy of English Heritage).

Claire Murray

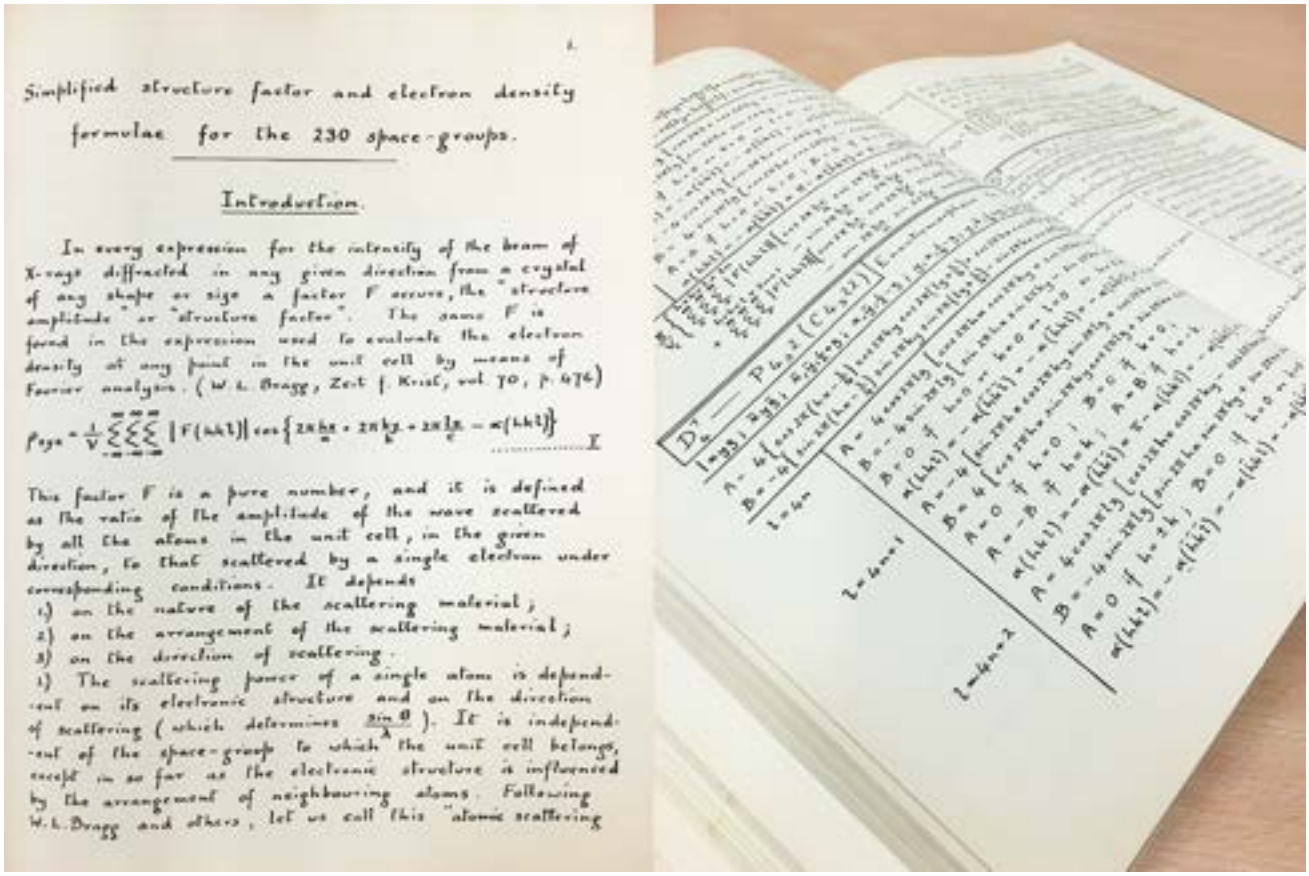
In a world where travel is currently limited, it is nice to dream about the future places we might visit. I'd suggest every crystallographer mark 19 Colenso Road, Seven Kings, London, UK, as a travel destination on their bucket list. This was the childhood home of Kathleen Lonsdale and is now commemorated by a blue plaque by English Heritage. These plaques are erected to identify people or events of historical interest linked to the location of the plaque, and they can be found all over London and the UK in various forms. They are a unique way to raise the interest and profile of the person or event through an extremely short summary in clear language. Kathleen Lonsdale's illustrious career in crystallography, and more, means she deserves this honour, and it is exciting to think of curiosity being sparked about her when people see the plaque. There is already so much about her online in the 643,000 hits in a Google search, including an obituary written about her by Dorothy Hodgkin and a review article by Mike Glazer, so people can explore her story, her achievements and her contributions to crystallography in various levels of detail.





Left: Kathleen Lonsdale's plaque marks her childhood home in Seven Kings, Redbridge, where she lived from 1911 to 1927 – a formative time encompassing her education, the First World War, and her early days at University College London and the Royal Institution. Photo courtesy of English Heritage. Right: Kathleen Lonsdale aged 50. Photo courtesy of The Lonsdale Family.

In considering her achievements, it seems tragically ironic that one of the latest things I learned about Kathleen Lonsdale was her work on *International Tables for Crystallography*, given these are a keystone in crystallography for new starters and trained professionals alike. They were clearly the work of many, but upon recently seeing her beautifully detailed work for a handwritten copy of the structure-factor tables, it struck me that it is easy to overlook the breadth of her contributions to the fundamentals of crystallography. It raises an interesting point about how we talk about scientists' achievements. General discussions where scientists are mentioned frequently result in a reduction of the scientist to short 'sound bites'. However, within the very field of a scientist, there is so much more opportunity to share, celebrate and reflect across the whole of a scientist's career, rather than defining them by one point or one moment in time. We lose part of our history that has played a critical role in shaping the developments in our field and provide a very selective story of how knowledge 'appeared.' Typewriters couldn't handle the mathematical transcriptions of the knowledge that Lonsdale and colleagues were building at the time, so she handwrote the first version of the tables whilst working at the Royal Institution for them to be reproduced effectively *via* high-resolution photocopies for about 20 crystallographic departments around the world. These tables were used to teach many hundreds of crystallographers and were regularly referred to by leading figures in and out of the field. The immense care and work Lonsdale put into painstakingly recording the 230 space groups by hand (!) is therefore something we should not forget.



Simplified structure factor and electron density formulae for the 230 space-groups.

Introduction.

In every expression for the intensity of the beam of X-rays diffracted in any given direction from a crystal of any shape or size a factor F occurs, the "structure amplitude" or "structure factor". The same F is found in the expression used to evaluate the electron density at any point in the unit cell by means of Fourier analysis. (W. L. Bragg, Zeit. f. Krist, vol. 70, p. 474)

$$\rho_{xyz} = \frac{1}{V} \sum_{-\infty}^{\infty} \sum_{-\infty}^{\infty} \sum_{-\infty}^{\infty} |F(hkl)| \cos\left\{2\pi\frac{hx}{a} + 2\pi\frac{ky}{b} + 2\pi\frac{lz}{c} - \alpha(hkl)\right\} \quad \ldots\ldots I$$

This factor F is a pure number, and it is defined as the ratio of the amplitude of the wave scattered by all the atoms in the unit cell, in the given direction, to that scattered by a single electron under corresponding conditions. It depends

1) on the nature of the scattering material;
2) on the arrangement of the scattering material;
3) on the direction of scattering.

1) The scattering power of a single atom is dependent on its electronic structure and on the direction of scattering (which determines $\frac{\sin\theta}{\lambda}$). It is independent of the space-group to which the unit cell belongs, except in so far as the electronic structure is influenced by the arrangement of neighbouring atoms. Following W. L. Bragg and others, let us call this "atomic scattering

Sample pages of Kathleen Lonsdale's handwritten structure-factor tables, reproduced by permission of the Royal Institution Archival Collection.

HISTORY OF CRYSTALLOGRAPHY

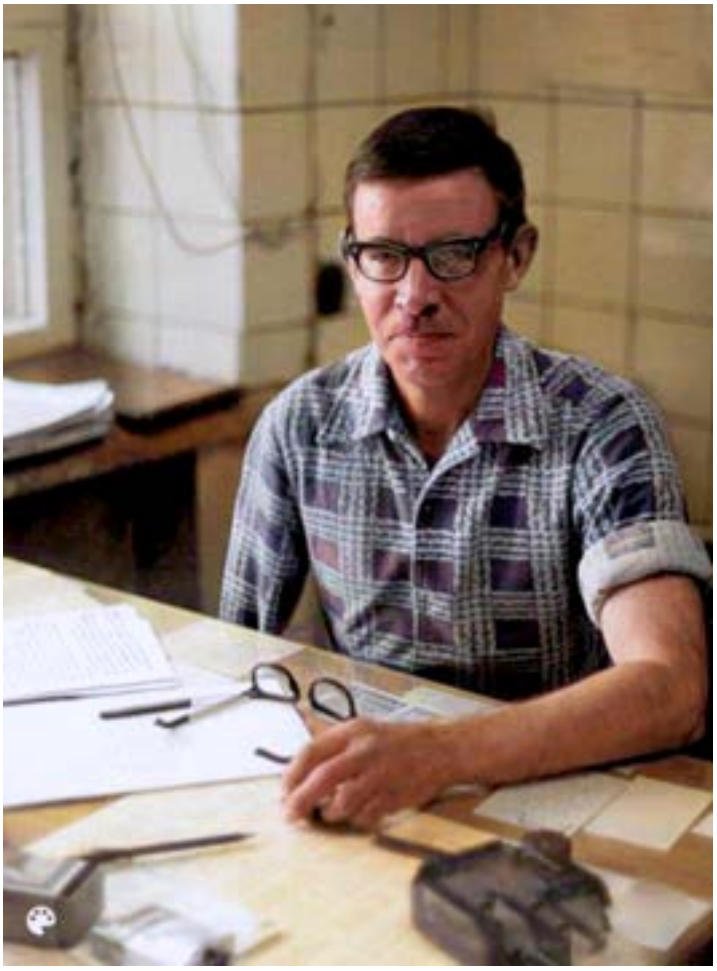
Yuri T. Struchkov: commemorating his 95th birthday



Yuri Slovokhotov, Alex Yanovsky

Yuri Timofeevich Struchkov was born on 28 July 1926 in Moscow, USSR. His parents, Timofey F. Struchkov and Olga P. Balakireva, graduated from Timiryazev Agricultural Academy and worked in the Ministry of Agriculture; Yuri was their only child. During his teenage years, Yuri Struchkov encountered perhaps his most dramatic hardships that had a lasting effect on his life. In 1937, he had his right arm amputated after an injury in a street accident; in 1938, in the times of Stalin's mass reprisals, his father was falsely accused and arrested; later, he perished in jail. From his very young years, Yuri was a very bright and curious child, especially interested in science, history and geography. Still, his path to education and eventual scientific achievements had never been easy and straightforward; it was full of hard struggles, as he grew up at tumultuous times and in a harsh environment.

In 1948, Yuri Struchkov graduated from the chemistry department of the Moscow State University. During his fourth year there, he joined the newly founded chair of crystallography, standing out in a group of nine chemistry and geology students with his excellent scores and hard work. He was a very determined sort of person and never considered an obstacle to be unsurmountable. Thus, in addition to a basic English course that he took at the University, he also learned German on his own in order to read Paul Niggli's book *Geometrische Kristallographie des Discontinuums*. In 1948, Struchkov became a graduate student in the Institute of Organic Chemistry (IOC), with Aleksander Kitaigorodsky as his supervisor. He earned his PhD in 1953 for the crystal structure determination of two metal complexes of unsaturated organic compounds with single-crystal X-ray diffraction data recorded on photographic films and visually estimated reflection intensities. After this, Yuri Struchkov became a key researcher in Kitaigorodsky's team in the Nesmeyanov's Institute of Organoelemental Compounds (better known by its Russian abbreviation INEOS). In 1973, Struchkov's X-ray diffraction group was spun out of Kitaigorodsky's Laboratory into a separate entity directly reporting to the administration of the institute.



Yuri Struchkov was one of the first Russian scientists to emphasise the great importance of X-ray diffraction in chemistry. In the famous book by Kitaigorodsky, *Organic crystal chemistry* (1955), Struchkov wrote a huge review chapter, discussing the 220 crystal structures of organic compounds determined by that time in the whole world; the chapter comprised almost a half of the overall volume of the book. During 1956–1957 he investigated the first series of ferrocene derivatives, a new class of organometallic compounds, which at that time was the main focus of the studies of the INEOS director Alexander Nesmeyanov and his collaborators. In 1961, Struchkov and co-workers determined the first ordered crystal structure of another iconic π-complex, bis(arene)chromium, in the form of a substituted cation of $(CH_3C_6H_5)_2Cr^+I^-$ salt [where methyl fragments hinder a strong libration characteristic of an unsubstituted bis(benzene)chromium molecule]. In 1964, he published a study of *para*-Me,Et-carborane, one of the first structures with an icosahedral carborane skeleton. It was at about this time that single-crystal X-ray diffraction emerged as the unique physical tool allowing unpredictable non-classical structures to be discovered in the novel fields of chemistry. Struchkov was among those few who were the first to realize the significance of these developments.

The growing demand for the results of structural studies on the part of synthetic chemists required the largest possible volume and highest possible quality of X-ray diffraction data. In 1969 and 1970, Yuri Struchkov visited the UK (nine universities in two months, with diffractometer practice in Oxford). He established working contacts with many British crystallographers and chemists, including Durward Cruickshank, Jeoffrey Wilkinson, Alan Mackay, Dorothy Hodgkin and Olga Kennard. In 1976, he was a guest at the Syntex Analytical Instruments headquarters in the USA. Two automatic diffractometers, which started non-stop operation in INEOS in 1974 (Hilger-Watts) and 1976 (Syntex $P2_1$ with a low-temperature attachment), along with an Eclipse S/200 computer (1977), transformed Struchkov's group into one of the leading X-ray laboratories in the world. With a staff of about 10 crystallographers, this laboratory was determining up to 200 structures per year. In 1983, Struchkov's Laboratory became the National Affiliated Centre of the Cambridge Crystallographic Data Centre and the distributor of the

Cambridge Structural Database system in the USSR. In 1989, the Laboratory was transformed into the Centre for X-ray Structural Studies of the USSR Academy of Sciences. This transformation, however, did not affect the staffing: the Centre never had more than 15–20 permanent employees. The tremendous range and significant impact of its structural studies were primarily based on the rare efficiency of its scientists, and the high quality and credibility of their work.

Struchkov's lab did not feature a multi-level management hierarchy, which was typical for Russian scientific laboratories of the time. By its structure, it was somewhat similar to a Western-type research team with no intermediate steps between rank-and-file group members and the team leader. Professor Struchkov participated actively and fruitfully in all the studies conducted by his staff and students. Each researcher in the lab was responsible for some field (or even for several fields) of modern chemistry in the USSR and abroad. The Laboratory, later the Centre, collaborated with 70 research institutions in all branches of small-molecule chemistry. Among *ca* 2000 papers published by Yuri Struchkov and co-workers during his 45 years in crystallography, there were about 200 review articles on very different topics of chemistry and physics. More than 60 PhD and 20 Dr Sci. theses were defended under Struchkov's supervision. It is noteworthy that Yuri Struchkov only received his own Dr Sci. degree (for crystal structures of *ca* 100 transition metal π-complexes determined over the course of 20 years) in 1977, at the age of 51; he always subordinated his formal recognition to actual scientific research. Among various other activities, back in the early 1960s, Struchkov participated in the foundation of the *Journal of Structural Chemistry* (*Zhurnal Strukturnoj Khimii*), and later for long years served as one of its most diligent scientific editors.

The virtually unlimited scope of Struchkov's research interests included all the growing fields of small-molecule chemistry. In the 1970s and 1980s, all single-crystal X-ray studies in INEOS and most structural data for new organometallic and organoelemental compounds in other Russian institutions were produced in Struchkov's Laboratory. Struchkov and his co-workers determined the structures of several hundreds of π-complexes (practically for all transition metals and many f-elements), performed comprehensive studies of carboranes and metallocarboranes, F- and P-organic compounds, polynuclear metal complexes, as well as metal clusters. Needless to say, the new structures, obtained and discussed in tight collaboration with synthetic chemists, considerably accelerated the development of all related fields of chemistry. Struchkov's Laboratory and his Centre were a flagship unit in Russian X-ray structural studies, promoting world-level science at the national scale. In 1990, Yuri Struchkov became a corresponding member of the Russian Academy of Sciences; in 1993 he was elected Vice-President of the IUCr.

Along with many other fellow chemists and crystallographers, we recall years spent working in Struchkov's Laboratory as a time full of great challenges and great inspiration. Whereas determining the structure of new, often unprecedented compounds was our main responsibility, Yuri encouraged us and all his colleagues to participate in the development of many fields of experimental X-ray crystallography. In the early 1970s, the Struchkov group began low-temperature structural studies of liquid and, later, gaseous compounds in normal conditions. The first data collection for the low-temperature structure of 1,1,4,4-tetramethyl-1,4-digermanacyclohexadiene, still made by photographic methods, required the hourly addition of liquid nitrogen to the system and continuous monitoring. So many laboratory staffers remembered this experiment especially well, as the X-ray camera plus a homemade cryostat was moved to Yuri Struchkov's room, which was by no means anything like the secluded office of a high boss, but, on the contrary, one of the most often visited places on the premises. More than 100 crystal structures of liquid or gaseous compounds under ambient conditions had been determined by 1995. Systematic studies of predecessors of liquid crystals and physiologically active compounds were also conducted in Struchkov's Laboratory. He was one of the first crystallographers in Russia who realized the significance of absolute configuration determination by X-ray diffraction based on anomalous X-ray scattering. Under his guidance, a program for refinement of an absolute configuration parameter (similar to the well-known Flack parameter) was developed, and in 1974, his team started determinations of absolute configurations quite regularly, including configurations of those molecules that do not contain any atoms heavier than

oxygen. From the same time, Yuri Struchkov and Michail Antipin (who inherited and maintained the fruitful work of the X-ray Structural Centre after Struchkov's untimely death in 1995) accomplished a considerable number of high-precision diffraction studies of electron-density distributions in small molecules including several metal clusters. In the 1990s, the Centre for X-ray Structural Studies started investigations into fullerenes and perturbation crystallography.

Incomparable energy, deep knowledge of the subject and overwhelming love for science made Yuri T. Struchkov one of the most prominent persons in Russia and in the broader world of crystallography. His results contributed to important advances in various branches of chemistry. The alumni of Struchkov’s X-ray Centre went on to work in X-ray crystallography and often to lead the research groups at the forefront of structural science in numerous institutes and universities in Russia and all over the world.



Yuri Timofeevich Struchkov died from complications developed after lung and heart surgery on 16 August 1995 in the hospital of the Medical University of South Carolina at Charleston, USA; he was 69 years old. He is buried in Donskoy cemetery in Moscow.

AWARDS AND PRIZES

Yvonne Mascarenhas receives Joaquim da Costa Ribeiro Award



José Miguel Delgado, Graciela Díaz de Delgado

Yvonne Mascarenhas, who in the early 1960s pioneered the introduction of structural studies of small molecules and established the first Crystallography Laboratory in Brazil, has recently been awarded the Joaquim da Costa Ribeiro Award 2021, granted by the Brazilian Physics Society (SBF) “for her pioneering research activities in X-ray crystallography and for starting a solid scientific community in this area in Brazil”. Professor Mascarenhas is the first woman to win this award, which recognizes the life-time contribution of a researcher in the area of Condensed Matter and Materials Physics in Brazil.

Professor Mascarenhas obtained her undergraduate degree in Chemistry in 1954 from the University of Brazil, currently the Federal University of Rio de Janeiro (UFRJ) and in Physics in 1955 from the University of the State of Guanabara, currently the University of the State of Rio de Janeiro (UERJ). During her Chemistry studies, she got her first contact with Crystallography in a course taught by Elisiario Tavora Filho, a respected geochemist who had just returned from the USA where he had completed his doctorate at the Massachusetts Institute of Technology (MIT) under the guidance of Martin J. Buerger. It must be mentioned that the mineral tavorite, $LiFePO_4(OH)$, was named in honor of Professor Tavora.

Professor Mascarenhas’s professional academic career began in 1956 at the School of Engineering of the University of São Paulo (USP) in São Carlos, where she was the first woman in the faculty. This was the first school of USP in São Carlos. An important step in Professor Mascarenhas’s career was the two years (1959–1960) that she spent working at the Crystallography Laboratory of the University of Pittsburgh, USA, after receiving a Fullbright scholarship. There, she worked under the guidance of George A. Jeffrey and Brian Craven. This was the beginning of her illustrious career of more than 60 years and marked the formal introduction and later consolidation of Crystallography in Brazil. This process was facilitated by her remarkable ability to attract national and, particularly, international collaborators and visitors to Brazil to teach and develop research collaborations. With the creation of the USP São Carlos Institute of Physics and Chemistry (IFQSC), she moved to this new institute until it was divided into the Institutes of Physics (IFSC) and Chemistry (IQSC). She has held important administrative positions such as head of her department, director of the Institute of Physics (the first director) and vice-coordinator of the Institute of Advanced Studies–Polo São Carlos, among other positions. Professor Mascarenhas remained in the Institute of Physics until her compulsory retirement in 2001, but without disconnecting from research and teaching activities. Over the last few years, Professor Mascarenhas has led several projects related to science teaching and outreach programs in public elementary and middle level schools to promote the participation of girls and young women in science.





Left: Yvonne Mascarenhas (bottom row, second from left) serving as one of the Brazilian delegates to the General Assembly at the 16th IUCr Congress in Beijing, China, in 1993. Photo contributed to the IUCr archive by Bill Duax. Right: Yvonne Mascarenhas with James Pflugrath (left) and Michel Fodje at the Siena Crystallographic Computing School in 2005. Photo contributed to the IUCr archive by Harry Powell. Professor Mascarenhas served on the IUCr Commission on Crystallographic Teaching from 1993 to 2002.

She was research or visiting professor in several prestigious international institutions, such as Princeton University (1966), Instituto Politécnico Nacional de México (1967), Harvard Medical School (1972–1973), Birkbeck College, University of London (1979–1980), among others. Professor Mascarenhas was one of the founders of the Brazilian Society of Crystallography (1971), which later became the Brazilian Association of Crystallography (ABCr). She has been president of ABCr on several occasions.

During her productive academic career, Professor Mascarenhas supervised more than 40 masters and doctoral students, published more than 200 indexed articles, and presented numerous regular and invited contributions in national and international events. She has received numerous prestigious awards and recognitions. Among them, the National Order of Scientific Merit in the Grand Cross Class, the IUPAC Distinguished Women in Chemistry or Chemical Engineering Award and the Simão Mathias Medal of the Brazilian Chemical Society. She is a member of the Brazilian Academy of Sciences and an emeritus researcher of the Brazilian National Council for Scientific and Technological Development (CNPq).

As members of the Latin American Crystallographic community, we are very pleased to add our voice to congratulate Professor Yvonne Mascarenhas for her recent award at the time when she is celebrating her 90th birthday. Congratulations Professor Mascarenhas!!

For a list of papers by Professor Mascarenhas appearing in IUCr journals click here.

AWARDS AND PRIZES

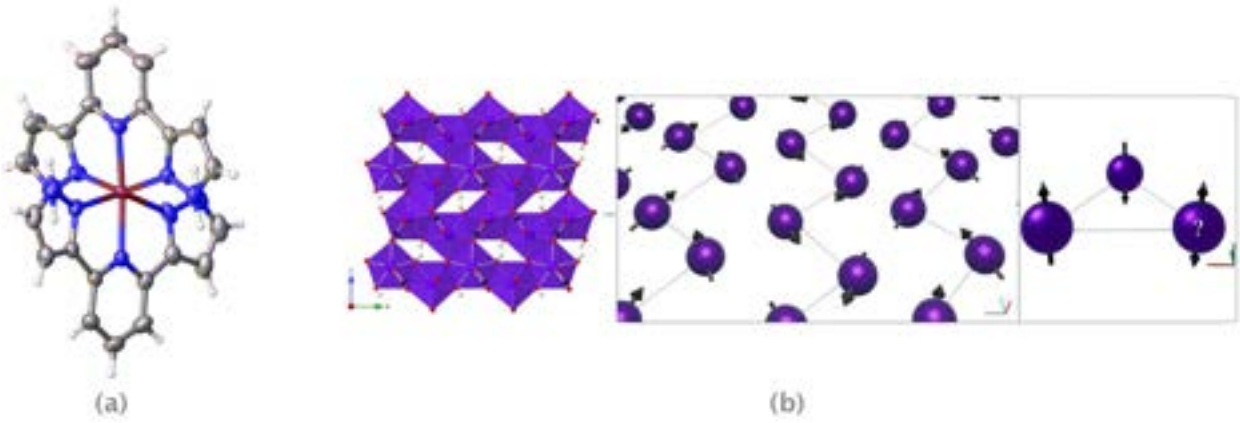
IUCr Poster Prizes at BCA BACG 2021



Lee Birchall (left) and Tom Hitchings, winners of the IUCr Poster Prizes at BCA BACG 2021.

John R. Helliwell, Petra Bombicz

The IUCr Poster Prizes at BCA BACG 2021 recognised any branch of crystallography or crystal growth, or indeed across more than one branch. They were awarded to two posters by PhD students and which were of equal high merit: Lee Birchall's was entitled "Design, synthesis and investigation of a series of isostructural spin-crossover co-crystal solvates using crystal engineering concepts" and Thomas Hitchings' was entitled "$LnOHCO_3$ Magnetocaloric Frameworks: A Potential Sustainable Replacement for Liquid Helium Vapour Compression". Both presenters were, by coincidence, from the School of Physical Sciences of the University of Kent, UK, but with different supervisors, respectively Helena Shepherd and Paul Saines.



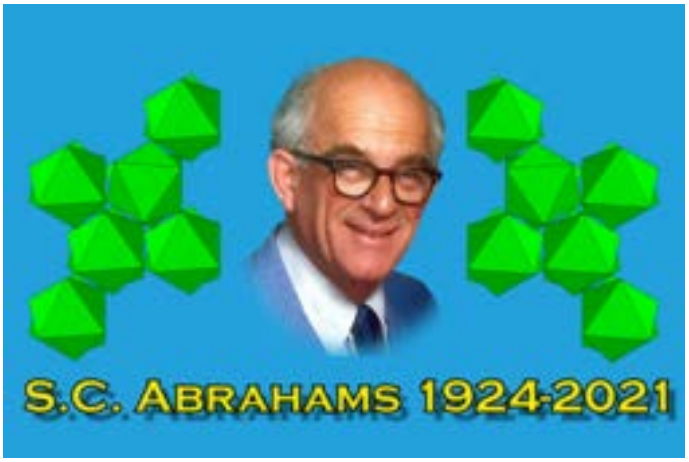
Figures from the posters showing (a) structure of the $[Fe(3\text{-bpp})_2]^{2+}$ complex (Birchall) and (b) short-range magnetic order (Hitchings).

This year's annual conference was a joint meeting of the British Crystallographic Association and the British Association for Crystal Growth; from memory, over the past 40 years, there have been three such joint annual meetings.

The judges for this Poster Prize were Petra Bombicz and John Helliwell. The winners each received a certificate, a copy of *A Little Dictionary of Crystallography* and a three-year student membership of the IUCr Associates Programme.

OBITUARY

Sidney C. Abrahams (1924–2021)



Carolyn P. Brock, Mike Glazer

Sidney Cyril Abrahams, distinguished scientist, founding member of the American Crystallographic Association (the ACA), and a pillar of the IUCr, died 9 February 2021 at the age of 96 in Ashland, Oregon, where he had lived for 30 years, most of them in active retirement.

He was born in London, England, in 1924, the first of three children of Aaron and Freda Abrahams. In 1938 his father, a customs inspector, was transferred to the port city of Greenock, on the south bank of the Clyde west of Glasgow where the river expands into the Firth; the family moved to the nearby town of Gourock. Sidney completed his secondary-school education at the historic Greenock Academy in 1942, a year after the nearby, and very destructive, Clydebank Blitz.

He went on to study chemistry at the University of Glasgow. In the physical chemistry laboratory his Demonstrator (*i.e.* Teaching Assistant) was Jack Dunitz, who says Sidney's brilliance was immediately apparent. During Sidney's undergraduate years he completed the military training required at the time, and he became friendly with fellow chemistry student Rhoda Banks, to whom he was married for more than 70 years and with whom he had three children.

After finishing his BSc in 1946 Sidney started work on a PhD in the group of J. M. Robertson; other students in the group at the time were J. D. Dunitz, A. McL. Mathieson and J. G. White. Sidney determined the crystal structures of two classic molecules: *p*-nitroaniline and naphthalene, with the latter being the first of 45 NAPHTA*nm* structures archived in the Cambridge Structural Database. Data for these two structures were reported as having been measured at room temperature, but the actual temperature would have depended on the time of year the data were collected. Jack Dunitz once estimated that the average temperature in that laboratory during the first post-war winters was *ca* 10°C.

While with Robertson, Sidney was one of 62 graduate students from Europe invited to spend the summer of 1948 at MIT. The timing was fortuitous because it allowed him to attend the first meeting of the IUCr at nearby Harvard University, an opportunity that created a deep impression on him, as is apparent from his Memoir on the ACA History Portal. Sidney eventually participated in 15 of the first 18 Congresses, with his last, in 1999, taking place back in Glasgow.

After completing his dissertation in 1949 Sidney spent a postdoctoral year with W. N. Lipscomb at the University of Minnesota. There he set up a device to measure data at low temperatures and to grow crystals of substances that are liquids under normal conditions. Papers reporting the structures of H_2O_2 (at 253 K) and thiophene (at 218 K) were the result.

Sidney next spent four years at MIT and four more at the University of Glasgow, which awarded him a DSc in 1957. He then moved to the USA to take a position at Bell Labs in Murray Hill, New Jersey, where

he stayed for 30 years. After his formal retirement in 1988 he was a Humboldt Senior Scientist at the University of Tübingen and a Visiting Professor at the University of Bordeaux before moving in 1991 to Ashland, Oregon, a small town in the Rogue River Valley where Southern Oregon University is located. There Sidney had an active retirement as an Adjunct Professor of Physics, publishing more than 65 papers during the period 1994–2010.



Sidney Abrahams at MIT in 1953.

Over the course of his career Sidney worked principally in the study of the crystal structures and the physical properties of inorganic materials. He was particularly well known in the ferroelectricity community. He published more than 300 research papers, about half of them in IUCr journals and another 20% in the *Journal of Chemical Physics*. As of March 2021 his papers had been cited nearly 16 000 times. During the years 2016–2020 his papers were still cited an average of 300 times per year.

The most important crystalline materials that Sidney studied were lithium niobate and lithium tantalate; they are used in optical waveguides, mobile phones, piezoelectric sensors, optical modulators and various other linear and nonlinear optical devices. Single crystals of lithium niobate are especially important; the global market for them was approximately USD 135 million in 2020. Five of his papers on these materials reported structure determinations as a function of temperature using X-ray and neutron diffraction. One of his most cited publications (the 1966 paper in *J. Phys. Chem. Solids* with J. M. Reddy and J. L. Bernstein, *Ferroelectric Lithium Niobate. Single Crystal X-ray Diffraction Study at 24 Degrees C*), is a landmark paper in the understanding of that material. Also important is his 1986 paper *Defect Structure Dependence on Composition in Lithium Niobate* in *Acta Cryst. B* with P. Marsh.

Sidney was also famous for studies of tungsten bronzes, particularly strontium barium niobate. He was one of the pioneers in combining absolute structure determination using anomalous X-ray scattering and measurements of electrical polarization to link the structure and properties of crystals. Similarly, he connected absolute structure with optical rotation, as in his 1977 papers on the classic examples $NaClO_3$

and $NaBrO_3$. Both types of measurements were tricky, as the X-ray diffraction and the physical property measurements had to be carried out on the same crystal to preserve the axial relationships.

Another much-quoted work in the field of ferroelectricity was Sidney's 1968 paper in *Physical Review* together with S. K. Kurtz and P. B. Jamieson on the relationship between atomic displacements and the Curie temperature and spontaneous polarization. He wrote an important review on atomic displacements in pyroelectric and piezoelectric crystals, published in the *Materials Research Bulletin* in 1978.

Sidney was also known for developing instrumentation, especially devices to allow extensive temperature variation. In the period 1950–1963 he published ten papers in the *Review of Scientific Instruments* and he then served on its Editorial Board from 1963 to 1965. Some of this work was done at Brookhaven National Laboratory, where Bell Labs maintained a presence. In 1959 Ted Prince and Sidney reported the development of the first automated single-crystal neutron diffractometer.

Sidney's most well known paper (730 citations as of March 2021) may be his 1971 publication in *Acta Cryst. A* with E. T. Keve describing the use of normal probability plots to compare independent determinations of the same structure. Analyses of many pairs of supposedly equivalent determinations have shown that their differences often exceed those predicted from the uncertainties given by the least-squares refinement programs.

For his scientific achievements Sidney was awarded honorary degrees by the Universities of Uppsala (1981) and Bordeaux (1997). In 1982 Bell Labs named him a Distinguished Member of its technical staff. He was a Fellow of the American Association for the Advancement of Science, the American Physical Society, IUPAC and the ACA.

Sidney Abrahams was also an exemplary citizen of the crystallographic community. He was a charter member (1950) and later President (1968) of the ACA. He chaired the IUCr's Commissions on Crystallographic Apparatus (1972–1975), Journals (1978–1987), Crystallographic Nomenclature (1978–2002) and Crystallographic Data (1978–1987); he also served on the Finance Committee (1981–1987). He was a member of the US National Committee for Crystallography (1966–1980) and chaired it during 1970–1972.

As Editor-in-Chief of the IUCr journals (1978–1987) Sidney oversaw major changes including moving all parts of page production to the Chester office and, in 1983, splitting Section *B* of *Acta Cryst.* into two sections (*B* and *C*). Sidney was an Editor from a traditional mold, unfailingly courteous and respectful of the rights and prerogatives of the author, but assured in his insistence on the primacy of the science in the reported research. Although conservative in many ways, he was also forward-looking. His encouragement of a terse 'telegraphese' style of writing in the structure reports of *Acta C* married the requirement of economy of paper with density and accuracy of information, and made it easier for the developers of the CIF standard in the early 1990s to translate the journal's requirements into a machine-readable format. Although Sidney's term of office finished before the adoption of CIF, he was well aware of the importance of computational science, both as an aid to communication and as a tool of scientific analysis in its own right. He was also responsible for the production in 1977 of the first computer-assisted edition (the 5th overall) of the *World Directory of Crystallographers*.

Sixteen of Sidney's papers were Commission reports, mostly on nomenclature but also on statistical descriptors and on the IUCr's *ca* 1970 single-crystal intensity-measurement project. The report *Atomic Displacement Parameter Nomenclature* is notable for its length (12 pages; Sidney was always thorough) and its number of citations (245). After his retirement Sidney published 11 obituaries and appreciations. Over the course of his career he also put into print nine *errata* and one *addendum*, not because he often made mistakes (he was very precise and careful) but because he was a stickler for accuracy and willing to admit mistakes.

Sidney was a very distinctive presence at meetings – dapper and very much a gentleman of the old school. He was always pleasant, always polite and correct, and often amusing. Sidney was also known to have very firm ideas that he sometimes promoted resolutely, but he was often right and always gracious.

He is remembered with affection by many, and is recognized both for his many scientific achievements and his many contributions to the crystallographic community.



Sidney and Rhoda Abrahams. Photo courtesy of Mark Abrahams.

OBITUARY

Joseph Yariv (1927–2021)



John R. Helliwell, Yariv family

Dr Joseph Yariv collaborated extensively with protein crystallographers over many decades. Through the greatest part of his research career he was in the Life Sciences Department of The Weizmann Institute in the section for membrane research and biophysics, retiring in 1992, age 65.

Joseph was born on the 13 June 1927 in Poland and spent his early years in Krakow, where he was particularly fond of skiing. When the Second World War broke out, he managed to escape from a German labour camp and rejoin his mother. They spent the rest of the war hiding in the basements of Polish peasants in exchange for payment which sometimes was obtained through risky sorties that Joseph had to make to relatives who were also in hiding. At the end of the war, they rejoined Joseph's father and sister who had already made their way to Israel just before the war started.

In Israel Joseph became a patriot, changing his surname from Leiser to Yariv, although he eventually became very disappointed with Israeli politics. His studies at the Hebrew University in Jerusalem were interrupted by the 1948 War of Independence, when he joined the students' regiment of the Etzioni infantry battalion as a first-aider, getting injured a number of times. Back for his studies at the Hebrew University he met his companion for life, Masha, who had just immigrated from Poland and was studying microbiology. Masha and Joseph spent 70 eventful years together, including four years of postdoctoral studies by Joseph in New York, and sabbaticals in the UK and France.

Masha was a researcher successively in departments of biochemistry (Columbia University), biophysics (The Weizmann Institute), agriculture (Hebrew University), hormone research and virology (both at The Weizmann Institute). After retirement they alternated between France and Israel until they eventually retired to Israel in 2009. They are succeeded by a son, a daughter and four grandchildren. Joseph passed away on 23 March 2021, two months after Masha succumbed to Covid-19 complications.

Joseph's PhD was in biochemistry on the *Toxicity of the extracellular phase of Prymnesium parvum cultures*. This was undertaken under the supervision of Professor Shlomo Hestrin, the founder of the Biochemistry Department in the Hebrew University (Yariv & Hestrin, 1961). Between 1959 and 1963, Joseph undertook postdoctoral studies at the Sloan Kettering Institute and Columbia University in New York. He returned to Israel as a research scientist at The Weizmann Institute Department of Biophysics (1963–1992) under Dr Ephraim Katchalski (Katzir). His work in all these years dealt with the themes of protein isolation, crystallization and structure solution. He published in *Nature* on the isolation of an L-fucose binding protein from *Lotus tetragonolobus* seed (Yariv *et al.*, 1967). He was the first to label a methionine in the active site of β-galacosidase of *Escherichia coli* and made extensive studies on this enzyme (Yariv, 1977). He produced crystals of concanavalin-A complexes with methyl glucoside and with methyl mannoside and participated in solving the structures of the protein binding site with these

saccharides (of which more below). He collaborated with physicists at The Hebrew University in Jerusalem in studying the state of iron in *E. coli* by Mossbauer spectroscopy, which led to the isolation of bacterioferritin. This was the first ferritin-like molecule to be found in bacteria and named as such, and the solution of its structure was with Felix Frolow and Joseph Kalb (Gilboa). This research was published in *Nature Structural Biology* and highlighted on the front cover of the July 1994 issue (Frolow *et al.*, 1994). With co-workers from the University of Bordeaux and the National Synchrotron Light Source in Brookhaven they reported a bacterioferritin in a new crystal form (Dautant *et al.*, 1998). In 1982–1983 Joseph had a research sabbatical at Sheffield University in the Molecular Biology Department with Professor Pauline Harrison, the crystallographer whose team solved the ferritin crystal structure. It was during this visit that I (JRH) was introduced to Joe when I was beamline scientist on SRS 7.2 at the UK's Synchrotron Radiation Source at Daresbury Laboratory.

Joseph had a very active long-term collaboration with his long-time colleague at The Weizmann Institute Joseph Kalb (Gilboa) [see the obituary by Helliwell & Frolow (2010)]. This collaboration, as well as research on bacterioferritin, led to a series of studies on concanavalin-A. These comprehensive investigations were brought to a crescendo with the study entitled *X-ray and molecular dynamics studies of concanavalin-A glucoside and mannoside complexes. Relating structure to thermodynamics of binding* (Bradbrook *et al*., 1998). Their collaboration spanned biophysics, computational chemistry and protein crystallography. Both Josephs, Kalb (Gilboa) and Yariv, were also unstinting in their support of methods developments for protein crystallography with synchrotron radiation and neutrons, and concanavalin-A crystals regularly provided a test bed, whether at ultra-high resolution, for optimized anomalous dispersion or for determining the bound waters as D_2O at the saccharide binding site.

Joseph was as passionate about art as he was about science and always excited about the beauty of the scientific models and photographs, which gave him as much satisfaction as the research. The book that Joseph published 24 years after his official retirement beautifully documents his interests and numerous collaborations (Yariv, 2016).

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This obituary was originally published in *J. Appl. Cryst.* (2021). **54**, 1025–1026.

For a list of papers by Dr Yariv appearing in IUCr journals click here.

OBITUARY

Sow-Hsin Chen (1935–2021)



Jill Trewhella, on behalf of the IUCr Commission on Small Angle Scattering

It is with sadness that we report the passing of Sow-Hsin Chen on 26 June 2021 in West Newton, MA, USA, at the age of 86.

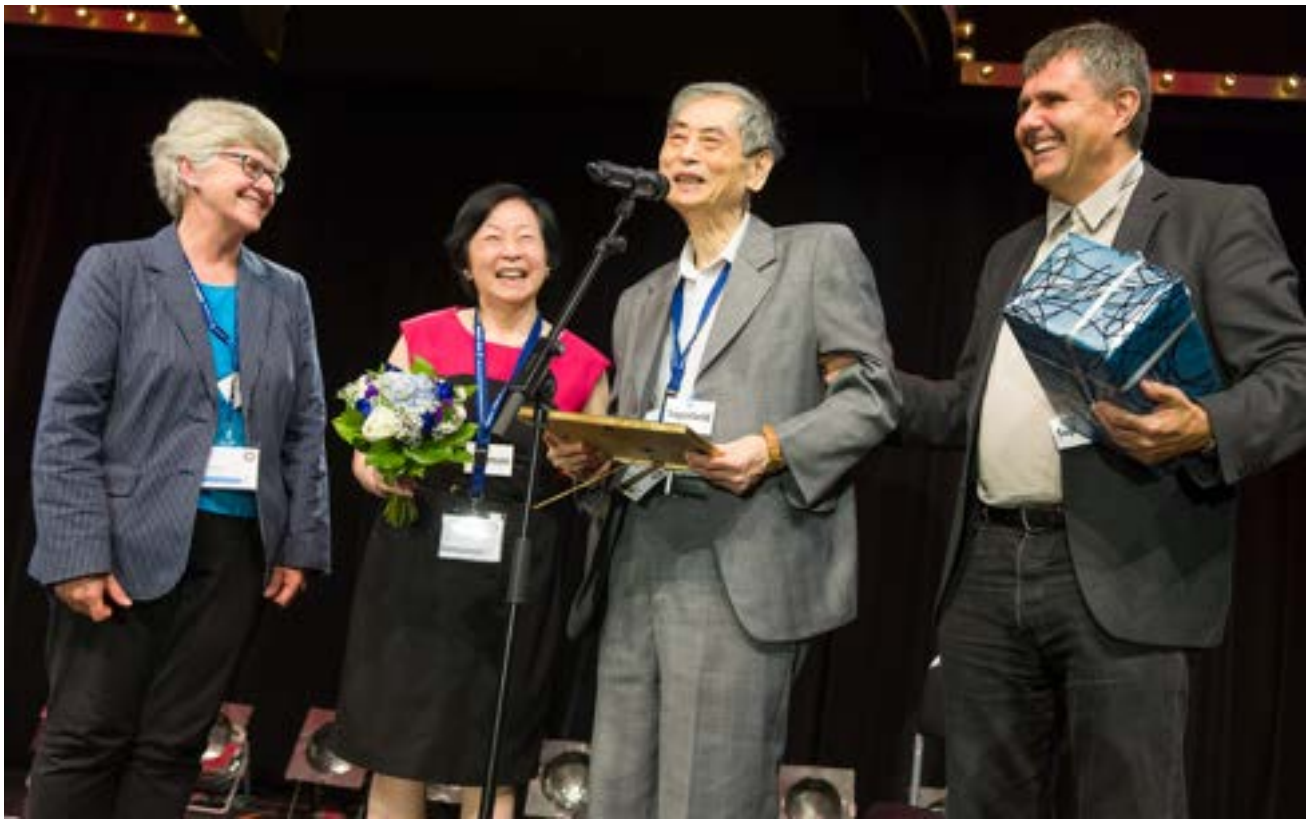
Sow-Hsin Chen was the IUCr/SAS2015 Guinier Prize winner in recognition of his "numerous original and novel contributions employing small-angle scattering in fundamental studies of soft condensed matter physics." In addition to the Guinier Prize, Sow-Hsin Chen was recognised with the 2008 Clifford G. Shull Prize from the Neutron Scattering Society of America "For seminal contributions to understanding the dynamical properties of supercooled and interfacial water using neutron scattering techniques, and for an exceptional record of training young scientists."

Educated first in his native Taiwan, Sow-Hsin Chen received a BSc in physics from the National Taiwan University in 1956 and an MSc in nuclear science from National Tsing Hua University in 1958. He came to the US on a highly competitive IAEA fellowship, first to Michigan, where he earned a second MSc degree in 1962 before pursuing his PhD at McMaster as the first PhD student of future Nobel Laureate Burt Brockhouse (graduating 1964). For his PhD project, Sow-Hsin Chen developed the method to classify the branches of the phonon dispersion relation according to the space-group symmetry of the crystallographic unit cell. With this distinguished start, he went on to Harwell, UK, to work with Peter Egelstaff to do pioneering research on single-particle and collective modes in liquids using a cold neutron time-of-flight chopper spectrometer constructed at the DIDO reactor. Another post-doctoral appointment with future Nobel Laureate Nicolaas Bloembergen at Harvard University saw the invention of photon-correlation spectroscopy to study slow critical fluctuations. Armed with this experience and a breadth of capabilities in both neutron scattering and light scattering, he joined the Department of Nuclear Engineering of the Massachusetts Institute of Technology (MIT) in 1968, where he remained. At the time of his passing, he was a very active Professor Emeritus of Applied Radiation Physics in the Department of Nuclear Science and Engineering at MIT.

Sow-Hsin Chen applied his remarkable creativity in both theory and experiment to tackle many soft condensed matter physics areas. He was a leader in the application of scattering techniques to solve problems in colloids and complex fluids, making major scientific contributions using a diversity of techniques. These included small-angle scattering (SAS), quasielastic neutron scattering, inelastic scattering spectroscopy, high-resolution X-ray inelastic scattering spectroscopy and photon correlation spectroscopy.

Among his numerous innovative applications of SAS was the use of small-angle neutron scattering (SANS) to discover a density minimum in deeply supercooled water that further demonstrated the plausibility of the existence of a second critical point in supercooled water (Liu *et al.*, 2007). He also

made long-lasting contributions to developments in the SAS technique, including the first use of neutron contrast variation to elucidate and propose the microstructure of SDS micelles and reversed micelles; formulation of the so-called decoupling approximation to handle the calculation of the SANS intensity distribution of interacting non-spherical particles in solution and to correct for the effect of polydispersity; a fractal approach to calculating the scattering intensity distribution of protein–detergent complexes in solution; devising a contrast variation method to measure the average mean curvature of surfactant films in a bi-continuous microemulsion; and proposing and implementing a Clipped Random Wave Model which enabled the successful measurement of the average Gaussian Curvature of the surfactant film.



Guinier Prize presentation in 2015. From left: Jill Trewhella, Ching-Chi Chen, Sow-Hsin Chen and Peter Fratzl.

Professor Chen's life-long teaching career produced over 45 PhD students, many of whom are leaders in their chosen fields. At MIT, his course *Statistical Thermodynamics of Complex Liquids* taught scattering methodologies and applications extensively. In a career spanning five decades, Sow-Hsin Chen published over 450 high-impact journal/review articles, a dozen monographic publications, and he wrote a comprehensive textbook on scattering methods in complex fluids. He was a regular contributor to the IUCr-sponsored triennial SAS meetings and a co-editor of the *Proceedings of the XIth International Conference on Small-Angle Scattering* (SAS99, Upton, New York, USA) published in *J. Appl. Cryst.* Part 3 No. 1, 2000 (69 papers for 319 pages). His well-cited paper, co-authored with his close collaborator Piero Baglioni and one of his outstanding former PhD students, Yun Liu, entitled "*The two-Yukawa model and its applications: the cases of charged proteins and copolymer micellar solutions*" was published as part of the *SAS2006 Proceedings* (Chen *et al.*, 2007).

Sow-Hsin passed away peacefully, surrounded by family. He is survived by his wife of 60 years, Ching-Chih Chen, their three children and their families. Sow-Hsin's passion for science was tireless, despite the increasing impact of an incurable genetic Parkinson's-like disease, and second only to his love for his family. In the words of his beloved wife, "Despite his dire physical conditions, he continued to work to the end of his 86-year life. He loves his family, but he has also given his last breath to science."

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Jill Trewhella is Professor Emerita at the University of Sydney, Australia.

OBITUARY

Jerry P. Jasinski (1940–2021)



Jerry Jasinski teaching in the chemistry lab. Photo credit: William Wrobel, Keene State College.

Ray Butcher

Jerry P. Jasinski passed away at his home in Springfield, Vermont, USA, in April 2021. Jerry was a Professor of Chemistry at Keene State College in Keene, New Hampshire. His research activities there spanned the fields of inorganic and physical chemistry and included structural and theoretical studies of laser dye complexes, and luminescence studies of lanthanide ion complexes of biologically interesting compounds and structural studies of pharmaceutically active compounds. His primary teaching interests were in General Chemistry, Inorganic Chemistry, Physical Chemistry, X-ray Crystallography and Biochemistry.

Jerry was born in Newport, New Hampshire, was an undergraduate at the University of New Hampshire (BA, 1964, following a year at Springfield College and a short stint in the U.S. Marine Corps) and did his graduate work at the University of New Hampshire (M.S.T., 1968) and Worcester Polytechnic Institute (M.N.S., 1968) receiving his PhD from the University of Wyoming in 1974 under the direction of Professor Smith L. Holt. He completed a summer NATO research fellowship at the University of Copenhagen, Denmark, under the direction of Professor Carl Ballhausen in 1973. After a predoctoral year (Associated Western Universities Fellowship) at the Los Alamos Scientific Laboratory, Los Alamos, New Mexico, under the direction of Dr Larned B. Asprey and John Wood, he completed a postdoctoral year at the University of Virginia, Charlottesville, Virginia, in the laboratory of Professor Paul N. Schatz. He spent nine years as a secondary school chemistry/physics teacher in the districts of Middleburgh, New York, Claremont, New Hampshire and Springfield, Vermont, both before and after his graduate work. While at the University of Wyoming he was a teaching fellow under the direction of Professor Geoffrey Coates.

He joined the faculty of Keene State College as an Assistant Professor in 1978 and was promoted to Associate Professor with tenure in 1983 and to full Professor in 1989. He received numerous grants to augment both his teaching and research activities while at Keene State College. In 1989 he established, within the Chemistry Department, the New England Molecular Structure Center upon receipt of a National Science Foundation Research In Undergraduate Institutions grant to fund a Single Crystal X-ray Diffractometer system and in 2009 obtained an National Science Foundation Major Research Instrumentation grant to replace the existing equipment with a dual source CCD diffractometer. He was involved in many collaborations with scientists throughout the USA, Europe and Asia performing single-crystal structural analysis in conjunction with their research activities and thereby actively involving many undergraduates at Keene State College in this unique type of scholarly activity. He published over 1000 papers in peer-reviewed scientific journals, many involving Keene State undergraduates as co-authors. In 1999 Dr Jasinski became the first Chairman of the Chemistry

Department at Keene State. In the spring of 2001 Dr Jasinski was presented Keene State College's first Faculty Distinction in Scholarship and Research Award for his outstanding research and scholarship success and his dedication to the College in teaching, research and coaching.

Dr Jasinski's professional activities include the following: American Chemical Society (Inorganic & Computational Divisions; Northeast Section of ACS), American Crystallographic Society, Sigma Xi, American Institute of Chemists (NEIC-Treasurer 1988–present; Board of Directors 1999–2001, President 2009–2011, Chairman 2016–2021), Council for Undergraduate Research, New England Association of Chemistry Teachers. He was a Co-editor of *Acta Crystallographic Section E* for over 10 years. Jerry held a Fulbright Teaching and Research Fellow at the University of Mysore in India from 2013 to 2014, conducting workshops and lecturing on the use of single-crystal X-ray crystallography as a state-of-the-art tool for determining molecular structure. Jerry was a caring, dedicated teacher, mentoring the careers of many undergraduates during his time at Keene State College. Jerry spent Summer 2001 in Bruce Foxman’s lab at Brandeis University, sponsored by NSF, and, over the next several summers together, they developed their Symmetry and Space Groups PowerPoint tutorial.[1] The tutorial contains >200 PowerPoint "slides", in five modules, arranged by crystal class; a sixth module covers special topics. Their hope was that the elements of guidance, inquiry and humor would make the learning process enjoyable, and indeed, the tutorial has enjoyed thousands of downloads worldwide. Bruce recalls: “Jerry spent each day working enthusiastically and tirelessly to make the tutorial a unique teaching tool. He provided an endless succession of fine ‘space group artwork’, solid teaching ideas, and great humor. Working with Jerry was a high point in my career.”

With achievements extending beyond Chemistry, Jerry was an outstanding high school athlete and set New Hampshire records that stood for many years. While at the University of New Hampshire he was participant in both their collegiate basketball and football programs. He was also an assistant coach of the Keene State men’s and women’s indoor and outdoor track and field teams for 18 years. Additionally, he officiated football, basketball and track and field at the secondary school level, and men’s and women’s collegiate basketball for 10 years. Jerry was very involved in the athletic life of his local community. For many years, in his hometown of Springfield, Vermont, Jerry initiated, ran and was commissioner of a PeeWee Football league. Jerry's biggest contribution to Vermont high school athletics was in track and field. He established the Vermont State Decathlon in 1976 and ran the event for 35 years. It is now called the Jerry P. Jasinski Vermont State Decathlon. Jerry led a full life making the most of every moment and was working on crystallographic matters until his very last day.

[1] The tutorial is available as a free download here.

For a list of papers by Professor Jasinski appearing in IUCr journals click here.

OBITUARY

Ekkehart Tillmanns (1941–2020)



Peter Paufler, Dirk C. Meyer

Shortly before reaching the age of 80, our member Ekkehart Tillmanns passed away unexpectedly at the end of 2020 in the circle of his family in Vienna.

In the same year, he had been awarded the Abraham Gottlob Werner Medal in gold by the German Mineralogical Society as the latest significant recognition of his internationally visible work in the field of mineralogical science. His work had previously received further special recognition. He was awarded the Distinguished Grantee Award 1998 of the International Centre for Diffraction Data (ICDD, USA) and the Erwin Schrödinger Prize of the Austrian Academy of Sciences in 2002; he was elected as Foreign Member of the Russian Academy of Sciences (Moscow) in 1999 and as Member of the German Academy of Sciences Leopoldina in 2000.

Born in Münster, he chose mineralogy as a field of study and work in which he actively participated at the Institute of Mineralogy and Crystallography at the University of Vienna until the end of his life. His path led him first to the University of Tübingen in 1961, where he obtained his intermediate diploma in mineralogy in 1964, and then to the University of Göttingen. There he met the Viennese mineralogist and geologist Josef Zemann, who headed the Institute of Mineralogy and Crystallography and supervised his diploma thesis until it was completed in 1966. The close link between mineralogy and crystallography characterised his scientific work from then on. At the time, however, he could not have foreseen that 25 years later he would succeed his diploma supervisor as Chair of Mineralogy and Crystallography at the University of Vienna and that he would be able to represent this geoscientific field of research together with him in the Leopoldina.

The following two years at the Mineralogical Institute of the Ruhr University in Bochum, which had only recently been founded in 1963, were of formative influence. He began his doctoral work in 1966 and received his Dr rer. nat. in 1968. In the working group of his doctoral supervisor Otto Wilhelm Flörke, after whom he later named a new mineral “Flörkeite” from the group of zeolites found by him and his co-workers, he was able to habilitate in 1973 with a thesis *Zur Kristallchemie der Verbindungen im System Bariumoxid-Titandioxid* (*On the crystal chemistry of compounds in the barium oxide–titanium dioxide system*) and thus set the course for a successful university teaching career. In the years 1968–1970, he had previously gained access to modern methods of X-ray crystal structure determination and crystal chemistry as a Visiting Assistant Professor at the Department of Geological Sciences at the University of Illinois, Chicago. The excellent application of both in his work soon earned him high recognition in the scientific community.

With the effective tool of X-ray crystal structure analysis, he was the first to experimentally determine extensive and precise data on the atomic arrangement of a wide range of inorganic and organic solids.

He not only used these data in his scientific publications to understand the properties of the substances but also made them available for quantitative use in all interdisciplinary research projects via relevant international databases.

His path then led him via a professorship for mineralogy and crystallography at the Institute for Geosciences at the University of Mainz (1974–1985) and via another professorship for general mineralogy/crystallography at the Mineralogical Institute of the University of Würzburg (1985–1991) to the Chair for Mineralogy and Crystallography at the University of Vienna (1991–2009). Looking at the results presented in his more than 185 original papers in scientific journals, the following focal points of his work can be identified, with which he has enriched the scientific field of geosciences to a particular extent: the crystal chemistry of minerals and inorganic compounds, the methodical development of the use of X-ray interference, and the growth of acentric crystals from aqueous solution, including the measurement of their physical properties.

Among the substances he studied, he made a special contribution to the group of zeolites, whose fields of application range from catalysts to wastewater purifiers, ion exchangers for water softening, desiccants in dishwashers and animal feed additives. Because the atomic frameworks are very flexible, there are many opportunities to use the knowledge of the structural researcher to specifically influence the properties of zeolites, *i.e.* to develop customized materials.

In addition to his successful work as a researcher and university lecturer, he has been committed to promoting the exchange of scientific ideas in his field, namely in positions of responsibility for the quality of the content of the *Zeitschrift für Kristallographie – Crystalline Materials* (Co-editor until 2000) and the *European Journal of Mineralogy* (Associate Editor until 2002, Chief Editor 2002–2010), as well as the journals *Mineralogy and Petrology* (Co-editor until 2003) and *Physics and Chemistry of Minerals* (Advisory Board).

In addition, Ekkehart Tillmanns has been an active member of national and international mineralogical societies and has played a formative role in their life. Above all, the German Mineralogical Society (DMG) should be mentioned here, where he was a member of the advisory board (1985–1987) and later a representative of the DMG on the board of the Working Group on Crystallography (1988–1991). In the framework of the Austrian Mineralogical Society (ÖMG), where he was a member of the board (1992–1997), he rendered outstanding services to the continuation of the close ties between DMG and ÖMG. In recognition of these services, he was elected by the ÖMG as its honorary member (2011).

Because of his scientific competence and willingness to constructively contribute to the further development of the field, he has repeatedly been entrusted with tasks of international standing. The chairmanship of the Austrian National Committee for Crystallography and – of particular importance – his work in the International Mineralogical Association (IMA) should be mentioned here. As its Vice-President (2006–2010), President (2010–2012) and Retired President (2012–2014), he was able to influence the work of this worldwide organization for eight years, including chairing the major IMA meeting in Budapest in 2010. In his honour, a mineral of the composition $(Ag_3Hg)(V,As)O_4$ from the French Alps was named “Tillmannsite” in 2003.

In particular the transfer of the IMA functions was, on the one hand, an expression of an extraordinary vote of confidence by the international community of mineralogists and, on the other hand, also a challenging opportunity to help shape the development of the IMA through his own initiatives. In doing so, he succeeded in effectively representing the interests of the Austrian and German professional community at the same time. Furthermore, he paved the way for a large number of students to enter the geosciences, who now gratefully and successfully continue his work. To his wife Uta Tillmanns-Niedereder and the rest of his family belong our special sympathy for the loss of Ekkehart Tillmanns, whom we now especially miss as a knowledgeable and kind colleague.

This article was originally published in *J. Appl. Cryst.* (2021). **54**, 1027–1028, and a similar version in *Crystal Research and Technology*.

For a list of papers by Professor Tillmanns appearing in IUCr journals click here.

OBITUARY

Nikolay I. Leonyuk (1941–2020)



Victor Maltsev



Nikolay I. Leonyuk (born 21 November 1941) sadly died on 24 May 2020. He held a DSc in Chemistry and Materials Science, and was a distinguished Professor and Scientific Advisor of the Crystallography and Crystal Growth Laboratory in the Geological Faculty of the Lomonosov Moscow State University (MSU).

His research primarily concerned the growth of crystals, crystallography, crystal chemistry, crystal physics of minerals and synthetic materials. Since the 1970s, his focus had been on anhydrous borates, and the study of these crystals revealed several important new physical effects. This made it possible to grow borate single crystals with sufficiently large sizes and high homogeneity and to consider some of the grown crystals as multifunctional materials for modern applications: laser and nonlinear optical

crystals with high efficiency, new laser media with a double function and the possibility of tuning, piezoelectric, acoustoelectric *etc. etc*. His interests included crystal growth and structure–property relationships in borate systems, as well as in other compounds: borosilicates, silicates, phosphates (berlinite), cuprates (high-temperature superconductors), tantalates, oxides such as corundum (ruby), alexandrite, calcite, quartz and diamond. He developed the principles of the relationship between the growth conditions, composition, structure and properties of these crystals. Also, a crystal-chemical approach was developed to optimize the conditions for crystal growth in complex systems and to search for new minerals and their artificial analogs for science and modern applications.

During Nikolay's career, he published over 700 papers plus 7 books and held 10 patents. He also engaged in teaching long-term courses on crystal growth for student crystallographers of the 3rd, 4th, 5th and 6th academic years at the MSU. The courses were entitled "Crystal Growth: Theory & Methods" (bachelor students) and "Crystal Growth: Low-dimensional crystalline materials" (Master students). He also organized a course of lectures and laboratory works entitled "Crystal Growth of Gem Stones" for student gemmologists of the MSU.

Professional activities

1972–1985: Senior Scientist (MSU)

1985–1988: Principal Scientist and Head of laboratory (MSU)

1988–2021: Professor and Head of laboratory (MSU)

Fellowships

1995: International Studies & Programs, University of Wisconsin-Madison, USA
1997: NATO-CNR, University of Parma, Italy
1998/1999: Cariplo Foundation, University of Milan, Italy
1999: Ministry of Education, Shandong University, China
2004: Visiting Research Scholar Award, Macquarie University Research Development Grant Scheme Round 2, Sydney, Australia
2004/2005 and 2009/2010: Cariplo Foundation, Politecnico di Milano, Italy.

Honours and Memberships

Member of the Russian Mineralogical Society since 1978
Soros Professor, 1997, 1998, 1999
"850 Years of Moscow Foundation" Medal, 1997
Laureate of Competition in the field of natural science "Grant of Moscow", 2001, 2002, 2004, 2005.

Nikolay was a person of many interests, always very open to strengthening scientific collaboration between Russian and international communities. He enthusiastically shared his expertise with students and — during international meetings — was often seen discussing unsettled questions with his colleagues. He will be sincerely missed.

OBITUARY

Graciela M. C. Punte (1944–2020)



Oscar E. Piro, Gustavo A. Echeverría

Suddenly and unexpectedly, physicist and crystallographer Dr Graciela Punte passed away in La Plata, Argentina, on 4 November 2020, aged 76, as a result of a hemorrhagic stroke. The sad news was announced by her husband, also a physicist and frequent collaborator, Dr Roberto C. Mercader.

Graciela was born in Buenos Aires, Argentina, on 2 July 1944. She graduated in solid-state physics from the Physics Department, Faculty of Exactas Sciences, National University of La Plata (UNLP), obtaining an MSc degree and then a PhD in Physics in 1972 with a thesis on “Electronic structure and magnetic properties of orthorhombic Cu(II) complexes.”

She pursued postdoctoral research in the UK on transition metal complexes, first at Oxford University (1974–1975) on hyperfine interaction and then on electron structure and bonding during a short stay at the Atomic Energy Research Establishment, Harwell.

Upon her return to Argentina and accepting a proposal from one of us (OEP), Graciela converted to crystallography by joining the X-ray laboratory at the Physics Department in 1977. This was a most fortunate incorporation as besides her scientific contributions, she later became the driving force behind the laboratory instrumentation and update.

The laboratory had been founded in 1968 by Dr Guillermo A. Alvarez, MSc Juan Andrieu and the then PhD student Eduardo E. Castellano, and was originally oriented to the physics of generation and properties of X-ray radiation. The laboratory eventually turned into one devoted to structural X-ray diffraction, following the return of Dr Castellano in 1971 after a two-year postdoctoral period in Oxford, UK. There he learned the basics of the methodology and introduced it for the first time to the UNLP by incorporating basic but complete facilities for single-crystal structure determination into the laboratory (X-ray generators, photographic recording of diffraction patterns with oscillation, Weissenberg and precession camaras, a suite of programs to solve and refine crystal structures, *etc*.). During these early times, the relative intensities of X-ray diffraction patterns were estimated from Weissenberg photographs by visual comparison against a standard set of data collected on five mammographic films per layer! The newly created research group on crystal structure determination followed the long tradition of the country in the methodology (Ewald, 1962) tracing back to 1929 when the chemist H. Damianovich traveled to Professor J.-J. Trillat’s laboratory in Paris to study by powder X-ray diffraction the structural effects on Pt electrodes exposed to He (Damianovich & Trillat, 1929).

Graciela was appointed Associate Professor at the Faculty of Engineering (FI), UNLP, in 1978, hence continuing a career in university education that started as early as 1966 as Teaching Assistant and culminated as Full Professor, first at the FI (1994) and then at the Physics Department (2009). She retired in 2014 but maintained a partial academic activity as a Consultant Professor.

In 1980 she became a fellow of the National Council of Scientific and Technical Research (CONICET). This allowed her, like many of us, to dedicate a substantial amount of academic activity to scientific research.

She co-authored about one hundred scientific papers on a wide range of subjects, mainly dealing with the relationship between crystal structure and physicochemical and spectroscopic properties of various materials. Since her arrival in the laboratory and up to the mid-1990s, she studied coordination solids of transition metals; noncovalent intra- and intermolecular interactions of organic molecules, both in solids and in solution; organic compounds of biological interest and also the then fashionable high-T_c ceramic superconductors.

From the mid-1990s, among other projects, she further studied packing forces in molecular solids. To enhance the experimental facilities that afforded the proper development of this and several other scientific projects that were in progress at the laboratory, she co-founded with Dr Blas E. Rivero in 1992 the National Laboratory of Diffraction for Research and Service (LANADI). She acquired a single-crystal Enraf–Nonius CAD-4 diffractometer equipped with a rotative copper anode and a point scintillation counter detector. The equipment was purchased with a CONICET grant in the late 1980s but several bureaucratic hurdles delayed its installation until late 1995.

This opened up experimental possibilities for determining the crystal and molecular structure of materials that were then unique in our country. As LANADI’s Coordinator, Graciela established several collaborations with Argentinian, Brazilian and European laboratories. She was the first in the country to pursue studies on the experimental determination of electron density of molecular crystals from X-ray diffraction data (Ellena *et al*., 2001).

In 2006, Graciela accepted a proposal from one us (OEP) to update LANADI's single-crystal X-ray diffraction capabilities through the presentation of a scientific project in the frame of the so-called Project for the Update of Research Laboratory Equipment (PME 2006) of the National Agency of Scientific and Technological Promotion (ANPCyT), Argentina. This project involved the purchase of a last-generation X-ray diffractometer. She succeeded again and, from 2009, we acquired an Oxford dual (Mo *K*α and Cu *K*α radiation) instrument with a CCD area detector and a low-temperature accessory.

This upgrade gave a great boost to structural studies, through multiple collaborations, on small- and medium-sized molecules in the country and the region. As a consequence, LANADI, now at La Plata Institute of Physics, IFLP (CONICET-UNLP), is currently one of the most important laboratories for structural X-ray crystallography of Argentina and Latin America.

During recent years, while still keeping an interest in the above studies, she opened up new lines of structural research on materials with potential applications such as pharmaceuticals and also in environment remediation, including bio-ceramics, nano-clays and biodegradable polymers.

She supervised four PhD students in physics and one in chemical engineering, and tutored several disciples, some of whom would later become accomplished structural crystallographers.

Graciela was deeply involved in promoting crystallographic methodology, both as a teacher of university graduate and postgraduate courses, schools and workshops, and as an organizer of national, regional and international courses (Punte, 2014), as well as congresses.




Left: Graciela Punte (centre) at the Delegates Reception at the 19th IUCr Congress in Geneva, Switzerland, in 2002. Photo contributed to the IUCr archive by Bill Duax. Right: Graciela lecturing at the MaThCryst International School on Fundamental Crystallography in La Plata, Argentina, in 2014. Photo contributed to the IUCr archive by Massimo Nespolo.

At the international level, Graciela served as a member of several bodies of the IUCr, including the Calendar Committee (1996–2002) and the Commission on Structural Chemistry (1996–2005). Furthermore, she was a member of the International Selection Committee for the ninth IUCr Ewald Prize (2011) and also as the Argentinean representative to the IUCr General Assemblies at Seattle (1996), Glasgow (1999) and Geneva (2002).

At the national and regional level, she was member of the ACA, re-founder of the Argentinean Crystallographic Association, AACr (2004), which she served as its vice-President (2004–2008), and also co-founder of the Latin American Crystallographic Association, LACA (2013).

Graciela was a devoted scientist, crystallographer and university educator and also an exceptional science and teaching organizer and administrator in experimental physics. These virtues make her a key figure in the modern development of X-ray crystallography in our country and an international reference in this area of condensed-matter physics. Her sudden decease is an irreparable loss for her loved ones, friends, colleagues, collaborators and students and also for the scientific crystallographic community at large.

She will be sorely missed.

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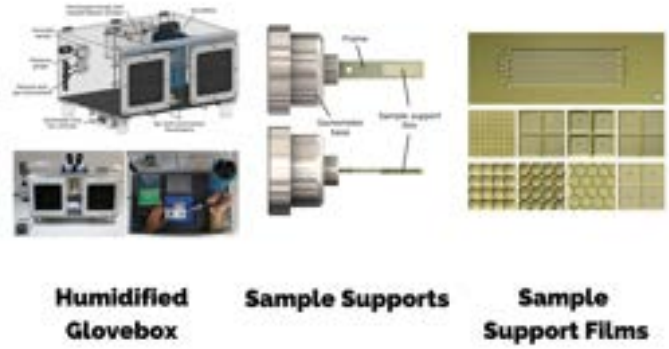
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For a list of papers by Dr Punte appearing in IUCr journals click here.

NEW PRODUCTS

Crystallography Sample Supports – the next-generation crystal mounting system from MiTeGen



MiTeGen announces the development of a next-generation crystal mounting system that will change how crystals are prepared for X-ray diffraction data collection.

This new system allows users to:

Mount single crystals or thousands for microcrystals

Optimize for serial crystallography or single-crystal XRD

Perform either cryogenic or room-temperature diffraction

How it works:

Crystals are mounted onto the supports utilizing a humidified sample loading box. The high-humidity box allows samples to be prepared without unintentional dehydration to the drops or crystals. The supports can then be plunge-cooled (for work at cryogenic temperatures) or sealed (for room-temperature work).

The system has multiple applications:

Conventional crystallography

Serial crystallography

In-situ crystallography

Room-temperature crystallography

And more

Major salient features include:

The system is compatible with existing infrastructure for high-throughput crystallography at synchrotrons and addresses major issues in maximizing data quantity and quality from large numbers of small crystals.

The sample supports are compatible with existing infrastructure for home source and mail-in storage-ring crystallography, including sample handling tools, storage cassettes/pucks, automounters and goniometer stages.

This integrated system addresses common problems in obtaining properly dispersed, properly hydrated and isomorphous microcrystals for fixed orientation and oscillation data collection. Its ease of use, flexibility and optimized performance make it attractive not just for serial synchrotron crystallography but also for single- and few-crystal data collection.

Why did MiTeGen design this system?

“We are excited about the developments we have been making to advance how crystals are delivered to the X-ray beam”, says Ben Apker, COO of MiTeGen. “We believe that the new sample support system will change how crystallographers prepare crystals for data collection, whether they are doing serial work on microcrystals or using larger single-crystal approaches.”

Collaboration and feedback

The system is still in continual development and MiTeGen is seeking input from interested parties for further refinement of the system.

Learn more about Crystallography Sample Supports here

Read the paper in *Acta Crystallographica Section D*

NEW PRODUCTS

ED-1: ELDICO’s novel electron diffractometer



ED-1: ELDICO’s novel electron diffractometer (the first of its kind) can produce accurate atomic structures from nano-sized samples in minutes.

With the revolutionary ED-1, ELDICO Scientific, a Swiss technology and solution provider, is pleased to introduce its brand-new electron diffractometer. The novel instrument combines an electron beam of radically simplified design with a goniometer precise down to the submicrometer. In a class of its own, the new tool combines the best of both worlds, enabling diffraction experiments at the nanoscale in an easy-to-install, user-friendly device. Equipped with this instrument, any crystallographic lab will be able to perform routine analysis on samples that have so far been considered prohibitive.

The ELDICO ED-1 combines the precision and workflow of X-ray diffraction instrumentation with the potential of electrons. With ELDICO’s disruptive instrument, the question of the availability of sufficiently large crystals – so far the most significant and most painful bottleneck in crystallography – may soon just be an anecdote of the past.

ELDICO Scientific has tailored the novel tool to strictly adhere to the most essential specifications. The device is a clever combination of a five-axis, 140° rotation, submicrometer-precise goniometer and a 160 keV electron beam with specifically designed optics. This tool makes it easy to produce accurate atomic structures from inorganic as well as organic samples within minutes. ELDICO’s ED-1 has Dectris inside: the powerful QUADRO is the most proven detector well suited for electrons. The cryo function, available as an option, provides cooling by conduction to support work near nitrogen levels.

With its superior features, the device outperforms any other method used for nano-sized samples. The diffractometer is designed to measure samples in the range from 10 to 1.000 nm and is targeted to provide resolution of up to 0.84 Å with at least 60–70% complete datasets having an R_{int} <20%. These data typically allow for structure solution and refinement down to R_1 values of <10% in 75% of cases, with unit-cell determination as accurate as 1:1000.

The ED-1 is dedicated solely to electron diffraction. With its disruptive horizontal design and innovative probe-handling mechanism, the diffractometer will help crystallographers enter the field of nanocrystallography and produce important structural information faster, of better quality and at a competitive price.

ELDICO’s product and service offering ranges from device procurement and CAPEX-friendly subscription solutions for industrial companies to special arrangements for academic users and measurements-as-a-service – and is thus perfectly tailored to the needs of any crystallographer. More

information: www.eldico-scientific.com

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