INTERNATIONAL UNION OF CRYSTALLOGRAPHY

IUCr Newsletter

Volume 19 · Issue 2

Collected archival edition
Original article text and available illustrations

Contents

> Extraction incomplete: missing article positions 1.

IUCr journals news

Translation calibration of inverse-kappa goniometers in macromolecular crystallography 3

High-pressure experimental study on Rb2S: antifluorite to Ni2In-type phase transitions 4

Six two- and three-component ammonium carboxylate salt structures with a ladder-type hydrogen-bonding motif, three incorporating neutral carboxylic acid molecules 5

Structure of HLA-A*0301 in complex with a peptide of proteolipid protein: insights into the role of HLA-A alleles in susceptibility to multiple sclerosis 6

4,4′-Bis[2-(3,5-dimethoxyphenyl)ethenyl]biphenyl 7

Crystal structure of small protein crambin at 0.48 Å resolution 8

Scattering functions of Platonic solids 9

Effective scavenging at cryotemperatures: further increasing the dose tolerance of protein crystals 10

Special report

Between the West and the East Introduction to Crystallography in South-Eastern Europe 11

Meeting report

The HEC community meets at Schöneck 30

Awards and prizes

Ninth Ewald Prize 31

IUCR JOURNALS NEWS

Translation calibration of inverse-kappa goniometers in macromolecular crystallography

Acta Cryst. (2011). A67, 219-228 (doi.org/10.1107/S0108767311004831)



EMBL/ESRF miniKappa goniometer head (MK3) implementing the inverse-kappa design as mounted on the MD2M diffractometer of the MX beamline ID14-4 at the ESRF.

Precise and convenient crystal reorientation is of experimental importance in macromolecular crystallography. While traditional multi-axis goniometers require all rotation axes to intersect a unique point at the sample position, recently developed miniaturized instruments are not as restricted. However, the samples must always be re-centered following a change in orientation. To overcome this inconvenience and allow the routine use of inverse-kappa goniometers, an automatic translation correction method and the corresponding calibration protocol have been developed at EMBL.

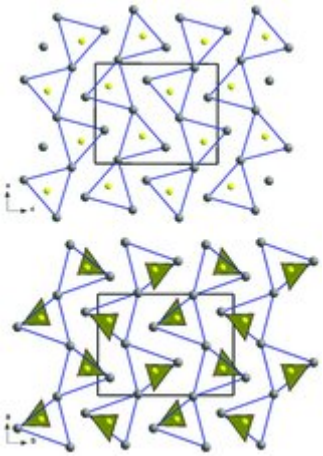
S. Brockhauser, K. I. White, A. A. McCarthy and R. B. G. Ravelli

IUCR JOURNALS NEWS

High-pressure experimental study on Rb2S: antifluorite to Ni2In-type phase transitions

High-pressure experimental study on Rb_2S: antifluorite to Ni_2In-type phase transitions

Acta Cryst. **(2011). B67, 109-115 (doi.org/10.1107/S010876811005370X)**



The crystal structures of anticotunnite Rb_2S (*Pnma*) at 0.7 GPa (top) and Rb_2SO_4 at room conditions (*Pnam*) (bottom), showing the similarities of both Rb_2S arrays. Colour key: Rb-grey, S-yellow, O-tiny-red. [SO_4] tetrahedra are olive coloured.

The crystal structures of two new high-pressure phases of Rb_2S are reported. Antifluorite Rb_2S firstly transforms into an anticotunnite structure below 0.7 GPa and, subsequently, into an Ni_2In-type structure at 2.6 GPa. Interestingly, the structures of these phases are identical to the cationic arrays of the two existing polymorphs of the corresponding sulfate, Rb_2SO_4. Once more, cations do not seem to be 'isolated entities' but they recognize themselves in spite of being surrounded by oxygen atoms.

D. Santamaria-Perez, A. Vegas, C. Muehle and M. Jansen

IUCR JOURNALS NEWS

Six two- and three-component ammonium carboxylate salt structures with a ladder-type hydrogen-bonding motif, three incorporating neutral carboxylic acid molecules

Acta Cryst. **(2011). C67, o92-o99 (doi.org/10.1107/S0108270111003714)**



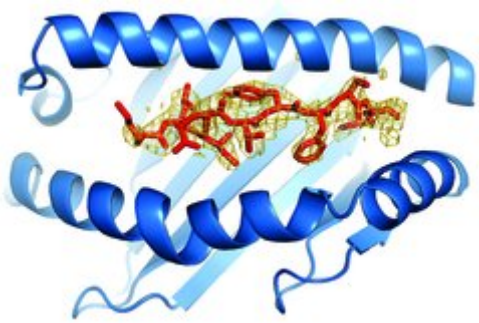
When combining two or more different molecules into one crystalline unit, complimentary hydrogen-bonding interactions are essential. One of the most reliable methods is to form an ammonium carboxylate salt by using two different molecules, one containing an amine and the second a carboxylic acid functional group. In this paper, we show that it is possible to hydrogen bond the neutral form of the carboxylic acid molecule to the charge-assisted hydrogen bonds of the salt by deliberately using half of the required stoichiometric amount of acid.

A. Lemmerer

IUCR JOURNALS NEWS

Structure of HLA-A*0301 in complex with a peptide of proteolipid protein: insights into the role of HLA-A alleles in susceptibility to multiple sclerosis

Acta Cryst. **(2011). D67, 447-454 (doi.org/10.1107/S0907444911007888)**



The crystal structure of HLA-A3 in complex with a ninemer peptide. The view is into the binding groove, with the peptide model in red and the corresponding electron density in yellow chicken wire.

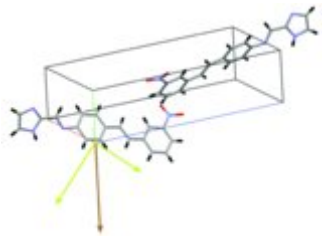
Major histocompatability (MHC) class I molecules present peptides from cellular proteins for inspection by the immune system. This sampling allows detection of infection; the peptides for a cell infected by a virus will include some from viral proteins. In autoimmune diseases the immune system attacks healthy cells. McMahon *et al.* report the crystal structure of a human MHC class I molecule, HLA-A*0301, in complex with a peptide from a nervous system protein. This complex is a candidate target for immune-mediated myelin destruction causing multiple sclerosis.

R. M. McMahon, L. Friis, C. Siebold, M. A. Friese, L. Fugger and E. Y. Jones

IUCR JOURNALS NEWS

4,4′-Bis[2-(3,5-dimethoxyphenyl)ethenyl]biphenyl

Acta Cryst. (2011). E67, o1112-o1113 (doi.org/10.1107/S1600536811012888)



Molecular (green) and unit-cell (brown) dipoles of an asymmetric organic semiconductor.

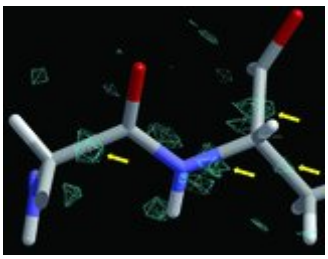
Considering the success of recently marketed consumer technology based on organic semiconductors, such as the OLED TV, it is clear that these materials have considerable potential for application in the opto-electronic devices of the future. Designing, synthesizing and characterizing new functional materials with properties tuned to match these applications remain central issues in the development of this technology. Detailed knowledge of their crystal structures is indispensible, in particular in the case of asymmetric systems which crystallize in polar space groups and display nonlinear optical activity.

A. Collas, M. Zeller and F. Blockhuys

IUCR JOURNALS NEWS

Crystal structure of small protein crambin at 0.48 Å resolution

Acta Cryst. (2011). F67, 424-428 (doi.org/10.1107/S1744309110052607)



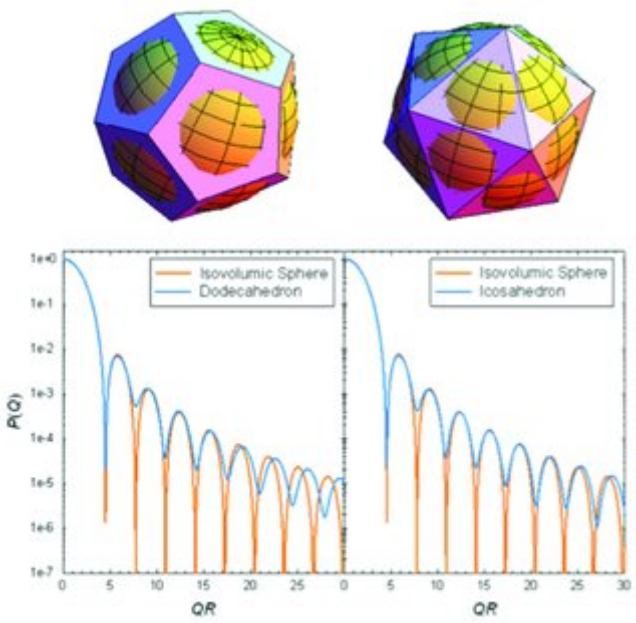
Electron density (cyan) around Arg10 calculated at 0.65 Å before refinement with *MoPro* commenced. The F_o–F_c map is contoured at 2.7σ (0.25 e Å^{-3}) above the mean. Bonding electron density is indicated.
Scientists at EMBL and DESY, Germany, and Boston College, USA, obtained the highest resolution X-ray crystallographic structure of a biological molecule, crambin, almost doubling the available data on this protein. The researchers hope that the availability of this data will drive software development beyond its current limits so that details of protein electronic structure could become attainable. This high-resolution structure was the first measured at the PETRA ring at DESY, where dedicated beamlines for biological samples are coming into operation.

A. Schmidt, M. Teeter, E. Weckert and V. S. Lamzin

IUCR JOURNALS NEWS

Scattering functions of Platonic solids

J. Appl. Cryst. (2011). 44, 545-557 (doi.org/10.1107/S0021889811011691)



The small-angle scattering form factors $S(Q)$ of dodecahedron (left), icosahedron (right) and the isovolumetric hard sphere.

Platonic solids, including tetrahedra, hexahedra, octahedra, dodecaheda and icosahedra, form an essential basis for the topology of condensed matter in a variety of contexts. For each given geometry, the Debye spatial autocorrelation function $\gamma(r)$, the pair distance distribution function $p(r)$ and the intraparticle structure factor (form factor) $S(Q)$ are calculated analytically and compared with the corresponding scattering functions of a spherical reference system with the same particle volume. As an illustrative example, the quantitative interpretation of small-angle scattering data from an icosahedral virus is presented.

X. Li, C.-Y. Shew, L. He, F. Meilleur, D. A. A. Myles, E. Liu, Y. Zhang, G. S. Smith, K. W. Herwig, R. Pynn and W.-R. Chen

IUCR JOURNALS NEWS

Effective scavenging at cryotemperatures: further increasing the dose tolerance of protein crystals

J. Synchrotron Rad. (2011). 18, 346-357 (doi.org/10.1107/S0909049511007163)



Electron density maps of the Cys6–Cys127 bond in lysozyme after doses of 2.3 MGy, 12.3 MGy and 22.3 MGy, showing a nearby bound ordered nitrate ion being reduced to NO_2 and then NO by X-ray induced electrons, and thus affording protection to the neighbouring disulfide bond.

Certain radical scavengers can reduce the rate of X-ray induced radiation damage to macromolecular crystals during both room temperature and cryotemperature (100 K) data collection. In HEWL crystals held at 100 K, sodium nitrate, an electron scavenger, was found to double the dose (Gy = J kg^{-1}) tolerance as judged from the rate of total diffraction intensity loss, and increase it by over 5 times when considering structural damage to the disulfide bonds. The sequential electron density maps show the radiation chemistry mechanism at work.

E. De la Mora, I. Carmichael and E. F. Garman

SPECIAL REPORT

Between the West and the East Introduction to Crystallography in South-Eastern Europe

Between the West and the East Introduction to Crystallography in South-Eastern Europe



(See also Part 2 and Part 3)

This issue of the *IUCr Newsletter* contains the first of three articles devoted to crystallography in fourteen countries from South-Eastern Europe - in many respects, a cradle of European civilization, and a portal where Europe meets the rapidly emerging economies of the Middle and Far East. The old socio-political theories from the last century and the political turmoil that followed their desertion have greatly affected scientific research in South-Eastern Europe. Driven by social, economic or professional factors, many chemists and physicists moved to leading research laboratories in Europe and the United States, including brilliant visionaries such as Nikola Tesla and Nobel laureates such as Fric Pregl (an Austrian chemist born in Ljubljana, awarded in 1923), Leopold (Lavoslav) Ružička (a Swiss chemist born in Vukovar and awarded in 1939) and Vladimir Prelog (of Croatian descent, born in Sarajevo, awarded in 1975). In the second half of the last century, researchers trained in the West returned to their native countries to teach and conduct research in mineralogy and crystallography. Through globalization, and improving economic stability in this part of the world there is real potential for a reversal of the brain-drain process to a brain-gain. We are witnessing an increase in contributions to crystallography research from the South-East European nations as they embrace globalization in a multipolar but integrated world. Divisiveness and fortress mentality are giving way to a spirit of openness and broader vision.

In South-Eastern Europe, our diversity is our greatest asset. Altogether, fourteen countries agreed to this joint contribution to the *IUCr Newsletter*. Active national scientific associations in the region include thirteen chemical societies [Hungarian Chemical Society (MKE), Turkish Chemical Society (TCS), Union of Chemists in Bulgaria (SHB), Chemical Society of Montenegro (HDCG), Association of Greek Chemists (EEX), Romanian Chemical Society (SChR), Slovenian Chemical Society (SKD), Serbian Chemical Society (SHD), Croatian Chemical Society (HKD), Society of Chemists and Technologists of Macedonia (SHTM), Society of Albanian Chemists (SAC), Pancyprian Union of Chemists (PUC) and Moldovan Chemical Society (MCS)] and many physics societies. Most crystallographers are members of these national organizations, many of which have sections, divisions or interest groups related to crystallography. There are at least

six national crystallographic organizations (Greek Crystallographic Association, Croatian Crystallographic Association, Slovenian Crystallographic Society, Turkish Crystallographic Association, Serbian Crystallographic Society, Bulgarian Crystallographic Society). Many of the societies publish journals where crystallographic reports appear: *Turkish J. of Chem.*, *Acta Chimica Slovenica*, *Croatica Chemica Acta*, *Revue Roumaine de Chimie*, *Hungarian J. Chem.*, *Acta Chimica Hungarica-Models in Chemistry* (merged with two European journals), *Journal of the Serbian Chem. Soc.*, *Bulgarian Chem. Comm.*, *Chemistry-Bulgarian J. of Chem. Ed.*, *Macedonian J. of Chem. and Chem. Eng.*, *Chem. J. of Moldova*, *Chimia Chronika* (merged with two European journals) and *Cyprus J. of Science and Tech.* The merits of cooperation across borders and the efficient utilization of human and technical resources has fostered the emergence of joint activities, including a biannual 'International Conference of the Chemical Societies of the South-Eastern European Countries' (ICOSECS), which was held in Halkidiki (1998 and 2000), Bucharest (2002), Belgrade (2004), Ohrid (2006), Sofia (2008) and Bucharest (2010). The conferences have fostered improved international cooperation in basic and applied chemical research.

The series of articles written for the *Newsletter* contain an impressive panoply of contributions from the researchers from South-Eastern Europe to crystallography. The roots of research in mineralogy and in crystallography in Croatia stretch back to the end of the 19th century. Active crystallographic associations in Croatia and Slovenia have organized joint crystallographic meetings with international participation since 1992. Hungary began optical crystal research in 1928 and hosts the Budapest Neutron Centre, the only such facility in South-Eastern Europe. Crystallography is well established in Greece as a result of the efforts of returnees from the Western Universities, especially in the field of protein research. Recently there has been a rapid increase in the quality and quantity of crystallographic research in Turkey, one of the fastest developing economies in the world. Researchers from Turkey and Cyprus are involved in building SESAME, the Middle East synchrotron source in Jordan. In Serbia, the Serbian Crystallographic Society, which together with other national crystallographic associations has evolved from the former Yugoslav Centre for Crystallography, is active in small-molecule research and has been also holding national meetings since 1992. In Romania increasing numbers of researchers have become involved in X-ray diffraction since a National Centre for X-Ray Diffraction opened in Cluj-Napoca. The Bulgarian Crystallographic Society in Bulgaria and the Division of Crystallography of the Society of Chemists and Technologists of Macedonia in Macedonia have been established recently. There are crystallographic activities in Moldova and structural research in Cyprus, and some crystallographic research, mainly on a collaborative basis, is also being performed in Bosnia and Herzegovina, Albania and in Montenegro.

Regarding the participation of the crystallographers from South-Eastern Europe in the international crystallographic organizations, Alajos Kálmán from Hungary was a member of the Executive Committee of the IUCr from 1984 to 1990 and Vice-President from 1990 to 1993. Drago Grdenić from Croatia was involved in writing the constitution of the European Crystallographic Committee (ECC) in 1972. He was succeeded by S. Šćavničar at the first European Crystallography Meeting (ECM1) in Bordeaux in 1973. Croatian crystallographer Boris Kamenar was Vice-President of the ECC during 1978-1981 and President from 1981 to 1984. After the successful organization of ECM22 in Budapest in 2004, Petra Bombicz from Hungary was elected as Secretary of the European Crystallographic Association (ECA, successor to the ECC) for the period 2006-2009 and re-elected for the period 2009-2012. Three meetings of the ECA were held in South-Eastern Europe, in Keszthely (Hungary, 1974), Budapest (Hungary, 2004) and Istanbul (Turkey, 2009). An ECM meeting planned to be held in Ljubljana in 1991 was shifted to Trieste due to the war. The outstanding success of the 2009 ECM25 meeting, organized by Engin Kendi and her team in Istanbul, demonstrates the potential to hold meetings, workshops and schools of crystallography in South-Eastern Europe.

The collection of texts in this thematic issue is a powerful testament to the ability of researchers, who come from different cultures and speak different languages, to work together for the benefit of

crystallography. It also shows that despite political differences the region is an inseparable part of Europe where people share common values.



Panče Naumov

I thank all contributors for their valuable time spent in meticulous collection and presentation of the information, and the Newsletter staff (Patricia Potter, Judith L. Flippen-Anderson and Bill Duax) for accepting the idea to publish these articles and the effort that they spent in creating it. I apologize for any unintentional errors that might have occurred during the process of editing and preparation of the contributions for publication.

Panče Naumov (pancenaumov@gmail.com)

Crystallography in Croatia

Introduction

Logo of CCA (abbr. in Croatian: hkz; colors of national flag: red, white, blue; author B. Gržeta, CCA).

Crystallography in Croatia began with courses in mineralogy in the last decades of the 19th century. M. Paić used powder diffraction in his PhD studies at the Sorbonne in Paris in the 1930s and in 1946 as the head of the Physics Dept. of Zagreb U. he continued research in crystallography. D. Grdeni&caute; studied crystal structures of mercury compounds by single-crystal XRD in Moscow from 1946 to 1948 with A. I. Kitaigorodskii. In 1948 he founded X-ray crystal structure analysis in Zagreb. Soon a group of talented younger crystallographers (K. Kranjc, S. Šćavnićar, A. Bonefačić, A. Bezjak, B. Kamenar, B. Matković, Z. Ban, M. Sikirica, M. Topić, B. Kojić-Prodić and S. Popović) began studying crystal structures and microstructures.



Drago Grdenić in the 1960s.

In 1966 the crystallographers in the former Yugoslavia established the Yugoslav Centre of Crystallography (YCCr), which acted under the auspices of the Yugoslav (now Croatian) Academy of Sciences and Arts in Zagreb. D. Grdenić was elected President and B. Kamenar Secretary. That same year, YCCr joined the IUCr (at the 7th Congress of IUCr in Moscow). Most of the members of YCCr performed their research in Croatia. From 1966 to 1991, annual crystallographic conferences were held in Yugoslavia, often organized by YCCr members from Croatia. The invited speakers at those conferences were eminent crystallographers from abroad [M. Nardelli, G. Allegra, M. Mammi, G. Gilli, C. Giacovazzo, D. Viterbo and L. Randaccio (Italy), A. Kálmán (Hungary), S. C. Abrahams (USA), I. Olovsson (Sweden), M. Porai Koshic and Y. Struchkov (USSR), J. Helliwell, C. K. Prout and Nobel laureate D. Hodgkin (UK)]. Six joint Italian-Yugoslav crystallographic conferences were organized in that period. Many crystallographers

from Croatia were trained in prominent international laboratories. A number of scientists from other republics of the former Yugoslavia obtained MSc and PhD degrees in Zagreb.

In the seventies and eighties the laboratories in Zagreb were equipped with modern instruments for XRD, TEM and associated methods, enabling research of crystal structures of inorganic, organometallic and organic compounds, as well as microstructure at ambient and elevated temperature. YCCr published a journal, the *Annual of the Yugoslav Centre of Crystallography*, containing (in English) papers based on plenary lectures and abstracts of short contributions given at annual conferences, lists of publications of the YCCr members, titles of MSc and PhD theses of the YCCr members, and minutes of annual YCCr assembly meetings. YCCr was also a member of the European Crystallographic Committee (ECC); B. Kamenar was the Vice-President and President of ECC in the period 1978-1984. Due to the war in Yugoslavia the 13th European Crystallographic Meeting scheduled for Ljubljana, Slovenia, in 1991 was transferred to Trieste thanks to good relations among crystallographers in Italy (L. Randaccio), Croatia (B. Kamenar) and Slovenia (L. Golič). A plenary lecture was given by the Nobel laureate R. Huber (Germany), and a satellite meeting on synchrotron radiation was organized.



Boris Kamenar

After the disintegration of Yugoslavia and proclamation of independence of Croatia in 1991, the Presidency of the Croatian Academy of Sciences and Arts (CASA) dissolved the YCCr. With an advisory board composed of B. Kamenar, B. Kojić-Prodić, S. Popović and B. Gržeta and the unanimous support of crystallographers in Croatia, the Croatian Crystallographic Association (CCA) was founded on February 28, 1992. B. Kamenar was elected President and S. Popović Secretary. The CCA joined the European Crystallographic Association in 1992 (ECM14 Enschede) and the IUCr in 1993.

Since 1992, CCA and the Slovenian Crystallographic Society have organized annual joint Meetings with international participation, alternately in Slovenia and in Croatia. The reports on the meetings can be found in the IUCr Newsletter. Invited speakers at the meetings have included C. Giacovazzo, G. Zanotti (Italy), M. Kaftory (Israel), I. Hargittai (Hungary), M. Jaskólski (Poland), W. Duax (USA), H. Flack, N. Ban (Switzerland), P. Naumov (Japan), R. Dinnebier, H. Fuess (Germany), B. Kobe (Australia), E. and G. Dodson (UK), and E. Tillmanns (Austria). Papers based on plenary lectures are published in *Croatica Chemica Acta* (Croatian Meetings) and *Acta Chimica Slovenica* (Slovenian Meetings). Members of CCA edited a special issue of *Croatica Chemica Acta*, **82** (2009), a *Festschrift* dedicated to D. Grdenić on his 90th birthday. D. Balzar was a member of the IUCr Commission on Powder Diffraction, B. Kojić-Prodić is a Co-editor of *Acta Cryst. C* and S. Popović was a member of the EPDIC Committee.

The CCA has about one hundred members conducting research in at least ten laboratories in universities, scientific institutes and industry. The crystallographers study biologically active molecules, proteins, organometallic compounds, pharmaceuticals, microstructure of composites and its relation to properties. Short reports on research in several prominent laboratories in Croatia are given below.

The members of the Executive Committee of CCA (since January 12, 2006; reelected on May 5, 2009) are: M. Luić, *Secretary*, marija.luic@irb.hr; D. Matković-Čalogović, *Vice Chair*, dubravka@chem.pmf.hr; S. Popović, *Chair*, spopovic@phy.hr; D. Tibljaš, dtibljas@geol.pmf.hr; A. Tonejc, atonejc@phy.hr; A. Višnjevac, aleksandar.visnjevac@irb.hr; K. Vlahoviček, kristian@bioinfo.hr; B. Kamenar, *Honorary Chair*, bkamenar@chem.pmf.hr.

More details on CCA can be found at www.hazu.hr/kristalografi.

Stanko Popović (spopovic@phy.hr)

Laboratory of General and Inorganic Chemistry (LGIC), Dept. of Chemistry, Faculty of Science, Zagreb U.



Boris Kamenar with Dorothy Hodgkin during her visit to Zagreb in 1966.

In 1952 LGIC became the center of crystallography led by D. Grdenić when he published a paper in *Acta Cryst.* describing the electron density distribution on mercury diethylene oxide. In 1955/56 Grdenić worked on the structure of ferroverdin in the laboratory of D. Hodgkin. Upon returning to Zagreb he continued his research of mercury compounds. *The Structural Chemistry of Mercury* published in *Quart. Rev. Chem. Soc.* in 1965 is still one of the most cited papers of a Croatian chemist.



Milan Sikirica



Drago Grdenić inspecting the new diffractometer. Zoran Bojanić (technician in charge of the diffractometers) is in the back.

He had begun with a Unicam camera and three Weissenberg goniometers. Later the Laboratory was equipped with a General Electric powder diffractometer and the first four-circle single-crystal Philips diffractometer (1974).

Grdenić's students and co-workers at the Faculty of Science included: B. Kamenar, M. Sikirica (intermetallic and organomercury compounds), A. Nagl, M. Bruvo, A. Hergold-Brundić, Z. Popović, D. Matković-Čalogović (organomercury compounds), Z. Ban, M. Pušelj (powder diffraction of intermetallic compounds and metal peroxides).



Structure of the antibiotic azithromycin.
B. Kamenar was a visiting scientist in D. Hodgkin's Lab, in All Souls College, at Auckland U. and Massey U. in New Zealand. In Zagreb, he studied inorganic, organometallic and organic compounds, complexes of molybdenum and vanadium (M. Penavić, B. Kaitner, N. Strukan, B. Marković), and pharmaceuticals such as azithromycin and its derivatives (D. Mrvoš-Sermek). I. Vicković worked on computing in direct methods with D. Viterbo in Torino, C. Giacovazzo in Bari, and at Texas Christian U. In Groningen he developed protein data collection strategy. In his doctoral thesis (1977), he was among the first in Croatia to use direct methods for crystal structure analysis.

In 1981, M. Sikirica obtained a second Philips diffractometer (one for molybdenum and one for copper radiation). An Oxford Diffraction Xcalibur 3 with a CCD camera was purchased on the fiftieth anniversary of the Laboratory in 2002.

The first doctoral thesis on protein crystallography was that of D. Matak-Vinković. M. Vinković determined structures of pharmaceuticals and worked at the pharmaceutical company Pliva in Zagreb. Later both Dijana and Mladen Vinković went to Cambridge. A. Nagl led a group at the Faculty of Textile Technology, Zagreb U., with G. Pavlović and M. Cetina. E. Meštrović formed a crystallography group at Pliva (N. Košutić-Hulita, M. Devčić).



Crystallographers in the Laboratory of General and Inorganic Chemistry, Dept. of Chemistry, Faculty of Science, University of Zagreb: (in front, from left to right) Marijana Đaković, Marina Tašner, Nenad Judaš, Zoran Bojanić, (sitting) Dubravka Matković-Čalogović, (back, from left to right) Dominik Cinčić, Biserka Prugovečki, Draginja Mrvoš-Sermek, Zora Popović, Marija Zbačnik, Vladimir Stilinović, Branko Kaitner.



Part of the protein crystallography group in Elettra, Trieste (left to right): Ivica Đilović, Dubravka Matković-Čalogović and Dalibor Milić.
Today four groups are active at LGIC. B. Kaitner's group studies intra- and intermolecular interactions and their effect on structures and properties using synthesis, crystal structure determination and solid-state analytical methods. Materials being studied include organic salts, triacylmethanes, Schiff bases, β-

diketones and coordination compounds of transition metals. Zora Popović's group is focused on preparation and structure determination of metal complexes of the late 3*d*-block and group 12 metals with molecules of biological importance to elucidate their role on the formation of polymorphic forms. M. Cindrić's group studies anion-directed self-assembly of flexible ligands into highly symmetrical organic solids, complex compounds, coordination polymers and polyoxometalates of vanadium, molybdenum and tungsten, and their interactions with amino acids, pyridoxal derivatives and thiosemicarbazones. They are investigating the catalytic role of vanadium and molybdenum in biologically active molecules. D. Matković-Čalogović's group works on small molecules and protein crystallography (with G. Za, G. Dodson and A. Antson in York). She had a Fulbright fellowship at the U. of Kansas Medical Center. Her group studies modified insulin structures (B. Prugovečki), native and mutated forms of tyrosine phenol-lyase (TPL) in complexes with the substrate analogues and bovine 3,4-dioxygenase, as well as small molecule model systems of enzymes and supramolecular architectures (N. Judaš). Courses on crystallography are taught at the Dept. of Chemistry.

Dubravka Matković-Čalogović (dubravka@chem.pmf.hr)

Dept. of Physics, Faculty of Science, Zagreb U.

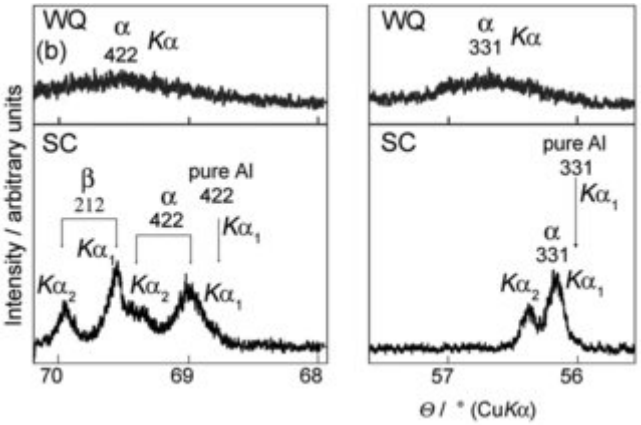


Department of Physics, Faculty of Science, University of Zagreb.

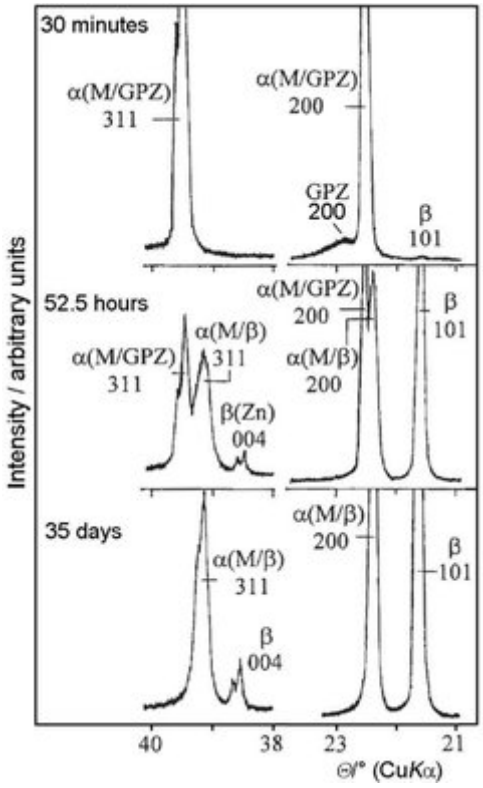
The earliest crystallographic activity at the Dept. of Physics of the Faculty of Science, Zagreb U., began in 1954 with the PhD work of K. Kranjc (1915-1989), *Investigation of poly-dispersive systems with small-angle X-ray diffraction*. Her supervisor, M. Paić (1905-1997), initiated crystallography in the Dept. after using the Debye-Scherrer technique at the Sorbonne in Paris. When he was appointed head of the Dept. of Physics in 1946 only a Unicam-25 camera was available. K. Kranjc continued to use SAX and Berg-Barrett X-ray diffraction microscopy to investigate the domain structure and Moiré fringes in electron micrographs. Kranjc worked in the laboratory of A. Guinier in Paris and A. Mackay at Birkbeck College. She was a friend of Rosalind Franklin who visited Zagreb in 1953.



Slo-Cro Cryst Meeting 2010: on the left Antun and Anđelka Tonejc, on the right Stanko Popović.



52Al-48Zn alloy: difference in microstructure between the quenched (WQ) and slowly cooled (SC) alloys from the solid-solution temperature to RT (*Croat. Chem. Acta* **82** (2009) 405-420).



62Al-38Zn alloy: a gradual transition of the quenched alloy, during ageing at RT, from a metastable system, α+Guinier-Preston zones, into a more stable system, α+β (*Croat. Chem. Acta* **82** (2009) 405-420).

A. Bonefačić earned his PhD (*X-ray structural analysis of some Ag-II sulfates*) with M. Paić as the supervisor. He worked on diffuse scattering in the Guinier lab. In 1966 he became the head of the research group in physics of metals, and in 1971 received the Croatian National Science Award for Physics (CNSAP) for his scientific research on crystallographic properties of metastable alloys. In 1967 Bonefačić and his colleagues (D. Kunstelj, M. Stubičar and Anđelka and Antun Tonejc) initiated structural studies of metastable metallic alloys obtained by rapid quenching from the melt using a Siemens diffractometer and Philips 120 kV transmission electron microscope EM300. Anđelka Tonejc was a postdoctoral student in the Guinier Lab in Orsay from 1974 to 1976, where she worked on rapidly quenched alloys using powder diffraction and TEM. In 1991 Antun Tonejc established the Laboratory for Microstructural Investigation of Materials with a new Philips powder diffractometer and JEOL 200 kV high-resolution transmission electron microscope. In 1995, Antun Tonejc became head of the laboratory and the staff expanded to include S. Popović (full professor) I. Đerđ, Ž. Skoko and I. Lončarek.

Anđelka Tonejc focused on high-resolution electron microscopy investigations and, with I. Đerđ, was one of the first researchers to use the Rietveld method for electron diffraction analysis. Antun Tonejc initiated structural examination of mechanically alloyed materials using high-energy ball milling and received the Science Award of Croatian Academy of Sciences and Arts (SACASA) in 2008 for investigations on phase transitions in nanostructural materials.

S. Popović concentrated on X-ray diffraction line profile analysis, microstructural investigations of Al-Zn and Al-Ag-Zn alloys, and mixed metal oxides in collaboration with the Dept. of Materials Chemistry, Ruđer Bošković Inst. Popović received the CNSAP in 2001 and became Fellow of CASA in 2004. Department courses are taught in solid state physics and crystallography.

Antun Tonejc (atonejc@phy.hr)

Institute of Mineralogy and Petrology, Faculty of Science, Zagreb U.

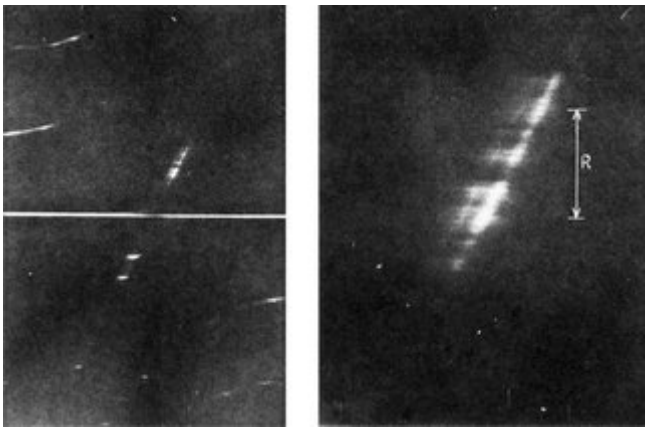
The early days of crystallography in Croatia were linked to minerology. The Chair of Mineralogy and Geology (CMG) was established in 1874, by decree of the Emperor of the Austro-Hungarian Empire. Dj. Pilar started teaching there in the spring of 1875 and crystallography has remained one of its primary activities. After Dj. Pilar, the CMG was headed by eminent professors, and members of the Academy of Sciences and Arts. The first mineralogists from Bulgaria, G. Bončev and L. Vankov, obtained their doctors degrees under the supervision of Dj. Pilar.

Early research activities involved morphological studies of crystals and symmetry determinations by microscopic methods which are often neglected now. Optical crystallographic methods were developed and applied to minerals and other crystalline substances. Lj. Barić contributed to the development and teaching of these methods. Equipment for X-ray analysis consisted of reflexion goniometers, polarizing microscopes, X-ray diffraction cameras, modern diffractometers and electron microscopes.

From 1955 S. Šćavničar headed crystallographic research at the Inst. determining the structures of natural and synthetic compounds. In the 1950s he achieved the first successful hydrothermal synthesis of beryl. He taught generations of masters and doctoral students how to use powder and single-crystal diffractometry, electron microscopy, methods of thermal analysis, Raman and IR spectrometry and microanalysis to determine new mineral structures and improve characterisation of known ones. More than 10 new minerals were identified in the Inst. of Mineralogy and Petrology. The research involves complex investigative methods and precise determination of crystal morphology, optical constants, specific gravity, hardness, cleavage, electric and magnetic properties, chemical composition and genetic information. Crystallographers collaborate closely with investigators in associated fields. New areas of research include experimental mineralogy, biomineralization processes and environmental mineralogy. V. Bermanec and D. Tibljaš are training younger colleagues at the Inst. and collaborating with researchers in Croatia, Macedonia, Slovenia, Bosnia and Hercegovina, Austria, Hungary, Canada, Brazil and Russia.

Vladimir Bermanec (vberman@public.carnet.hr)

Dept. of Mineralogy, Faculty of Mining, Geology and Petroleum Engineering, Zagreb U.

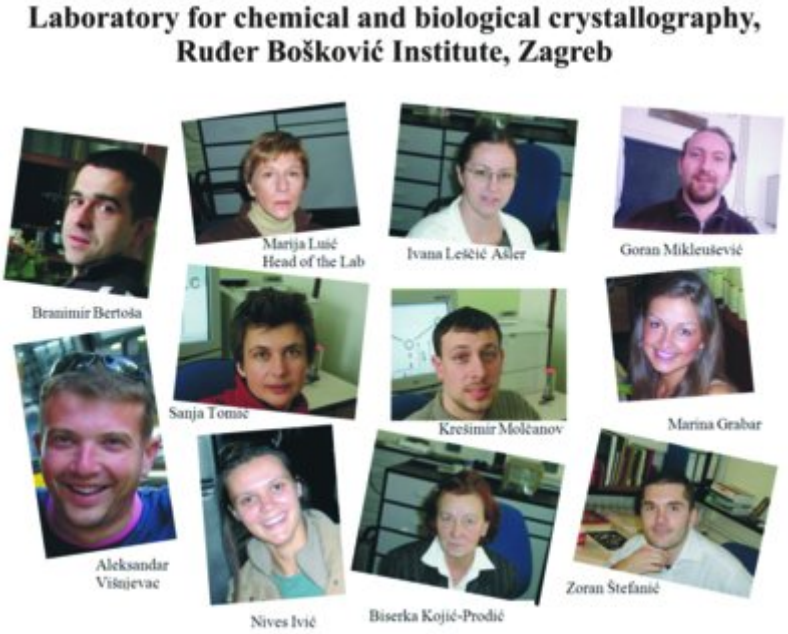


Weissenberg pattern of a biotite polytype with 17 single mica layers.

Microstructures of phyllosilicates, biotites and muscovites, taken from the Papuk Mts, have been studied by single-crystal and powder diffraction methods and related to physical and chemical properties. Polytypes of variable complexity up to 20 single mica layers were detected in biotite. In a number of cases an epitactic overgrowth of ordered and partially ordered polytypes were observed. The parameters of the 1M sub-cell of biotite, deduced from zero-layer rotation patterns, depended on the chemical composition and not on the particular polytype.

Dragutin Slovenec (bvlainic@rgn.hr)

Lab of Chemical and Biological Crystallography, Ruđer Bošković Institute (RBI), Zagreb



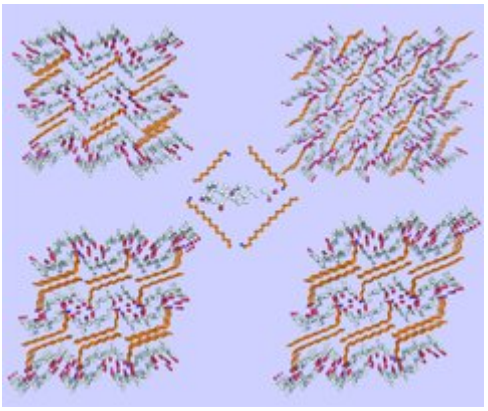
Staff of the Laboratory of Chemical and Biological Crystallography, Ruđer Bošković Institute, Zagreb.

X-ray structure analysis was initiated in Croatia by D. Grdenić after his doctoral studies in Moscow. His first rotation photograph was taken on New Year's Eve 1948 in the Physics Dept. of the Faculty of Science. A Goverment Decree established the Ruđer Bošković Inst. (RBI) for fundamental research in physics and chemistry. The institute was founded by I. Supek to foster collaborative research with scientists from Zagreb U. In 1951 the first Weissenberg goniometer was purchased, data were collected on film and a subscription to *Acta Crystallographica* was started. D. Grdenić was the founder and first head of the Dept. of Structural and Inorganic Chemistry, and supervised the work of A. Bezjak, S.

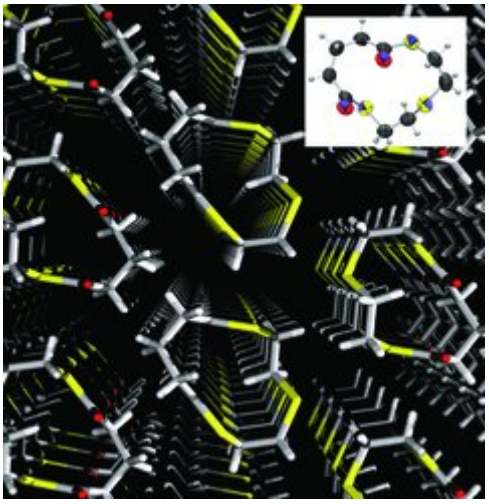
Šćavničar and B. Matković. Scientific results included determinations of structures of organic molecules, phthalylurea and mellitic acid solved by original direct methods designed by Bezjak. Structures were solved from projections. The existence of a novel mercurated oxonium species, as a part of PhD thesis of Šćavničar, was published in *Nature* (1953). The structure of thorium(IV) acetylacetonate, solved by Matković, revealed an Archimedean antiprism as the coordination polyhedron around thorium.



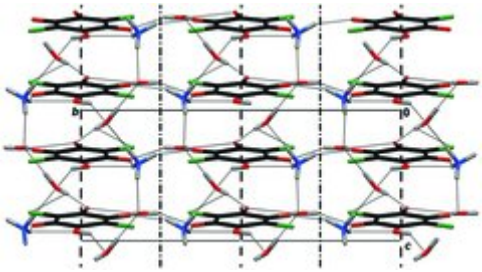
Two views of the single-stranded DNA-binding protein from *Streptomyces coelicolor*, the first X-ray structure of an SSB protein from a member of the genus *Streptomyces* (Z. Štefanić, D. Vujaklija and M. Luić, *Acta Cryst.* D**65** (2009), 974-979).



Co-crystals of cholic acid (CA) with *n*-alkylammonia (*n* = 10, 12, 14, 16) represent first examples of CA's co-crystals with molecules of comparable size. In one of the compounds a completely new type of bilayer arrangement was found (upper right). (V. Tomašić, Z. Štefanić, *CrystEngComm* **9** (2007) 1124-1128).



Solid-state tubular assemblies of thiolactones(I. Vujasinović, J. Veljković, K. Mlinarić-Majerski,K. Molčanov, B. Kojić-Prodić, *Tetrahedron* **62** (2006) 2868-2876).



π-Stacking of quinoid rings, described for the first time, in crystals of ammonium hydrogen chloranilate dihydrate (K. Molčanov, B. Kojić-Prodić, A. Meden, *CrystEngComm* **11** (2009) 1407-1415).
Since the founding of the laboratory in the 1950s two research lines have been maintained: the development of experimental techniques and numerical methods of analysis. Subjects of study included synthesis of heavy metal alkaline phosphates by a melt technique and transition metal complexes. A new type of ferroelectric without hydrogen bond was discovered. When direct method programs, such as *Multan*, became available, biologically active molecules became the targets of research. These included the plant growth hormone auxin and its analogues, peptides, sugars, and pharmaceutically active compounds (*i.e.* cimetidine, ranidine and diltiazem). Since 1985 the Cambridge Structural Database has been available in Croatia via the National Affiliation Centre of Cambridge Crystallographic Data Centre. In 1989 the first diffractometer was purchased. For over forty years chemical crystallography has been combined with spectroscopic methods, bio-assays and molecular modelling to understand intramolecular and intermolecular interactions, the topology of hydrogen bonds and the physical properties of chemical and biologically active molecules and their relation to functions. As the activities were more and more concentrated on biologically active molecules and their interactions, the Laboratory for Chemical and Biological Crystallography was founded in 1997 for research with protein crystallography. Structures of enzymes complexed with substrates and inhibitors are revealing catalytic mechanisms at the molecular level by combining structural, biochemical and genetic methods of analysis.

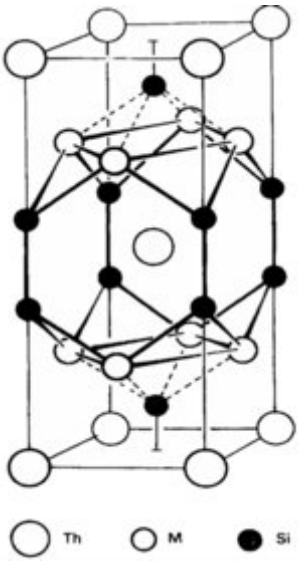
Biserka Kojić-Prodić (kojic@irb.hr)

Powder Diffraction at RBI, Zagreb

Powder diffraction is used in the laboratory for analysis of micro- and nanocrystalline materials. New methods for accurate unit-cell parameter measurement have been developed based on the separation of adjacent diffraction lines and by combining single-crystal zero-layer rotation and Weissenberg patterns with powder diffraction patterns. Techniques for quantitative phase analysis of multicomponent samples are being developed. Phase diagrams of semiconductors are related to electrical properties. New high-temperature phases are characterized. Microstructure and phase diagrams of a series of mixed metal oxides (*e.g.* ferrites, orthoferrites, alumina, zirconia, titania) were systematically studied by X-ray powder diffraction, Raman, FT-IR and Mössbauer spectroscopies, in collaboration with the Dept. of Materials Chemistry (RBI). The processes of biomineralization of *bivalvia* have been studied in detail in collaboration with the Center for Marine Research (RBI).

Stanko Popović (spopovic@phy.hr)

Laboratory of Solid State Chemistry, RBI



The ThM_2Ge$_2$ structure type published in *Acta Crystallographica*.

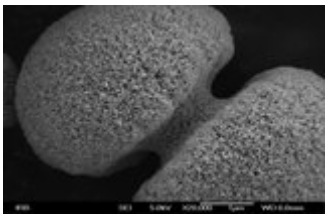


Germanium single crystals prepared by float zone crystal growth technique.

Crystallographic analysis began in the Lab for Solid State Chemistry (formerly High Temperature Materials) at RBI in the 1950s with studies of the crystal morphology and (di)electric properties of Rochelle salt (M. Topić). Subsequently, float zone crystal growth, uranium and thorium ternary silicides and germanides containing transition metals were analyzed with neutron and X-ray powder diffraction methods (M. Tudja). Chemical vapour transport technique was used for preparation of ternary uranium compounds of composition UNTe, UNAs and UAsSb. Powder diffraction analysis revealed that they crystallized in the tetragonal structure of the PbFCl type (Z. Despotović, R. Trojko). The technique of isothermal transport reaction between metals in molten alkali chloride mixtures was developed and applied to systems containing combinations of Cu, Ni, Ag, Pt, Cr and Fe metals (M. Paljević). Research on multicomponent intermetallic compounds started in the 1970s. A systematic study of phase equilibrium was carried out on selected rare earth-iridium systems. Substitution of one or both components in binary intermetallic compounds of the general composition AB_2 and AB_5 with other metals or metalloids has been used as a standard method for tailoring materials suitable for hydrogen storage purposes. Thermodynamic properties of the corresponding intermetallic compound-hydrogen systems were determined from pressure composition isotherms at various temperatures obtained by tensimetric methods (Ž. Blažina, A. Drašner, B. Šorgić). The crystal structures of fifty intermetallic compounds have been deposited in Pearson's Handbook of Lattice Spacings and Structures of Metals, the Powder Diffraction File Search Manual of Inorganic Compounds and the Metal Hydride Reference List of Sandia National Laboratories. Recent intensive investigations concern theoretical studies of intermetallic compounds based on electronic structure calculations designed to parametrize the structural basis for the thermodynamic, electrical, chemical and magnetic properties of these materials (G. Miletić).

Želimir Blažina (blazina@irb.hr)

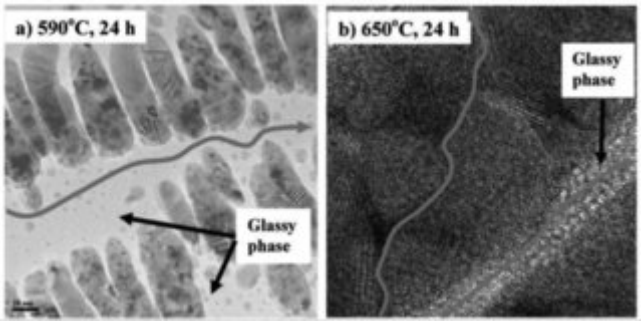
Dept. of Materials Chemistry, RBI



Two interconnected cupolas of α-Fe_2O_3 precipitated from dense α-FeOOH suspension with added ammonium amidosulfonate (M. Žic, M. Ristić, S. Musić, *J. Mol. Struct.* **235** (2009) 924-926).
Almost four decades ago S. Musić initiated research in chemistry of metal oxides at the RBI. He prepared metal oxide carriers labelled with cyclotron radioisotopes for applications in nuclear medicine and investigated hydrolytic processes and precipitation of iron oxides. A research group was formed and started systematic investigations of other metal oxide systems such as ZrO_2, TiO_2, ZnO, Ga_2O_3, Nb_2O_5 and WO_3. The goals were to determine the relationship between the synthesis route and specific properties of metal oxide particles and the kinetics and mechanisms of precipitation of metal oxides. The formation and properties of ferrites with spinel-, perovskite- or garnet-type structures were investigated. The application of powder diffraction in combination with other techniques (Mössbauer, FT-IR, Raman, electron microscopy) made it possible to study the structure, phase composition, phase diagrams and chemical bonds, as well as the size and morphology of the particles of metal oxides.

Svetozar Musić (svetozar.music@irb.hr)

The Glass Laboratory, RBI



Easy conductive pathways in partially crystallized 40Fe_2O_3-60P_2O_5 (mol%) glass through a) glass matrix and b) grain boundaries.
The Glass Laboratory at RBI (A. Moguš-Milanković, A. Šantić, L. Pavić) employs crystallography in conjunction with electrical characterization to study the relationship between the composition, structural and electrical properties of various materials. Principal techniques in electrical characterization include Impedance Spectroscopy (IS) and Thermally Stimulated Polarisation Current (TSDC/TSPC) measurements combined with Raman spectroscopy and XRD techniques. Research programs cover transition metal oxide glasses, bioactive materials and ionic liquid composites. Special interest is focused on the mechanism of crystallization of amorphous systems (phosphate, silicate, bioactive glasses), determination of crystalline phases, particle size and volume fractions, and their influence on electrical properties. Part of the research considers a surface activity of the electrically polarized bioactive materials.

Andrea Moguš-Milanković (andrea.mogus-milankovic@irb.hr)

Crystallography in Bosnia and Herzegovina

A few scientists began crystallographic research at the Faculty of Science of the U. of Sarajevo in Bosnia-Herzegovina in 1960-1970. The equipment was in the Dept. of Physics. B. Ribar worked with chemistry and physics majors on identification of materials and X-ray structure analysis. At that time, a chemist,

two physicists and a geologist were trained in crystallography. Education in crystallography was acquired at the Zagreb U., the Ruđer Bošković Inst. in Zagreb or the Vinča Inst. in Belgrade. By 1991, X-ray analysis of single crystals was used for identification of organic, inorganic and synthesized compounds, and powder diffraction methods were used for minerals.

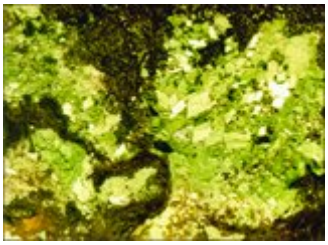
Crystallographers focused on acquiring instruments for experimental work and used their crystallographic knowledge to teach students. The facilities at their disposal were extremely limited, and their pioneering efforts were laudable. Until 1991, there were crystallography laboratories at the Technology faculty in Tuzla, at the Inst. Kemal Kapetanović in Zenica, and at the laboratory in the alumina factory in Zvornik. The Fund for scientific research in Bosnia-Herzegovina supported most of the crystallography-related projects.

The crystallographers of Bosnia and Herzegovina collaborated and published their results together with colleagues from Zagreb, Belgrade and Ljubljana. They were members of the Yugoslav Centre of Crystallography (YCCr). Conferences were held once a year at various places in former Yugoslavia. The proceedings of the meetings were published in English in the *Annual of the Yugoslav Centre of Crystallography*.

Chemist M. Šljukić and physicist F. Gabela are active crystallographers, who collaborate with scientists from the Ruđer Bošković Inst., where they had worked on their PhD theses. F. Gabela acquired his doctoral degree in 1977 from the Faculty of Science and Mathematics, Sarajevo U., under the supervision of B. Kojić-Prodić with the thesis 'Crystal structures of peroxopentafluoroniobate and oxoperoxotetrafluorotungstate'. His research includes studies of zirconiumtetrafluorides, gall stones and kidney calculi. M. Šljukić obtained his doctoral degree from Zagreb U. under the supervision of B. Matković (1968), for structural characterization of double phosphates of Zr(IV), Hf(IV), Th(IV) and U(IV) with alkali metals.



Large crystal twins of the mineral hyalophane, (K,Ba)[Al(Si,Al)Si_2O_8] from Busovača, Bosnia and Herzegovina.



Mineral gaspeite, (Ni,Mg,Fe)CO_3, from Duboštica, Bosnia and Herzegovina, which was first determined in 1998 (Bermanec, V., Sijarić, G., Kniewald, G., Mandarino, J. A. *Can. Miner.* **38** (2000), 1371-1376).
G. Sijarić, professor of mineralogy and crystallography, was educated at the Inst. of Mineralogy and Petrography of the Natural Faculty of Sciences, Zagreb U., and at the Ruđer Bošković Inst. She obtained a

doctoral degree in 1975 from the Faculty of Science and Mathematics of Sarajevo U., under the supervision of S. Šćavničar of Zagreb U. In her thesis, for the first time, she applied quantitative X-ray analyses to bauxites (oxides and hydroxides of Al, Fe, Ti) and silicates (serpentines, zeolites, plagioclases). Their structures and order were defined in plagioclases, and the structures of new minerals and their distribution in Bosnia-Herzegovina was described. In a 1978-1980 project, crystallographic data on numerous minerals, including those from the hundred-years-old collection at the National Museum in Sarajevo, were catalogued. Other targets for study included kidney stones and gall stones of citizens from Sarajevo. G. Sijarić has taught crystallography and mineralogy to chemistry, geology and geography majors at the Sarajevo U., Tuzla and Priština.

During the war in Bosnia-Herzegovina in the 1990's all instruments and facilities for scientific research were devastated. Because it has not been economically possible to repair or replace the needed instruments, research depends on cooperation with scientists and institutions from neighbouring countries.

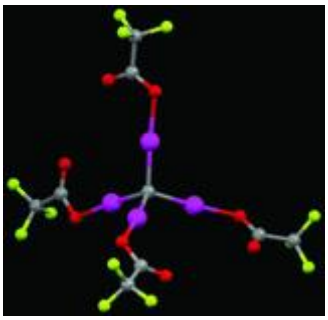
At present, there are a few scientists using crystallographic data at the Chair of General and Inorganic Chem., Chem. Dept., Faculty of Natural Sciences. The Head of this group E. Kahrović (emira_kahrovic@yahoo.com) received a PhD for crystallographic characterizaton and structures of Pb-azides at the Natural Sciences Faculty in Sarajevo in 1996. She now studies synthesis and crystallographic characterization of ruthenium(III) complexes with monobasic (NO) and dibasic (ONO) Schiff bases derived from salicylaldehydes.

Bosnia-Herzegovina currently has no crystallographic Association. Researchers network with the associations in Slovenia and Croatia to gain information about conferences and other events. Financial support is very limited. The only X-ray diffraction instruments are in the factories of aluminium in Mostar and alumina in Zvornik, where they are used for quality control.

Galiba Sijarić (gsijaric@bih.net.ba)

Crystallography in Macedonia

The Republic of Macedonia is a small country (~25 700 km^2) located in the central Balkan peninsula in South-Eastern Europe, with a population of about 2 million. The first X-ray scattering measurements reported by a Macedonian researcher were those of S. Pocev of the Faculty of Technology and Metallurgy (SS. Cyril and Methodius U.) in Skopje, who worked with G. Johansson at the Royal Inst. of Technology in Stockholm, Sweden. Pocev published the paper *An X-ray Investigation of the Coordination and the Hydrolysis of the Uranium(IV) Ion in Aqueous Perchlorate Solutions* in *Acta Chemica Scandinavica* in 1973. Later, he completed his PhD thesis on structures of mercurated oxonium and sulphonium salts at the U. of Zagreb (1978), and the results were published in the *Journal of the Chemical Society*.



The crystal structure of tetrakis(trifluoroacetoxymercury)methane (*J. Chem. Soc. Chem. Comm.* **546** (1974), 646-647).

From 1972 through 1974, G. Jovanovski from the Inst. of Chemistry (Faculty of Natural Sciences and Mathematics, FNSM) in Skopje was a student in the X-ray Laboratory of D. Grdenić and B. Kamenar at the U. of Zagreb, where he determined the crystal structure of tetrakis(trifluoroacetoxymercury)methane,

the first example of a carbon atom bound to four mercury atoms. Later, Jovanovski determined crystal structures of metal saccharinate complexes for his PhD thesis at the same University. With J. Thomas (Uppsala U.), he studied the deformation electron density of potassium oxalate monohydrate.

Returning to Skopje in the 1980s, Jovanovski introduced a course in crystallography at the SS. Cyril and Methodius U. in Skopje as a part of the Structure of Molecules major, in the Dept. of Chemistry at FNSM. At that time, two powder diffractometers were available in Macedonia - a JEOL instrument at FNSM, and a Philips instrument operated by the Faculty of Technology and Metallurgy. The instruments were used for routine analyses, characterization, simple experiments and teaching. In the following years, a small group of crystallography researchers was established at the Inst. of Chemistry, including A. Mirčeva, O. Grupče, P. Naumov and P. Makreski. Sporadically, X-ray diffraction was employed for structure determination and characterization by other members of the Inst. of Chem.: B. Šoptrajanov, V. Jordanovska, V. Petruševski, S. Aleksovska, V. Stefov, Gj. Petruševski and T. Runčevski.



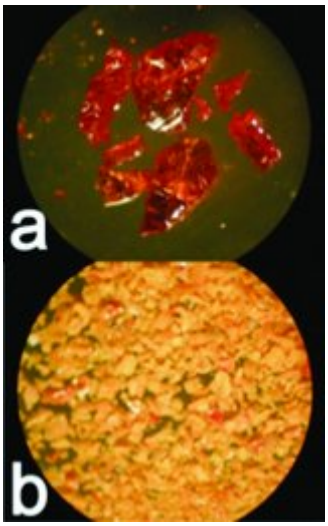
From left to right: Petre Makreski, Gjorgi Petruševski, Gligor Jovanovski, Panče Naumov and Tomče Runčevski.

When the country was part of SFR Yugoslavia, Jovanovski attended the annual meetings of the Yugoslav Centre of Crystallography and organized the 1981 meeting in Skopje. Since 1996, Jovanovski has participated in the annual Croatian-Slovenian crystallographic meetings, together with other Macedonian crystallographers (S. Pocev, V. Jordanovska, A. Mirčeva, O. Grupče, P. Naumov and P. Makreski). Recently, a Division of Crystallography was established as part of the Society of Chemists and Technologists of Macedonia.



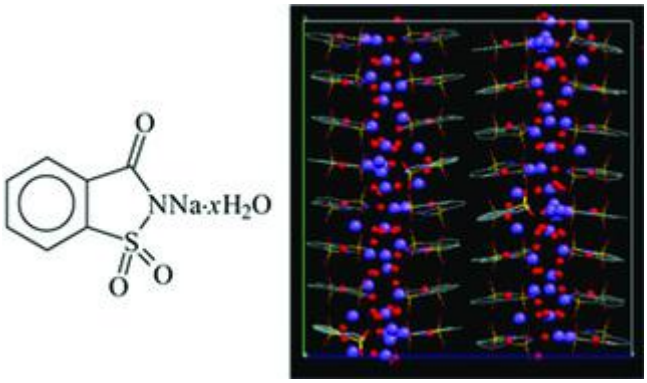
Genuine crystals of the mineral lorandite ($TlAsS_2$) from Alšar, Macedonia.

None of the national or private universities in Macedonia has modern crystallographic equipment. Active Macedonian crystallographers have conducted research in foreign universities, or in collaboration with such institutions. P. Naumov and P. Makreski, who were mentored by Jovanovski, are currently active in the field. The main studies underway in Macedonia concern chemical crystallography (especially metal-coordination compounds as models for biological effects of food additives), inorganic compounds (spectra-structural correlations in isomorphous and isotypic series), organic compounds with interesting electronic and optical properties, solid-state reactivity (photochromism and thermochromism), and polymorphism of pharmaceuticals. An *Atlas of Minerals from the Republic of Macedonia* is to be published soon (in English) by the Macedonian Academy of Sciences and Arts.



Crystals of the genuine Macedonian mineral realgar (As_4S_4) before exposure to visible light (a) and after photochemical conversion to pararealgar (b) (*J. Am. Chem. Soc.* **132** (2010), 11398-11401). Copyright: American Chemical Society.

One of the most active research groups of the Division of Crystallography in Skopje (G. Jovanovski, O. Grupče and P. Naumov) has over 40 publications in international journals, which are mainly related to the coordination chemistry of saccharin (an artificial sweetener with suspected carcinogenic activity), thiosaccharin and their mixed complexes with aromatic bases. In 2004 P. Naumov earned a PhD degree in diffraction at the Tokyo Inst. of Technology, under the supervision of Y. Ohashi. He established a laboratory for the study of photoinduced phenomena and solid-state chemistry at the National Inst. for Materials Science in Japan (2004-2007), and now leads a research group at Osaka U. (Japan). In 2009 Naumov was appointed associate professor of the Inst. of Chemistry in Skopje, where he has been spearheading the development of collaborative crystallographic research in Macedonia. The current interests of the group are materials exhibiting photoinduced and thermal phase transitions for (opto)spintronics applications, X-ray photodiffraction and time-resolved diffraction using synchrotron X-rays.



Sodium saccharinate, $NaC_7H_4NO_3S.xH_2O$, listed in most catalogues as a dihydrate (x = 2), has been extensively used as a food additive and has constituted the basic component of the diabetic's diet for about 125 years. This crystal structure, with Z' = 16 and a very large unit cell, represents one of the most difficult cases for a small molecular species such as the saccharinate ion. The composition is best described as $Na_{64}(C_7H_4NO_3S)_{64}.120H_2O$ (*Angew. Chem. Int. Ed.* **44** (2005), 1251-1254).

Macedonian crystallographers P. Naumov and G. Jovanovski, together with B. Kaitner (Zagreb), D. A. Rae (Canberra) and S. W. Ng (Kuala Lumpur), determined the crystal structure of the sweetener sodium saccharinate, $Na_{64}(C_7H_4NO_3S)_{64}.120H_2O$, a commercial compound incorrectly assumed to have been a dihydrate. This extremely complicated modulated structure with a unit cell of 15.6 nm^3 and Z' = 16 (Z = 64) was ranked #6 in the world's database of small-molecule structures with high Z' (U. of Durham). The Macedonia research team has also described the first direct observation of an all-solid autocatalytic

reaction set, a group of photo-triggered reactions proceeding in the crystals of the natural mineral realgar (*J. Am. Chem. Soc.* **132** (2010), 11398-11401). Ways and means of upgrading crystallographic instruments in Macedonia are being sought, and the advice and assistance of the international community would be greatly appreciated.

Gligor Jovanovski (gligor@pmf.ukim.mk) and Panče Naumov (npance@wakate.frc.eng.osaka-u.ac.jp)

(See also Part 2)

MEETING REPORT

The HEC community meets at Schöneck

Schöneck, Germany, September 23-25, 2010
www.uni-leipzig.de/~straeter/hec13

The community of macromolecular crystallographers in central Europe may be of recent vintage but it already has its own unique traditions. The young protein crystallographers in the heart of Europe have been gathering each year for the Heart of Europe bio-Crystallography (HEC) meetings. The 2010 HEC-13, held in Schöneck in the rolling hills of Vogtland, Germany, assembled 121 participants from protein labs in Germany, Poland, the Czech Republic and Austria. The community has grown from 9 groups at HEC-1 (1998) to 22 groups at HEC-13. This simple statistic shows that the idea behind the HEC initiative, *i.e.* fostering protein crystallography in the region, is bearing fruit. The 26 talks delivered mostly by students and postdocs were of very high quality in scientific content and the form of presentation. The HEC awards, sponsored by the IUCr, went to Bernd Gardill (U. of Erlangen) for 'Don't behave like a rat: Corticosteroid-binding globulin and the serpin *S*→*R* transition' and to Ulrike Bräuer (MLU Halle) for 'Structural studies of the aminotransferase LivB'.



Participants of the HEC-13 meeting.

The lively discussions that followed the talks were dominated by the younger participants. The first session was skillfully chaired by Udo Heinemann. If gauged by activity age^{-1} of the discussants, HEC-13 must be considered the most successful to date. Group leaders are allotted time to briefly introduce new groups. This year, Markus Wahl presented the research profile of his team at the Free U. of Berlin and Mikio Tanabe introduced himself as a junior group leader at Martin Luther U. (Halle). Three other groups, from the Max-Delbrück-Center (Berlin), the International Inst. of Molecular and Cell Biology (Warsaw) and Humboldt U. (Berlin), will be introduced by their leaders at HEC-14. The program traditionally includes a special HEC lecture by a key methods developer or highly respected scholar. For HEC-13 this lecture was presented by Alexander Popov (ESRF), who discussed the practical aspects of diffraction data quality with an emphasis on the effects of radiation damage.

The meeting was organized by Norbert Sträter and his team. Special thanks go to Christian Roth, Bartholomeus Küttner and Gabriele Büttner (secretariat). The program offered an excursion to a space travel museum, a hike into the Vogtland hills, or a few relaxing hours in the Ferienpark's AquaWorld. In summary, the HEC family has had another successful reunion, with excellent science, ample time for exchanging ideas, good atmosphere, good food, and excellent company.

HEC-14 (September 29-October 1, 2012) will be organized in Żagań, Sagan, to commemorate Johannes Kepler, whose book 'De Nive Sexangula' ('On the Six-Cornered Snowflake') heralded in 1611 the birth of modern crystallography.

Mariusz Jaskolsk

AWARDS AND PRIZES

Ninth Ewald Prize



Eleanor Dodson



Carlo Giacovazzo



George M. Sheldrick

The IUCr is pleased to announce that E. Dodson (Dept. of Chemistry, U. of York, UK), C. Giacovazzo (Inst. of Crystallography-CNR, Bari, Italy) and G.M. Sheldrick (Lehrstuhl für Strukturchemie, Göttingen, Germany) have been awarded the ninth Ewald Prize for the enormous impact they have made on structural crystallography by designing new methods and providing these in algorithms and constantly maintained, renewed and extended user software. Their invaluable contributions to the computational side of the field have led to leadership with the program suites *CCP4*, *SIR* and *SHELX*, respectively. Daily all over the world thousands of crystallographers are profiting from their excellent achievements. The presentation of the Ewald Prize will be made during the Madrid Congress Opening Ceremony on August 22, 2011.