

INTERNATIONAL UNION OF CRYSTALLOGRAPHY

IUCr Newsletter

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Original article text and available illustrations

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LETTER FROM THE PRESIDENT

Letter from the President

'Never send to know for whom the bell tolls, it tolls for thee' - John Donne

The devastating tsunami reminds us all of how fragile life is and how vulnerable we all are. We are especially concerned about the impact of the tragedy upon crystallographers in the countries directly affected by the disaster.

The US community was stunned to learn that among those lost were Muttaiya Sundaralingam and his wife, Indrani, who were attending a family reunion in Sri Lanka. The many students and colleagues that Sunda touched during his long and illustrious career mourn his passing.

At the Budapest meeting of the Executive Committee of the IUCr, the many excellent nominations submitted by the National Committees for officers and membership of the IUCr Executive Committee were reviewed. The Executive Committee as a whole felt a responsibility to select a subset of the nominations to be placed on their official list of nominees. The list of nominees proposed by the Executive Committee was sent to all National Committees on November 9, 2004. The list appears on page 2. According to the bylaws, additional nominations for all positions (other than Past President) may be made by the delegates to the Congress. The procedures to be followed are clearly defined in the bylaws.

A summary of other matters addressed by the Executive Committee in Budapest, masterfully prepared by the Executive Secretary, Mike Dacombe, appears on Page 4.

A review of the records of the composition of the Executive Committee over the course of its 40 year history reveals that the 19 past presidents have come from only 10 of the 40 member countries. They include four presidents each from the UK and USA, three from France, two from The Netherlands and one each from Germany, Italy, Japan, New Zealand, Russia, and Sweden. The 199 members of the Executive Committee during its 50-year history are heavily represented by persons from European member countries (64%), with the rest coming equally from AsCA (17%) and ACA (18%) regional member countries. There are 15 member countries that have never had a representative on the Executive Committee in its 50-year history.

I have previously suggested in these pages that it may be appropriate to consider expanding the size of the Executive Committee to involve leading crystallographers from more member countries in order to better serve the needs of the whole community. This will be particularly so if we are successful in expanding the total membership of the Union to over 50 countries. Some mechanism to insure more equitable representation by our regional affiliates could contribute substantially to the expansion of country membership since the AsCA and ACA regions currently include many potential new member countries.

Resistance to expanding the committee is usually based on the argument that a large committee is unmanageable and ineffective. I am of the opinion that if members of a committee are dedicated and hardworking, increased size is an asset.

The Union needs may well warrant consideration of expansion. Although the IUCr Executive Committee is currently comprised of 10 members, its membership was as high as 12 in the past. An expansion to three members from each of the Regional Affiliates together with the four officers (with no restriction on their regional affiliation) would only number 13, just one more than the IUCr Executive Committee membership in 1966-69.

A review of the list of candidates for membership of the Executive Committee submitted by 24 National countries indicates how well each of these excellent candidates could have served the needs of crystallographers throughout the world. An expansion of the size of the Executive Committee would require revisions to the bylaws, another matter for possible consideration by the delegates to the Florence General Assembly.

The Executive Committee of the IUCr also reviewed recommendations from the National Committees of the Union for membership in the many commissions of the Union. Nominations for membership on the commission were forwarded to the Secretary of each Commission. I was pleased to receive a copy of a letter that Ian Robinson, the Chair of the Commission on Synchrotron Radiation, sent to all members of his commission requesting their suggestions for the composition and activities of the Synchrotron Commission for the next triennium. Because the Union makes no formal recommendations concerning how the nomination process should be conducted by individual commissions, I thought that other commission Chairs might benefit from reading Ian's letter.

I asked Ian if I could circulate a copy of his letter to the other Commission Chairs and he agreed. I realize that the needs and goals of the various commissions vary, nevertheless, I think Ian's letter might prove useful to all commission members. If you are currently a member of an IUCr Commission, I urge you to provide useful input to the Secretary and Chair of your commission along the lines recommended by Ian.

This issue of the Newsletter includes the second and final installment of the survey of crystallography in Russia gathered and organized by Leonid Aslanov. Material for an issue on crystallography in Australia and New Zealand, gathered by Jenny Martin has been received. We hope to have contributions from all 40 member countries, but no formal plan exists and no order of appearance has been established. As of now, we will publish the articles in the order received. If necessary, the Editor may solicit contributions (especially from Executive Committee members).

Bill Duax

duax@hwi.buffalo.edu

Dear Members of the IUCr SR commission

In the run-up to the IUCr general assembly in Florence at the end of next August, there will be some organizational activities for the SR commission. I have reviewed the 'General Statement of Principles' governing IUCr commissions (on the web) which outlines the procedure. We need to decide who should rotate off the roster and who should be added. This means we need to have an election.

The SR commission is fairly new and has not fully settled into a clear routine, but is now close to steady-state. We consist of 9 members with a maximum term of 9 years. For a 3-year cycle, it makes sense that we should aim to rotate 3 members every cycle. I have received a list of 15 nominees, originating from the national affiliate organizations of the IUCr, from which we should select 3. Two of these nominees are already on the commission, so I think we do not need to consider them again.

So please tell me your preferences from the following list of nominees. Send me an email reply with 3-5 (ranked) choices BY JANUARY 15, 2005. I will tabulate the votes at that time and announce the selection.

G. Aritoli	Italy	powder
G. Castro	Spain	surface
S.-L. Chang	ROChina	theory
M.A. Garcia-Granda	Spain	powder
R. Garrett	Australia	instrumentation
J.L. Hodeau	France	single crystal
K. Lawniczak-Jablonska	Poland	surface
K. Kjaer	Denmark	surface
C. Nave	UK	protein instrumentation
R. Paniago	Brazil	interfaces
S.M. Sharma	India	high pressure
M. Takata	Japan	powder
S. Wakatsuki	Japan	protein

To decide which of the class of 1999 should rotate off, let me first ask for volunteers. This is an extraordinary situation, given that we have only been in existence for 2 IUCr cycles. If this does not work, I will suggest who should stand down in order to try to strike a balance of subject areas and expertise. Less active members risk being contacted first! FYI our current roster is:

I. Robinson (C)	USA	1999	surface
LTJ Delbaere	Canada	1999	proteins
H. Graafsma	France	2002	instrumentation
SM Gruner	USA	1999	biophysics
H. Kitamura	Japan	1999	undulators
GN Kulipanov	Russia	2002	SR instrumentation
T. Matsushita	Japan	1999	x-ray optics
D. McMorrow	UK	2002	neutrons
J. Schneider	Germany	1999	single crystal

Last, but not least, we need to choose a NEW CHAIR. Though not a statutory requirement, it is customary (and I think it is a good idea) to rotate every cycle to maintain the vitality of the commission. So please nominate one of the current roster that you think would be desirable. Self-nominations are especially welcome. I will collate the nominees, and ask you to vote if necessary. The deadline for nominations is also JANUARY 15, 2005.

In other business, we have the opportunity to request money from IUCr to support our activities before MARCH 2005. In the past we have endorsed relevant conference activity and sponsored attendance at relevant workshops, for the purpose of reporting back to the IUCr.

Our commission tries to promote the access to SR facilities by crystallographers that do not otherwise have easy access. It tries to disseminate relevant technical information about advances in instrumentation and help establish standards between facilities where appropriate. We have a special interest in X-ray detectors, the development of which is often neglected by the facilities themselves. Thank you all for your efforts to help reach these goals.

Ian Robinson
SR Commission chair

LETTER TO THE EDITOR

The first Poster Exhibit?

Dear Judy



N.V. Belov presenting a poster at the VIII IUCr Congress at Stony Brook, NY (USA) in 1969.

In 1969, I was appointed Chairman of the Non-Commercial (Instrument) Exhibit of the VIII IUCr Congress. In addition to providing space for people to exhibit their instruments, I made announcements in several publications and contacted the editors of some journals to find names of people who had submitted articles on crystallographic equipment. These people were invited to submit reprints, preprints or posters of their instruments, if they could not bring them to the meeting. We ended up with what was probably the first Poster Exhibit at a scientific meeting. On the first day of the meeting, we were surprised to see Prof. N.V. Belov of the USSR Institute of Crystallography show up toting a large poster. This poster was a tribute to the Braggs and showed the structures of a number of silicate minerals that had been determined by Belov's group. We gave him a suitably prominent location to display the poster and I took a photograph showing him, in a relaxed mood, next to the poster.

Reuben Rudman

IUCR ACTIVITIES

International Tables Volume G available soon



Volume G, *Definition and exchange of crystallographic data*, is the latest addition to the *International Tables for Crystallography* series from the IUCr. It will be available in spring 2005 from Springer (hardback, ~550 pp., ISBN 1-4020-3138-6, see www.iucr.org/iucr-top/comm/commit/volg.html). Edited by S.R. Hall and B. McMahon, this volume describes the standard data exchange and archival file format (the Crystallographic Information File, or CIF) used throughout crystallography. It will be an essential guide and reference for programmers writing crystallographic software and for data managers handling crystal-structure information. It will provide in-depth information vital for small-molecule, inorganic and macromolecular crystallographers, mineralogists, chemists, materials scientists, solid-state physicists and others who wish to record or use the results of a single-crystal or powder diffraction experiment. The volume will provide the detailed data ontology necessary for data managers in other fields to design interoperable computer applications and databases. An accompanying CD-ROM will contain the dictionaries in machine-readable form and a collection of libraries and utility programs.

IUCR ACTIVITIES

Nominations for Executive Committee of the IUCr 2005-2008

The Executive Committee met in Budapest Hungary, August 24 and 25. At the meeting all nominations for officers and members of the Executive Committee proposed by the National Committees of member countries were considered. The Committee voted in favor of having only one candidate proposed for each office (President, Vice President and General Secretary and Treasurer) and it was agreed that six candidates should be presented for Executive Committee membership for the likely three six-year vacancies and one for the likely three-year vacancy.

The Executive Committee felt it important to rotate the Presidency among the three areas covered by the Regional Associates (ECA, ACA, and AsCA) and that, accordingly, it was appropriate to have a President from Asia - especially as the 2008 Congress will be held in Japan. The Executive Committee felt strongly that it was essential that the General Secretary and Treasurer should be located within easy traveling distance of the Chester Office since the expanding responsibilities of the General Secretary and Treasurer made frequent visits essential.

The high number of excellent nominations received for ordinary members of the Executive Committee was much appreciated by the Executive Committee. Selection of a shortlist was necessarily difficult and there were lively discussions. However, it was clear that not all names could go forward and as it is the responsibility of the Executive Committee to make a selection it was decided that six nominations for the three likely six-year vacancies and one for the likely three-year vacancy would be put forward.

The Executive Committee welcomes the increased participation of the National Committees that the more open procedure followed this time has produced. In fact, about twice as many member countries have made suggestions than has typically been the case in the past. According to By-laws 8.2 and 8.4 you may still make your own nominations in Florence, should you so wish.

Concerning the voting procedure, only two responses were received to the Circular concerning the possibility of adopting a single-transferable-voting procedure and so it is clear that there is no mandate for changing the present simple-majority method.

IUCr Executive Committee Nominations

- President: Y. Ohashi (Japan)
- General Secretary and Treasurer: S. Lidin (Sweden)
- Executive Committee Membership:
- Six-year terms: P. Coleman (Australia), G.R. Desiraju (India), C. Gilmore (UK), M. Kovalchuk (Russia), C. Lecomte (France), M. Perez-Mato (Spain);
- Three-year term: I. Torriani (Brazil).

A nominee for Vice President agreed to by the Executive Committee in Budapest did not accept the nomination. The Executive Committee will decide upon a new nominee for Vice President at a later date.

IUCR ACTIVITIES

IUCr Executive Committee report of meeting in Budapest

The IUCr Executive Committee met in Budapest, Hungary, August 25-26 2004. Principal topics discussed were: the launch of a new electronic-only journal in 2005; open access; development of CIF handling software; putting *International Tables* online; the Florence General Assembly and Congress; and nominations for the Executive Committee and Commissions and voting procedures at General Assemblies.

Excerpts from the Minutes of the Executive Committee meeting

Review of journals. It was noted that the impact factors of IUCr journals for 2003 had all increased.

The IUCr had been successful in its application for a grant from the UK Joint Information Systems Committee (JISC) as part of the open access initiative; in return for the grant, the IUCr's open access charge for submissions from UK institutions had been waived for 2004.

It was noted that in early 2004 the Commons Science and Technology Committee of the United Kingdom Parliament had undertaken an inquiry into Scientific Publications. Following a Press Release and invitation to submit evidence, the IUCr had written a report on its publishing activities and policies, which was submitted as evidence to the inquiry in February 2004.

The Editor-in-Chief reported on progress with the electronic-only journal *Acta Crystallographica Section F: Structural Biology and Crystallization Communications* that would be launched in January 2005.

It was noted that grants had been made available from the Journal Grants Fund to institutions in thirteen countries. The IUCr journals were also included in Blackwell's Synergy and were thereby available as part of the Programme for the Enhancement of Research Information (PERI), through the International Network for the Availability of Scientific Publications (INASP), whereby qualifying developing countries received access at reduced cost.

STAR/CIF. Good progress was reported for two projects: (a) to develop procedures and templates to ensure that mmCIF data files deposited with the Protein Data Bank contained all the information necessary for publication in IUCr journals; and (b) for the development of CIF handling software.

Internet services. It was noted that the Crystallography Journals Online service received about 108,000 requests per day from about 20,000 unique hosts.

Archiving arrangements. Steps being taken to ensure the permanent long-term archiving of all IUCr publications were discussed and IUCr cooperation with a Southamptonbased initiative to allow institutional self-archiving of crystal structures by chemists was welcomed.

International Tables for Crystallography. Progress with new volumes and revisions of existing volumes was reviewed. A proposal to make the complete series available online was approved. Additional interactive features would be developed. It was agreed that the charging model for the online version should be subscription based to the complete package.

Progress with a revision of the contract with Springer (formerly Kluwer) for publishing print and online versions of *International Tables* was noted.

World Directory of Crystallographers. The Executive Committee agreed that the database should be streamlined by including fewer entries for fields of interest and discussed ways of speeding up the updating process by increasing the involvement of the National and Regional Editors.

IUCr Newsletter. The Executive Committee was particularly pleased with the issues covering crystallography in Latin America, Italy and Russia and noted that issues for Germany, Australia and Japan were pending.

Ewald Prize. Prof. Michiyoshi Tanaka had agreed to chair the Selection Committee for the Seventh Ewald Prize, to be presented in Florence in 2005.

Regional Associates. The Executive Committee received reports from the representatives and were pleased to note a Latin American initiative of the American Crystallographic Association.

Relations with other Scientific Unions. The Executive Committee agreed to support the application of the International Commission for Acoustics (presently an Associated Commission of the International Union of Pure and Applied Chemistry) to become a member of ICSU.

Florence Programme. The Executive Committee discussed the programme and approved guidelines for future Congresses that had been recommended by the Florence Programme Committee.

Nominations for members of the Executive Committee and Commissions/voting procedure.

The Executive Committee discussed its nominations for the new Executive Committee membership for the next triennium. [The President has subsequently written to the National Committees for Crystallography detailing the recommendations and explaining the reasons for making them].

Only two responses had been received to the President's circular concerning the possible adoption of a single-transferable-voting procedure at General Assemblies. The Executive Committee therefore decided that there was no mandate to change the existing procedure.

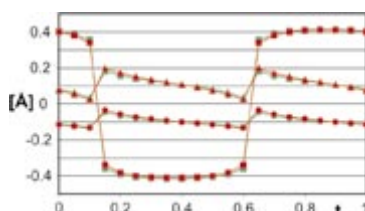
Audited accounts for 2003. The Executive Committee approved the audited accounts for 2003.

Status of membership. Argentina's membership had been reinstated by the Executive Committee following commitments by Spain and the USA to pay the outstanding arrears. Ukraine (also suspended) had indicated that it was now in a position to pay annual subscriptions and the Executive Committee agreed to continue to explore possible ways of covering the arrears.

The summer 2005 meeting of the Executive Committee will be held in Florence, prior to the Congress.

Ab initio determination of incommensurately modulated structures by charge flipping in superspace

Acta Cryst. (2004). A60, 604–610 [doi:10.1107/S0108767304022433]



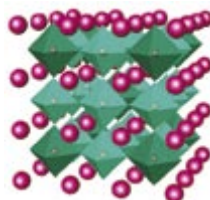
The x, y, and z components (circles, squares, and triangles, respectively) of the modulation function of a Br atom as obtained from the charge flipping algorithm (red symbols) and from the final structure model (green symbols).

The *ab initio* structure solution algorithm dubbed 'charge flipping' [Oszlányi & Sütö (2004). *Acta Cryst. A* **60**, 134–141] has been generalized towards (3+d)-dimensional superspace. This generalized algorithm allows for the solution of incommensurately modulated structures directly in superspace. This eliminates the necessity of solving the basic structure of the modulated compound, which can be an extremely difficult task in the case of complex modulations. The algorithm succeeded in solving seven of eight tested incommensurately modulated structures. The method has been demonstrated on the modulated structure of tetraphenylphosphonium hexabromotellurate(IV) bis{dibromoselenate(I)}.

Lukáš Palatinus

Octahedral tilting in cation-ordered perovskites - a group-theoretical approach

***Acta Cryst.* (2004). B60, 674–684 [doi:10.1107/S0108768104019901]**



Structure of the cation-ordered perovskite $\text{Ba}_4\text{LiSb}_3\text{O}_{12}$.

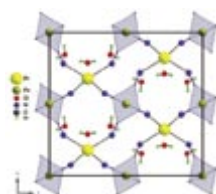
The perovskite (ABX_3) structure can be visualised as a three-dimensional network of regular corner-linked BX_6 octahedra, the A cations being located in the cavities between them. Much of the structural variability arises from different patterns of BX_6 octahedral tilting. This paper deals with perovskites in which the octahedra contain two types of cation, in ordered arrangements, at stoichiometries $\text{A}_3\text{BB}'_2\text{X}_9$ and $\text{A}_4\text{BB}'_3\text{X}_{12}$. Group-theoretical methods are used to derive details of the structures obtained when this cation ordering occurs in combination with octahedral tilting.

C.J. Howard and H.T. Stokes

IUCR JOURNALS NEWS

Pseudo-merohedrally twinned praseodymium hexacyanoferrate(III) tetrahydrate

***Acta Cryst.* (2004). C60, i104-i106 [doi:10.1107/S010827010402092X]**



The structure of $\text{Pr}[\text{Fe}(\text{CN})_6] \cdot 4\text{H}_2\text{O}$ viewed along the *a* axis. FeC_6 octahedra are shown in blue color. The article describes the structure of the title compound, diaqua-hexa- μ -cyano-ferrate(III)praseodymium(III) dihydrate, $\text{Pr}[\text{Fe}(\text{CN})_6] \cdot 4\text{H}_2\text{O}$ or $[\text{PrFe}(\text{CN})_6(\text{H}_2\text{O})_2] \cdot 2\text{H}_2\text{O}$. The crystals, although orthorhombic, space group *Cmcm*, are pseudo-merohedrally twinned with three components, simulating thus the hexagonal system. The Pr atom is coordinated by eight atoms, viz. six N and two symmetry-related water O atoms. The Pr polyhedron is linked to an FeC_6 octahedron through N atoms, forming an infinite array. The second (symmetry independent) water molecule is not included in coordination and is weakly hydrogen bonded to N atoms.

V. Langer, L. Smrcok and Y. Masuda

IUCR JOURNALS NEWS

Coot: model-building tools for molecular graphics

Coot: model-building tools for molecular graphics

***Acta Cryst.* (2004). D60, 2126-2132 [doi:10.1107/S0907444904019158]**

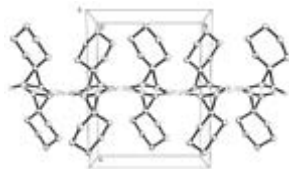


Coot is a new CCP4-friendly molecular graphics program for crystallographic model-building. It is built on a range of free software libraries, including Eugene Krissinel's 'mmdb' and Kevin Cowtan's 'Clipper'. An intuitive, modern user interface was an important design consideration. *Coot* currently provides functionality useful for both experienced and novice crystallographers, including rigid body refinement, interactive real space refinement using modified conjugate gradient (BFGS) minimization, 'continuous crystal' electron density and skeleton, interactive Ramachandran plots, NCS density comparison and other model-building and validation tools.

P. Emsley and K. Cowtan

3,3-Pentamethylenediaziridine

***Acta Cryst.* (2004). E60, o1921-o1922 [doi:10.1107/S1600536804023888]**



A view of the unit cell, showing the disordered hydrogen-bond network along the *b* axis. Hydrogen bonds are shown as dashed lines. H and C atoms are shown as small spheres of arbitrary radii. H atoms attached to C atoms have been omitted.

N,N-unsubstituted diaziridines are very useful moieties, being precursors by oxidation to diazirines, more stable structural isomers of diazo compounds, that can be used as carbene precursors. Surprisingly, only crystal structures of either *N*-substituted or *N*-complexed diaziridines compounds have been reported so far. The title compound is the first free *N,N*-unsubstituted 3,3-dialkyldiaziridine to be structurally characterized. The analysis was only successfully achieved using the sealed capillary method. The crystal structure indicates the presence of intermolecular N-H...N hydrogen bonds, resulting in a three-dimensional network.

A. B. Charette, C. Legault and F. Bélanger-Gariépy

Structure of *Mesorhizobium loti* arylamine N-acetyltransferase 1

Structure of *Mesorhizobium loti* arylamine N-acetyltransferase 1

Acta Cryst. (2005). F61, 14–16 [doi:10.1107/S1744309104030659]



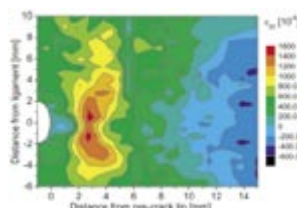
Secondary structure representation of *M. loti* NAT1 (mauve) superimposed with other published NAT structures.

Arylamine N-acetyltransferases (NATs), first identified through their ability to inactivate the anti-tubercular therapy isoniazid, have now been observed in a broad range of prokaryotic and eukaryotic species. This article presents the 1.8 Å structure of NAT1 from *Mesorhizobium loti*, a nitrogen-fixing bacterium that lives in symbiosis with leguminous plants. Despite low sequence identity, the NAT1 structure is remarkably well conserved with that of other NATs. This structure adds to the growing number of published NAT structures and will complement current work at identifying bacterial NAT antagonists.

S. Holton, J. Dairou, J. Sandy, F. Rodrigues-Lima, J.-M. Dupret, M. Noble and E. Sim

High-resolution strain mapping in bulk samples using full-profile analysis of energy-dispersive synchrotron X-ray diffraction data

***J. Appl. Cryst.* (2004). 37, 883–889 [doi:10.1107/S0021889804023349]**



The elastic strain measured in the vicinity of a crack tip in a 25 mm thick compact-tension steel specimen.

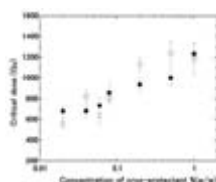
This article demonstrates the feasibility of making strain measurements with both high spatial and strain resolution in bulk (engineering) components. The experiments were undertaken using energy dispersive X-ray diffraction with synchrotron X-rays between 100 and 300 keV on beam line ID15A at the ESRF. The data analysis was improved by using a multiple-peak Pawley-type refinement on the recorded spectra with an asymmetric peak profile. An important area of application is the characterisation of stress fields around crack tips in bulk components.

A. Steuwer, J. R. Santisteban, M. Turski, P. J. Withers and T. Buslaps

IUCR JOURNALS NEWS

Radiation damage to a protein solution, detected by synchrotron X-ray small-angle scattering: dose-related considerations and suppression by cryoprotectants

***J. Synchrotron Rad.* (2004). 11, 462-468 [doi:10.1107/S0909049504019272]**



Dependence of critical dose, a threshold of radiation insensitivity, on the concentrations of glycerol and ethylene glycol. Open and solid circles correspond to the plots for glycerol (148 Gy/s) and ethylene glycol (148 Gy/s), respectively. Beyond 1.0% the aggregation is negligible and critical dose could not be determined.

In all synchrotron experiments on biomolecules one has to face radiation damage. In small-angle X-ray scattering experiments at high-brilliant synchrotron sources, protein aggregation results from radiation damage. This paper first describes the quantitative evaluation and understanding of the dose-related phenomena of radiation-induced aggregation that was evident over 400 Gy in the lysozyme solution. Furthermore it was shown that adding small amounts of cryoprotectants in the hundred millimolar concentration range effectively reduced the radiation damage.

S. Kuwamoto, S. Akiyama and T. Fujisawa

SPECIAL REPORT

Crystallography in Russia

Part 2

Kirensky Institute of Physics



Kirensky Institute of Physics (main building) – Siberian winter.

Investigations of crystal physics and development of their applications are the main activities of the Kirensky Institute of Physics (Krasnoyarsk). Researchers at the institute study piezoelectrics, ferroelectrics, ferroelastics, and magnetic dielectric crystals, and crystals containing rare earth ions for optical applications.



Garnet crystals – as grown by group flux-melt method.

Methods of group flux melt growth developed here provide a means to obtain high quality bulk crystals of copper-germanium oxide, lead gallium germanate, and copper metaborate under controlled conditions. Neodymium-activated crystals of gadolinium-gallium garnets synthesized by this method appear to be a single-center medium with more than 10 at.% of Nd^{3+} .

Searching for new crystals demands systematic analysis of known structures to provide a foundation for a reliable prognosis. Such analysis has been performed for oxide- and halogen-based perovskites, antiperovskites, elpasolites, anion- and cation-deficient and layered perovskite-like structures, including high temperature superconductors. Group-theory and crystallographic analyses of phase transitions in these structural families have been made as well.

Structures of synthesized crystals are investigated using powder and single crystal X-ray analysis. Neutron scattering data are used in collaboration with the Joint Institute of Nuclear Research (Dubna, Russia), the Hahn-Meitner Institute in Germany and the Laboratoire Leon Brillouin at the Saclay Neutron Center in France.



A. D. Vasiliev at single crystal X-ray diffractometer – deciphering a structure of a new crystal.

Fundamental goals are to correlate physical properties of materials with their crystal structures and to determine the impact of external forces on crystal parameters and phase transitions. Physical properties studied include electric and magnetic parameters, acoustic and optics measurements, heat capacity and thermal expansion. With Krasnoyarsk State University, nonlinear electro mechanic properties higher order elastic coefficients, electrostriction, and nonlinear piezoeffects are studied. A study of β - K_2SO_4 crystals revealed a complex sequence of phase transitions that include disordered and incommensurate phases. Radio spectroscopic and optical second harmonics investigations of incommensurate phases are conducted. The concept of solution density measurements by NMR analysis was formulated here and Raman scattering selection rules for these phases have been developed and experimentally tested. Recent investigations have focused on complex phase transitions of halogenides and oxyhalogenides with perovskite-like structures including structurally disordered ferroelectric-relaxors.

A theory of structural phase transitions is being developed in parallel with experimental investigations. Phase transition sequences with interacting order parameters are developed based on group theory and thermodynamics and applied to families of crystals. Attention is focused on model descriptions of these phase transition sequences. *Ab initio* approaches are being developed that describe the stability, lattice dynamics and physical properties for complex crystal structures like layered perovskites and elpasolites.

In 1976 the Krasnoyarsk School of Crystal Physics began a series of Soviet (now - Russian) - Japanese symposia on the physics of ferroelectrics that continues to the present day.

Contact: K.S. Aleksandrov kaleks@iph.krasn.ru

Material studies in Petrozavodsk

At the X-ray laboratory of the Petrozavodsk State University, amorphous oxide films, amorphous and crystalline powders and bulk materials are studied. X-ray diffraction patterns from symmetric and asymmetric reflection and transmission geometry are obtained on a DRON-4 diffractometer using monochromatized radiation of various wavelengths.

The short-range order characteristics of amorphous, amorphous-crystalline and small dispersion materials are defined using the Finbak-Warren approach: the $D(r)$ curves are presented as a sum of pair functions. We have identified the short-range order characteristics of amorphous oxide films of aluminum, silicon, tantalum, niobium, tungsten, yttrium, and vanadium, produced under different conditions of anode oxidation; WO_3 films, thermally evaporated in vacuum; silicon films, produced by monosilane pyrolysis at various temperatures; thermal silicon dioxide films; and manganese dioxide produced by pyrolysis of manganese dihydrate. Computer simulations of the structures of amorphous materials are calculated using the methods of continuous static relaxation, molecular dynamics, and non-ordering networks.

Octahedrally and tetrahedrally coordinated cations with distorted fcc packing of oxygen atoms in the system 'aluminum-vacant cationic positions' were considered in terms of short-range order coefficients. The cationic subsystem of amorphous aluminum oxides is characterized by a short-range order qualitatively analogous to the arrangement of aluminum atoms in the boehmite and pseudoboehmite

modifications of the $\gamma\text{-Al}_2\text{O}_3$ phase. The correlation length in the system 'aluminum-vacant cationic positions' is not less than 5 Å. It was shown that the short-range order in amorphous oxide films of Ta_2O_5 and Nb_2O_5 can be regarded as similar to the atom positions in the crystal modifications $\beta\text{-Ta}_2\text{O}_5$ and $\gamma\text{-Nb}_2\text{O}_5$. The X-ray study and molecular dynamic computer simulation of amorphous anodic tungsten oxide showed that the arrangement of W and O atoms in the coordination spheres corresponds to the characteristics of the crystalline $\text{WO}_3 \cdot (1/3)_2$ modification. WO_3 films, thermally vaporized-on in vacuum, have a quasi-amorphous structure characterized by the presence of crystallites shaped like orthorhombic phase parallelepipeds with dimensions 15 x 8 x 20 Å.

The short-range order in amorphous oxide films and powder of Y_2O_3 depends on anode oxidation conditions. Moreover, the first coordination number changes from 5 in colored oxides to 7 in black and powdered oxides. The short-range order is described in terms of models of disordered networks of octahedra, pyramids, and structural units, consisting of seven oxygen atoms. In amorphous fulleride C_{60} short-range order corresponds to the lonsdeillite crystalline phase. The main publications describing this work are in *Crystallography Reports* and *Acta Crystallographica*.

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Crystallography in Novosibirsk



Almost 1.5 million people live in Novosibirsk, more than 30 000 are involved in the work of the Novosibirsk Scientific Center. Novosibirsk State University is surrounded by about 40 research institutes of the Russian Academy of Science. Integration of the Novosibirsk State University with the Russian

Academy of Sciences was always exceptionally high for Russia. Crystallography is widely applied in many institutes, ranging from traditional applications in chemistry, physics, biology, and ending with archeology. Novosibirsk is a huge high technology industrial center with vast potential for studies with broad applications.



Summer schools attract participants of all ages.



H. Ahsbals from Philipps University (Marburg/Lahn, Germany) and A. Achkasov from Novosibirsk State University discuss some technical details of a high-pressure experiment.



J. Lipkowski of Poland gives a lecture on crystallography and physical chemistry of inclusion compounds.



A single crystal diffractometer STADI-4 (STOE, Darmstadt) was specially adapted on the request of the group for high-pressure data collection.



GADDS D8 diffractometer with a 2D-detector (Bruker, Karlsruhe) enables a rapid data collection from various samples – ranging from very small amounts of powder samples to pieces of ceramics and rocks.



Thermal analysis and calorimetry – TG, DSC, TMA - (NETZSCH, Selb) are important techniques complementary to X-ray diffraction.

Solid state chemistry and mechanochemistry

The group of Elena Boldyreva divides its activities between the Institute of Solid State Chemistry and Mechanochemistry (Siberian RAS, Novosibirsk) and the Novosibirsk State University.

Research

Boldyreva is a physical chemist who specializes in studies involving solid-state kinetics, inorganic solids, coordination compounds, and organic molecular solids with a special emphasis on pharmaceutical and biomimetic systems. Fields of research include solid-state reactivity, crystal engineering, polymorphism, crystallographic computing, database analysis, high-pressure and low-temperature crystallography using single-crystal and powder X-ray structure analysis, systems studied include Co(III)-ammine complexes and polymorphs of paracetamol, glycine, serine, hexafluorosilicates, benzoquinone, and sodium oxalate. The effect of high pressure on a solid has been systematically compared with that of cooling. Diffraction studies are complemented by IR- and Raman spectroscopy, thermal analysis and calorimetry, and optical microscopy. The group collaborates with other research teams studying mechanochemical synthesis and mechanochemical modification of pharmaceuticals. They also work with colleagues from the Institute of Mineralogy, the Institute of Catalysis, the Institute of Semiconductor Physics, the Institute of Genetics, and the Institute of Archeology in Novosibirsk. With the latter, they have characterized samples of ancient Siberian ceramics. The group is actively involved in research in a multidisciplinary Research and Education Center REC-008 'Molecular Design and Ecologically Safe Technologies', and in the Center of Joint Exploitation of Equipment 'Integration'.

Education

The group is active in teaching solid state chemistry, crystallography, structural analysis and supramolecular chemistry at Novosibirsk State University. All chemistry students at the university attend the courses. Former students work at various research institutes of the Russian Academy of Sciences, in industry and abroad. In recent years the group has included PhD students and post-docs from Barnaul, Tomsk, Kemerovo, and Petrozavodsk. The teaching activities of the group were described in articles in the *Journal of Chemical Education* [*J. Chem. Educ.*, 1993, **70**(7), 551-556 and 2000, **77**(2), 222-226]. E. Boldyreva, a lecturer at international schools, has translated E. Wood's '[Crystals](#)' and 'Supramolecular chemistry' by J.-M. Lehn into Russian. In 2000 the group initiated a series of Novosibirsk summer and winter schools on 'Hot Topics of Chemistry, Biology, and Physics', which are attended by schoolchildren and teachers from all over Siberia. The group is an active member of the SigmaXi International Research Society and participates in the 'Distinguished Lecturers' program.

International collaborations

Boldyreva has been a visiting research scientist in Germany, France, Italy, and Great Britain. Many of her former hosts and 'western colleagues' have visited Novosibirsk, to lecture, exchange ideas, and test equipment. The group has traditional collaborations in Europe, good contacts in the USA, and has recently signed an Agreement for Cooperation with Wits-University (Johannesburg, South Africa).

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***in vivo* X-ray diffraction analysis of cystic calculus**

The potential to use synchrotron radiation for medical diagnosis via *in vitro* X-ray diffraction is being explored at the Synchrotron Center of the Institute of Nuclear Physics in Novosibirsk.

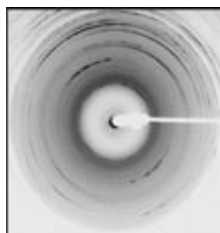


Figure 1.

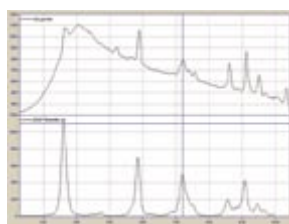


Figure 2.

Urologists remove cystic calculi via lithoclasty without an abdominal operation, and fragments of calculi are excreted through the urinary tract. Analysis of the collected biominerals permits determination of a specific form of urolithiasis, which can guide therapy and prevent recurrence. These methods analyze cystic calculi only after their removal. It would be helpful to determine the composition of a crystal calculi without surgery.

We have attempted to model *in vivo* X-ray diffraction analysis of a cystic calculus. A surgically obtained calculus (5 x 3 x 3 mm) was placed into pig tissue with a high fat content. The investigation was carried out with quanta in the range 30–34 keV at an X-ray diffraction station^[1] installed at the 4th synchrotron radiation beamline of the VEPP-3 storage ring in the Synchrotron Center.

First, a diffraction pattern of the cystic calculus was obtained. Then, the calculus was placed into the model fatty tissue and a new diffraction pattern was recorded. An example of a diffraction pattern from a cystic calculus in fatty tissue is shown in Fig. 1. Fig. 2 illustrates a comparison between the calculus embedding in fatty tissue (top) and the pattern for a sample of monohydrate calcium oxalate (bottom).

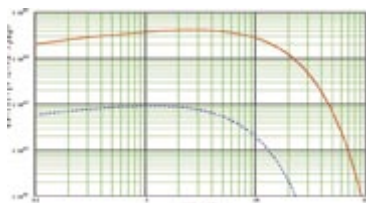
Such an investigation does not require a detailed analysis of the diffraction pattern, because most of the calculi are of three main types (oxalates, phosphates and urates). The diffractograms of these types differ sharply and identification of a calculus can be made with the signal/noise ratio as low as ~10. This method of recording the diffractograms does not require precise positioning of the sample.

As shown in Fig. 2, the type of the calculus can be determined from such data. These experiments demonstrate the feasibility of collecting the diffraction pattern of a cystic calculus embedded in fatty tissue. Although current radiation doses required for *in vivo* analysis exceed permissible levels, the development of more sensitive detectors could make the technique feasible.

[1] Ancharov A.I. *et al.*, 'New station at the 4th beamline of the VEPP-3 storage ring' *Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment*, **470** (2001), 1–2, P.80–83

Contact: B. P. Tolochko (b.p.tolochko@inp.nsk.su)

Superconducting wigglers and shifters



Photon flux from the usual 1.5 Tesla bending magnet and 3.5 Tesla 49-pole superconducting wiggler for ELETTRA storage ring (2 GeV and 200 mA).

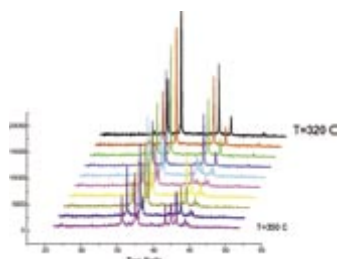


7.5 Tesla 3 pole shifter installed at the BESSY storage ring.

Budker INP at the Institute of Nuclear Physics, Novosibirsk has significant experience in the development and construction of superconducting insertion devices (ID). The first superconducting 20 pole wiggler was assembled in 1979 for the VEPP-3 storage ring. During the last 10 years, new types of superconducting wigglers and shifters for a number of storage rings around the world have been constructed. Strong field wigglers or wavelength shifters are installed in the straight section of the storage ring to enhance the performance of the machine for short wavelength users and to provide new possibilities for SR experiments. Reasons to install wigglers or shifters on a storage ring include: (1) to shift the spectrum to the hard X-ray region by using the higher magnetic field of the wiggler (shifter); (2) to increase the photon flux due to many poles (multipole wiggler); (3) to obtain new features of radiation such as polarization; (4) to obtain flexibility for experiments due to the possibility of changing the wiggler field during the experiment; (5) to decrease or increase the emission of the storage ring; (6) to decrease the polarization time of the electron (positron) beam; and (7) to create a slow positron source of high brightness. Generally a wiggler or shifter consists of a magnetic system, a cryogenic system, a vacuum system, a control system and power supplies.

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Inorganic crystallography



OD-3 measurement: The phase transition in MgZnAl.



OD-3 measurement: behavior of mixture $0.7\text{NiO} + 0.3\text{WO}_3$ at 720°C .

The Crystal Chemistry Laboratory, established in the Siberia Nikolaev Institute of Inorganic Chemistry, SB RAS / Crystal Chemistry Laboratory in 1958 by Prof. G. Bokii (1909-2001), is the oldest and largest crystallography center in Siberia. The laboratory staff consists of 16 researchers, several engineers and students involved in X-ray structural and crystallochemical studies. The laboratory is equipped with powder and single crystal X-ray diffractometers. Research at the laboratory includes studies of inorganic and coordination compounds (S. Borisov, N. Podbereskaya, and S. Solodovnikov); transition metal coordination compounds including molecular magnetic materials and volatile complexes (N. Pervukhina, I. Baidina, S. Gromilov, L. Glinskaya, V. Alekseev, and T. Polyanskaya); complex oxides including molybdates, tungstates, hypo-phosphites, chlorites, natural and modified zeolites (R. Klevtsova, S. Solodovnikov, D. Naumov, and V. Bakakin); transition metal and boron cluster compounds (A. Virovets; S. Solodovnikov, T. Polyanskaya, and D. Naumov); mercury-containing minerals and their analogs (S. Magarill and N. Pervukhina); and supramolecular compounds containing transition metal complexes and cucurbit[*n*]urils (A. Virovets and N. Pervukhina).

High-quality powder patterns of bulk materials and films prepared in the institute (S. Gromilov) have been provided for the ICDD Grant-in-Aid Program (V. Lisoivan). Software that includes searches for cation sublattices and cavities, comparison and visualization of structures has been written (D. Naumov).

The laboratory produces about 100 crystal structures and 50 publications per year. Interesting recent results include:

- The structure-forming role of cations in some classes of inorganic compounds that results in regular close-packed cationic arrays;
- The structure-forming role of Hg-O, Hg-S, and Hg-Hg covalent bonds that results in the formation of rigid mixed mercury-anion clusters, ribbons, layers and 3D frameworks in inorganic mercury-containing compounds;
- Algorithms for generating 3D tetrahedral clathrate frameworks using the duality of polyhedral clathrate hydrates and intermetallic structures.

The laboratory takes an active role in the education process of students in the Chemistry Department of Novosibirsk State University and cooperates with colleagues from Russia, Germany, UK, and Spain.

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Helically polarized radiation sources development in Budker INP, Novosibirsk

In recent years Budker INP has designed and manufactured several insertion devices for the generation of helically polarized radiation.



General view of one of two halves of elliptical electromagnetic undulator for advanced SR source SLS (Switzerland).

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Advanced X-ray detectors, INP



OD-3 electronics super-module with shapers, FLASH ADC's, Processor Unit and RAM.

A fast, parallax-free, one coordinate detector OD-3 has been designed for angular measurements in diffraction experiments on a synchrotron X-ray beam with a photon energy around 10 keV within an angular aperture of 30 degrees. The OD-3 detector consists of a proportional chamber, CAMAC crate with electronics and a host IBM PC compatible computer. The proportional chamber of the OD-3 has a drift volume where the coordinate of quantum along the anode wire is detected by measuring the charge induced on the strips of the lower cathode plane. A peculiar shape of cathode strips 'focused' on the object under study provides parallax elimination. The design of the detector allows the minimum focus distance to be 350 mm with conventional ones of 350 mm and 1.5 meters. Events are stored in the Incremental RAM (256K x 32) in the Processor Unit and can be read into the computer for processing, visualization, and long-term storage.

Main parameters of the OD-3 detector	
inlet beryllium window	200 mm x 10mm x 0.2mm
conventional operational gas mixture	Ar/10% CO ₂
excessive pressure in gas chamber	0.2 atm
photon energy range	5-15 keV
detection efficiency (for 8 keV photons)	30 %
maximum detection angle	± 15 degrees at the focal distance 350 mm, ± 3.5 degrees at the focal distance 1.5 meter
scale	3328 channels
channel width	60 μm (0.01 degree at f = 350 mm)
maximum number of frames	1024 frames
frame time range	1 μs / 1024 seconds
space resolution (E _y = 8 keV, STP), FWHM	150 μm (0.025 degree at f = 350 mm)
linearity	0.15 %
differential nonuniformity, R.M.S.	< 2.5 %
counting rate (at 50 % non-efficiency)	10 MHz/detector

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The structure of quantum dots by XAFS

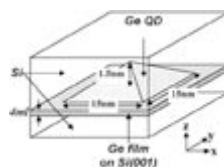


Fig. 1. Circuit of Ge nanocluster (QD) on Si(001).

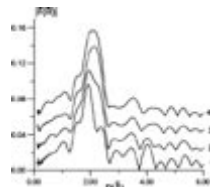


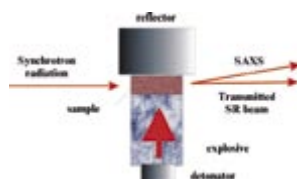
Fig. 2. Fourier transform magnitude of $k^3\chi(k)$ GeK EXAFS data at $E||\text{Si}(001)$: for pseudomorphous 4-monolayer (4ML) 2D- films on Si(001) – curve 1, for Ge nanoclusters on pseudomorphous 4-monolayer films on Si(001) with effective thickness equal to 6ML – curve 2, equal to 8ML – curve 3, equal to 10ML – curve 4.

Surface sensitive EXAFS- (Extended X-ray Absorption Fine Structure) and XANFS- (X-ray Absorption Near Edge Structure) techniques are used at the Nikolaev Institute of Inorganic Chemistry, Institute of Semiconductors Physics and Budker INP SB RAS, Novosibirsk to determine the spatial and electronic properties of heterogeneous surfaces^[1,2]. Uniform germanium nanoislands deposited on Si(001) and Si(111) substrates via molecular beam epitaxy (MBE) (Fig.1) exhibit quantum dot (QD) properties. The influences of effective thickness of the Ge film, Ge nanocluster sizes and deposition temperature on the QD microstructure parameters were determined by EXAFS and XANFS techniques. The effective thickness varied from two to ten monolayers for the films studied. Two-dimensional pseudomorphous Ge films have been grown up to a critical thickness of four monolayers on Si(001). As a result of continuing deposition, pyramid-like Ge islands were grown in the Stranski-Krastanov mode. Local microstructure parameters are linked to nanostructure morphology and models are proposed. The Ge islands that form during growth are characterized by interatomic Ge-Ge distances of 2.41 Å (0.04 Å less than in bulk Ge). Pure Ge nanoclusters are covered by a 1-2 monolayer film with an admixture on the average of 50 % Si-impurity due to interface diffusion from blocking Si layers at 500°C. The monotonic size of germanium nanoclusters were determined as a function of film thickness and the influence of temperature change was measured. That microstructural parameters of Ge/Si heterosystems are largely influenced by elastic deformation at the boundaries due to a mismatch of lattice parameters of the nanocluster and substrate was detected by direct measurement showing that EXAFS spectroscopy is a useful tool for the study of materials containing nanostructures.

[1] Erenburg, S.B., Bausk, N.V., Stepina, N.P., Nikiforov, A.I., Nenashev, A.V., and Mazalov, L.N., *Nucl. Instr. & Meth. Phys. Res. A.* (2001) **470**/1-2, 283-289. [2] Erenburg, S., Bausk, N., Mazalov, L., Nikiforov, A., and Yakimov, A., *J. Synchrotron Rad.* (2003), **10**, 380-383.

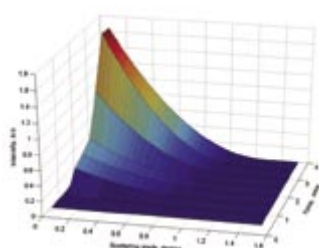
Contact: B. P. Tolochko, B.P.Tolochko@inp.nsk.su

Nanoparticles nucleation under extreme conditions



The experimental scheme for *in situ* SAXS investigation of shock wave impact on various materials. Production of materials with new properties can be achieved via synthesis under high temperatures, high pressures and nonequilibrium conditions. To collect data under these conditions, equipment with

nanosecond time resolution, high sensitivity to phases at low concentrations and high penetrating depth (millimeters) should be used. Synchrotron radiation has the required characteristics.



The time dependence of small angle X-ray scattering during diamond nanoparticle nucleation and growth under shock wave compression. Time $t = 1 \mu\text{s}$ corresponds to room temperature and pressure, time $t = 1.5\text{--}3.5 \mu\text{s}$ – to high temperature and pressure.

For generating high temperatures and pressures we use explosions and shock waves. During the explosions in our experiments pressures can reach 2 Mbar with temperatures up to 8000° C. We have developed an 'extreme conditions' synchrotron radiation beamline which allows us to investigate the dynamics of phase transformations during explosion and shock wave impact. In particular we have investigated nucleation and growth of diamond and metallic particles by small angle X-ray scattering (SAXS). We have observed nuclei with sizes near 30 (at $t=1.5 \mu\text{s}$) and dynamic of growth up to 70 during $2 \mu\text{s}$. The influence of different conditions on kinetics of nanoparticles growth have also been investigated.

[1] B.P. Tolochko, N.Z. Lyakhov *et al.* *NIMA*, v. **467-468**, Part 2, 2001, p. 990.

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An X-ray detector for imaging explosions

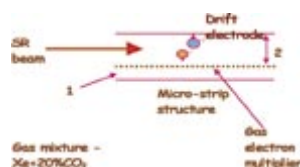


Figure 1. Design of the detector.

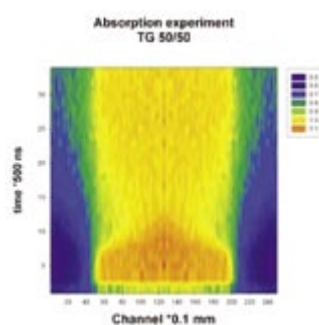


Figure 2. Projective image of an explosion. Vertical scale is time in units of 500ns. Horizontal scale is position in 0.1mm channels.

Very short pulses of synchrotron light irradiated by individual electron bunches allow imaging of the development of a detonation wave and the changes of electron density within a volume of exploding materials. Such experiments require an exceptional set of parameters from the detector. In order to view independent images from different electron bunches, time resolution of the detector has to be less than bunch crossing time. A new detector for imaging fast dynamic processes and explosions with SR beam (DIMEX) was constructed in Budker Institute of Nuclear Physics. Details of the instruments construction are reported in the references below. In order to investigate the structure and velocity of a detonation

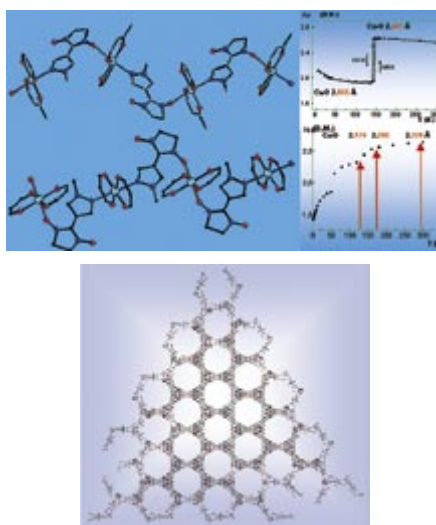
wave in an explosion, as well as density distribution inside and around the exploding sample, the projective absorption experiments were conducted using DIMEX. The collimated line-shaped beam passed through the sample and the distribution of X-ray flux was measured. Our beam was ~12 mm wide and 1mm high. The samples were 12.5 mm in diameter and 100 mm cylinders made of a mixture of hexogen and TNT. The sample was positioned with its axis either parallel or perpendicular to the beam plane. The start of the measurement sequence could be synchronized with the detonation to within ~0.5s. A sequence of small angle scattering (SAXS) images give information about the concentration of particles with different dimensions in an object. The result of a series of projective absorption experiments is shown in Fig. 2. In order to improve the precision, the results of 10 measurements were summed with proper synchronization. The horizontal axis of the figure is the position perpendicular to the axis of the sample. Time axis is in vertical in units of 500 ns. The figure shows the detonation wave and the reaction zone with very high density just after the detonation front. The technique of very fast imaging opens opportunities in an area of fast dynamic SAXS and WAXS (wide-angle X-ray scattering) studies of an objects under the influence of either different external factors like temperature, pressure, light etc., or internal meta-stable or excited states.

[1] A.N. Aleshaev *et al.*, *Nucl. Instr.and Meth.* **A470** (2001), 240.

[2] B.P. Tolochko *et al.*, *Nucl. Instr.and Meth.* **A467-468** (2001), 990.

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International Tomography Center



The projection of a framework $[\text{Mn}_6(\text{O})_2(\text{Me}_3\text{C}_2\text{O}_2)_{10}(\text{L})_2]$; $R\text{-}3c$, $a = 27.476(4)$, $b = 27.476(4)$, $c = 66.576(8)\text{\AA}$, $V = 43526(10)\text{\AA}^3$.

Current research at the International Tomography Center of the Siberian Branch of the Russian Academy of Science (ITC SB RAS) in Akademgorodok, Novosibirsk) is centered on designing molecular magnets. This work involves the synthesis of solid organic, metalloorganic or coordination compounds that are formed from molecules or ions containing paramagnetic centers (i.e. molecules or ions having unpaired electrons) and for which a magnetic phase transition in the magnetic-ordering state can be determined. The current state-of-the-art in synthetic chemistry allows one to produce solid compounds with desired structural features starting from molecular precursors in solution. Crystals grown from these solutions can be designed to form chained, layered or frame polymers. However, to produce a magnetic phase transition in a ferromagnetic state not only the formation of layered or frame structures is required, but the presence of effective exchange channels between the paramagnetic centers is also necessary. Understanding the molecular and crystal structure of magnets allows the elucidation of the system of exchange clusters in solids and reveals magneto-structural correlations; the interrelation between the structure of a paramagnetic ligand, its coordination, the nature of the central atom, the character of

molecular packing, and magnetic properties. In some cases a series of X-ray diffraction experiments covering a wide range of temperatures is necessary to identify the structural changes occurring in solids of coordination compounds leading to a change in magnetic properties. At the ITC SB, X-ray diffraction studies of stable nitroxides and heterospin nitroxide-containing coordination compounds have been performed. The discovery of so-called 'breathing' crystals is the most significant result in recent years. 'Breathing' crystals are a group of Cu (II) hexafluoroacetylacetonate complexes with spin-labeled pyrazoles L^R ($R = \text{Me, Et, Pr, Bu}$) - $\text{Cu}(\text{hfac})_2L^R$, having a chain polymer structure with a 'head-to-head' or 'head-to-tail' motif that results in bridging coordination of L^R through the N atom of pyrazole and the O atom of the nitroxide group. These complexes are characterized by a reversible magnetic phase transition and a 10% compression of a unit cell up to 300 Å in absolute value. As temperature is lowered single crystals retain the quality necessary for X-ray diffraction, despite the occurrence of a structural phase transition. This allowed us to study compounds at different temperatures and to reveal the most important components of structural dynamics. Phase transition manifests itself in a sharp change in the coordination polyhedron of Cu(II). Also, weak ferromagnetic properties of Cu (II)-O•-N< clusters can be exchanged for strong antiferromagnetic ones. X-ray diffraction studies over a wide temperature range as well as investigations of crystal structures with long unit cell parameters became possible due to the acquisition of a Smart Apex (Bruker AXS) CCD diffractometer. Currently more than 300 structures per year are determined in ITC SB RAS.

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Neutron diffraction in Russia



Figure 1. Experimental hall of the IVV-2M reactor with neutron spectrometers of the 'Neutron investigations of condensed matter' centre, Institute of Metal Physics (Yekaterinburg).

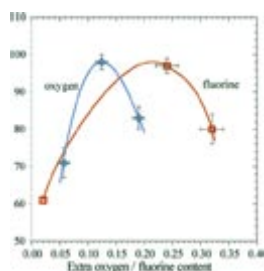


Figure 2. The behavior of T_c vs. extra oxygen or fluorine content in $\text{HgBa}_2\text{CuO}_4(\text{O},\text{F})_\delta$. Compounds with extra oxygen (O^{2-}) content $\delta \approx 0.12$ and extra fluorine (F^{1-}) content $\delta \approx 0.24$ demonstrate the same maximal $T_c \approx 98$ K.

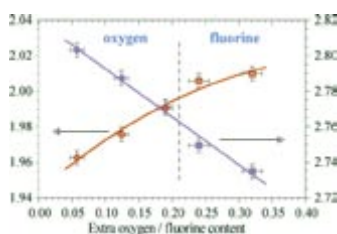


Fig. 3. Bond distances Hg-O2 (left scale, open symbols) and Cu-O2 (right scale, full symbols) as a function of extra oxygen or fluorine content. In both compounds with oxygen content $\delta \approx 0.12$ and fluorine content $\delta \approx 0.24$, $T_c \approx 97$ K.

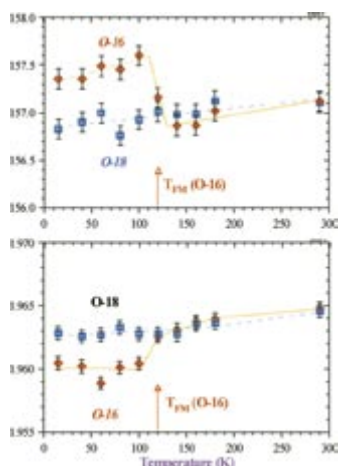


Figure 4. Comparison of the temperature dependencies of the average $\langle \text{Mn-O} \rangle$ bond length (at the bottom) and the average $\langle \text{Mn-O-N} \rangle$ valence angle (at the top) for two $(\text{La}_{0.25}\text{Pr}_{0.75})_{0.7}\text{Ca}_{0.3}\text{MnO}_3$ compounds with oxygen isotopes ^{16}O (O-16) and ^{18}O (O-18). The arrow indicates the temperature of the O-16 sample transition into the ferromagnetic state.

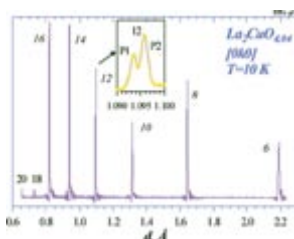


Figure 5. Diffraction pattern of the $\text{La}_2\text{CuO}_{4.04}$ single crystal measured at 10 K with HRFD. Figures denote the reflection orders. Each line is split, as shown for 12th order in the insert, due to crystal phase separation on the oxygen rich and oxygen poor phases.

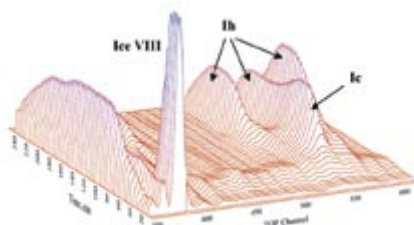


Figure 6. Phase transformations of high pressure heavy ice VIII, studied by neutron diffraction at DN-2 instrument. At the beginning time/temperature scale $T=94$ K, at the end $T=275$ K. The heating rate was ≈ 1 deg/min. Diffraction patterns have been measured each 5 min. Phase VIII is transformed into cubic phase Ic and then into hexagonal ice Ih.

Use of neutron diffraction for crystal structural investigations began in Russia in the early 1960s. The IBR reactor in the Joint Institute for Nuclear Research (JINR) (Dubna, Moscow Region) was the site of many scientific achievements, including the first pulsed neutron source in the world where time-of-flight methods were used for crystallographic experiments. At present neutron diffraction studies of atomic and magnetic structures in Russia are carried out at 4 large scientific centers with operational high-flux neutron sources: the Kurchatov Institute (Moscow), the Petersburg Nuclear Physics Institute (Gatchina), the Institute of Metal Physics (Yekaterinburg), and the Joint Institute for Nuclear Research (Dubna).

In the Kurchatov Institute, a IR-8 steady state reactor of 8 MW nominal power and average neutron flux of about 1×10^{14} n/cm²/s is used. The single crystal MOND and powder DISC diffractometers are the main instruments used for crystal studies. The PG double-monochromators are used at both diffractometers, which helps in varying wavelengths over a wide range (0.7 Å - 5.5 Å for MOND) with a 1×10^6 n/cm²/s flux at a sample position if $\lambda=2.4$ Å. With the MOND instrument dynamical effects were discovered in neutron magnetic scattering. ('Magnetic Pendellosung effect in neutron scattering by

perfect magnetic crystals' *Acta Cryst. A*, 1992, v.**48**, 100). Resonance magneto-acoustic and acousto-magnetic effects were experimentally found in perfect crystals of weak ferromagnets. It was found that measured neutron magneto-acoustic resonances are essentially non-linear in nature, which can be seen in the conditions of their stimulation, shape of resonance peaks, and evolution of oscillations over time. It was also established that magneto-acoustic non-linearity is connected with anharmonicity of a magnetic sub-system (*Physica B*, 1998, v.**241-243**, 736).

The multi-counter diffractometer DISC is intended for structural studies of microsamples. The low background levels and large solid angle of the detector system allow measurement of diffraction patterns from samples about 1 mm³ in volume in reasonable time. It permits crystal structure studies at very high external pressure in sapphire or diamond anvil cells. The main advantages of these cells are their small dimensions and the possibility of putting them into a refrigerator and cooling them down to helium temperatures. At DISC atomic structures and phase transitions in hydrides, oxides, fullerenes and amorphous substances are studied (see, for instance, 'Pressure induced spin-orientation transition in FeBO₃' *High Pressure Research*, 2000, v.**17**, 179).

The steady state reactor IVV-2M of 15 MW power (Fig. 1), which operates up to 6000 hours annually, supports the 'Neutron investigations of condensed matter' Centre, which belongs to the Institute of Metal Physics (Yekaterinburg). In this Centre there are several neutron diffractometers and special devices for investigations of physical properties of both conventional and radioactive samples. Among them: a multi-counter high-resolution powder diffractometer ($\lambda=1.515 \text{ \AA}$, $\Delta d/d=0.002$), two medium-resolution diffractometers, and a multi-counter four-circle single-crystal diffractometer. They are all equipped with special cells for radioactive samples studies in 4.2/1000 range after their irradiation in the reactor core. For high-pressure experiments, cylinder-piston cells up to 1.2 GPa are used. In the Centre, study of the influence of various defects (doping, non-stoichiometry, disorder caused by irradiation with fast neutrons, light or heavy ions, electrons) on crystal structure is the main topic. The main goal of these studies is to study the relation between real (defect) crystal structure and physical properties. In the Centre, studies of structural and magnetic phase transitions, charge ordering in oxides and inter-metallic rare earths or 3d-transition element compounds are also carried out (*J. of Alloys and Compounds*, 2001, v. **315**, 82).

At the Joint Institute for Nuclear Research (JINR) in Dubna, neutron scattering experiments are performed at the IBR-2 pulsed reactor with record average power (2 MW) and pulsed neutron flux ($1 \times 10^{16} \text{ n/cm}^2/\text{s}$). The IBR-2 set-up includes three diffractometers for structural studies of single crystals and powders and three diffractometers for texture and internal stress measurements.

The Fourier high-resolution diffractometer (HRFD) is analogous to the mini-SFINKS facility in Gatchina, but with higher d -spacing resolution ($\Delta d/d=0.001 - 0.0005$). A study of mercury-based high- T_c superconductors with various percentages of oxygen or fluorine in the basal plane is an example of an experiment using the HRFD (Figs. 2 and 3). In these studies several results of significance for understanding high- T_c superconductivity in copper oxides have been obtained. Recently, a series of diffraction experiments with doped CMR manganites has been performed. Precision structural analysis of several compounds, including compositions enriched with ¹⁸O isotope, provides unique data (*Eur. Physical J. B*, 2001, v.**19**, p.215, Fig. 4). HRFD can be used for single crystals if its very high resolution is needed. A typical example of such a problem is the mesoscopic phase separation in La₂CuO_{4+ δ} , which appears to be due to low-temperature diffusion of extra oxygen. Despite extremely small difference in lattice parameters arising from a homogeneous state, the HRFD helped in measuring split diffraction peaks and in determination of sizes of the coherent regions with antiferromagnetic and superconducting phases (Fig. 5).

DN-12 is a time-of-flight complementary version of the DISC diffractometer for micro-samples. Its

parameters allow one to study powders of about 1 mm^3 volume inside sapphire or diamond anvils cells. The crystal and magnetic structures of manganites $\text{Pr}_{0.7}\text{Ca}_{0.3}\text{Mn}_{1-y}\text{Fe}_y\text{O}_3$ and $\text{Pr}_{0.8}\text{Na}_{0.2}\text{MnO}_3$ with the CMR effect were investigated at pressures up to 4.5 GPa and in a temperature range of 16-300⁰. In these compounds which have significantly different magnetic structures at normal pressure, stabilization of the AFM state of A-type takes place at high pressures and low temperatures (*High Pressure Research*, 2003, v.**23**, p.149 and *J. Magn. Mat.*, 2003, v.**267**, p.120). With this instrument it is possible to simultaneously measure elastic (diffraction) and inelastic neutron scattering. This mode was used for investigation of structure, phase transitions and atomic dynamics in ammonium halides at pressures up to 10 GPa (*High Pressure Research*, 2000, v.**17**, p.251).

The DN-2 diffractometer is used for single crystals. Very high neutron flux ($\sim 10^7\text{ n/cm}^2/\text{s}$) at the sample position and an extremely large d -spacing interval available at DN-2 are used in real time experiments with powders. Simultaneously with diffraction patterns, small angle scattering data can also be collected (Fig. 6).

The FSD diffractometer ($\Delta d/d \approx 0.004$) continues the development of neutron Fourier techniques at long-pulse neutron sources, which is carried out in collaboration with PNPI. FSD is optimized for internal stress measurements in bulk materials (*Applied Physics A*, 2002, v.**74**, S86). Auxiliary equipment (loading device, mirror furnace, Huber goniometer etc.) allows one to broadly vary experimental conditions. EPSILON ($\Delta d/d \approx 0.003$) is used for stress measurements with rocks. SCAT is a diffractometer with a ring detector intended for texture analysis on rock samples. It is equipped with a high pressure chamber ($P_{\text{max}} \approx 1.5 \times 10^4\text{ N}$, $T_{\text{max}} \approx 700^\circ\text{C}$) in which the diffraction study of textures of amphibolites and gneisses from the super deep borehole SG-3 in the Kola Peninsula and their analogues from the surface were performed (*XXVIII General Assembly of the European Seismological Commission*, Italy, Genova, 2002).

Broad perspectives for neutron diffraction studies in Russia will be opened when a new steady state high-flux reactor PIK ($W=100\text{ MW}$), which is under construction in PNPI (Gatchina) opens. Very high neutron flux from PIK, modern equipment, and wide experience in diffraction studies will all help in solving both fundamental and applied problems.

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L.Ya. Karpov Institute of Physical Chemistry

The X-ray Laboratory in Karpov Institute established in 1938 by G. Zhdanov has become one of the largest centers of X-ray analysis in the former Soviet Union and Russia. The laboratory staff is engaged in projects with chemists from the Russian Federation, Georgia, Latvia, Moldova, the Ukraine, Sweden, Denmark, Spain, Portugal, and South Africa. Several thousand structures have been determined and deposited in the the Cambridge and Karlsruhe databases.

Special procedures have been developed to speed the process of data collection using programs PROFIT (profile fitting) and PAN32 (profile analysis) to treat data collected on Syntex, Nicolet and Enraf Nonius diffractometers. A method has been developed for evaluating the contribution of thermal diffuse scattering (TDS) to structure factors based on scanning peak profiles (program DISCONT). The Rietveld method is also widely used in powder crystal experiments.

High-precision X-ray diffractometry has allowed us to perform electron density studies and a new method for determination of the electron localization function from electron density has been developed. Recently, new computer software for charge density studies (WinXPRO2003) was released as part of a

joint project with the Mendeleev University, Moscow.

Contact: www.nifhi.ac.ru/~adam

St Petersburg State University, Department of Crystallography

The Department of Crystallography at St Petersburg State University was established in 1924 by students of E.S. Fedorov. During the last ten years investigations have focused on structural chemistry, structural mineralogy, and crystal growth. Almost every year a national or international conference is organized by the Dept. The *XV International Conference on X-ray Diffraction and Crystal Chemistry of Minerals* in September, 2003 was organized by the Commissions of X-ray Analysis of Minerals and Crystal Chemistry of the Russian Mineralogical Society RAS. The Department has collaborations with institutes and universities in Germany, USA, Switzerland, Netherlands, and Austria, etc. Below we provide a brief description of the main scientific groups.

The Borate and borosilicate group (Rimma S. Bubnova, rimma_bubnova@mail.ru and Stanislav K. Filatov, filatov@crystal.pu.ru) investigates borate and borosilicate crystals and glasses in collaboration with Peter Paufler (Paufler@physik.tu-dresden.de) at Dresden Technical University, Germany. The first crystal structure determinations of borates at elevated temperatures demonstrated the rigidity of boron-oxygen groups that maintain their configuration and size on heating. Highly anisotropic thermal expansion of 40 borates has been described for the first time and has been interpreted as a result of hinge deformations. (S.K. Filatov, R.S. Bubnova, *Phys. Chem. Glasses*, 2000, **41**, N 5, 216-224).



S.K. Filatov working at the Bauxite field, Tolbachik volcano, Kamchatka, Russia, in 2000.

Since 1977, 150 minerals of volcanic eruptions from Kamchatka volcanoes were characterized by the Volcanology group (S. K. Filatov, filatov@crystal.pu.ru) in collaboration with Lidia P. Vergasova (vlp@kcs.iks.ru; Institute of Volcanology RAS, Petropavlovsk-Kamchatskiy). In the course of these studies, about 25 new mineral species were discovered, most of them being oxosalts. Many of these structures contain additional oxygen atoms coordinated by four metal atoms M (Cu, Pb, etc.), forming oxo-centered OM_4 tetrahedra, a new concept in inorganic crystal chemistry, (S.V. Krivovichev, skrivovi@mail.ru and S.K. Filatov). Microbiological activity was revealed in the transformation of volcanic products to bauxites.



Natural (a, b) and synthesised (c, d) pseudomorphs, inheriting (a, c) and losing (b, d) the primary face relief. a, b – goethite after pyrite (Mining Museum collection, St. Petersburg), c – copper chromates after copper vitriol; d – potassium chromates after alum.

The Group of O.V. Frank-Kamenetskaya (Olga@of3102.spb.edu) studies the structure and classification of minerals with atomic defects (solid solutions, mixed crystals, compounds of non-

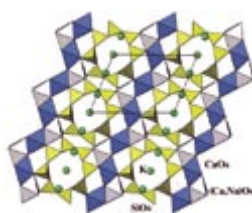
stoichiometric and variable composition). The group uses X-ray based analytical approaches to study chemically inhomogeneous 'single crystals' and have characterized a series of solid solutions (fluorides, sulfides, oxides, silicates). O.V. Frank-Kamenetskaya, I.V. Rozhdestvenskaya, *Crystal Chemistry*. V. 33, 2nd Revised Edition, SPb: Yanus, 2004.



Boat trip along Neva River: Top row (left to right): S. Ghose (USA), M.G. Krzhizhanovskaya (Russia), Th. Schleid (Germany), G. Ferraris (Italy), V.S. Urusov (Russia), R.S. Bubnova (Russia), P. Paufler (Germany), B. Albert (Germany), V.V. Dolivo-Dobrovolskiy (Russia). Bottom row: S.K. Filatov and students (Russia), September 2003.

The crystallogenes group headed by Arkadii E. Glikin (glikin@ag2460.spb.edu) has elaborated upon and extended the fundamentals of crystal formation in solutions to describe solid phase interactions typical of minerals: metasomatic replacement and joint growth of different crystal phases, mixed crystal formation, aggregate recrystallization, epitaxial and quasi-epitaxial overgrowth as well as crystal habit formation. A.E. Glikin. *Polymineral-Metasomatic Crystallogenesis*. St. Petersburg; Ed. Journal 'Neva', 2004. 320 p. In September 2001, An International Conference on Crystallogenesis and Mineralogy was organized.

The Crystal chemistry of paraffins group (Elena N. Kotelnikova, elena@ek7740.spb.edu, and S.K. Filatov) has studied normal alkanes C_nH_{2n+2} as representatives of the rotatory state of crystalline matter. Experimental data on structural deformations, phase transitions, solid solutions, and phase equilibria of synthetic ($n=17-24$) and natural ($n=17-37$) n -paraffins have been obtained and generalized as functions of homological composition and temperature. In additions, phase diagrams of binary paraffin systems have been developed ('Neva', 2002, 352 p. (in Russian)). The First Russian Meeting on Organic Mineralogy was held in 2002 (chairmen: E.N. Kotelnikova and S.K. Filatov).



Crystal structure of tubular silicate frankamenite $K_3Na_3Ca_5[Si_{12}O_{30}](OH)F_3 \cdot H_2O$.

The Crystal chemistry of uranyl and heavy metal compounds group (Sergey V. Krivovichev, skrivovi@mail.ru) investigates uranium and heavy-metal minerals and inorganic compounds relevant to the safe disposal of radioactive waste and environmental pollutants (in collaboration with Peter C. Burns, University of Notre Dame, USA). As a result of this joint effort, more than 90 original structure determinations have been completed. This provides a unique basis for understanding the stability of uranium and heavy metal minerals in the environment and their role in environmental pollution.

The Pathology of crystals group of Yurii Punin (Head of the Dept., olga@os2489.spb.edu) investigates crystal growth instability that leads to the drastic distortion of the outer form and inner structure of crystals during their growth. As a result of this research, a theory of autodeformation defects has been elaborated. The group also developed a complex approach to the problem of growth

dissymmetrization and the nature of optical anomalies in crystals on the basis of extensive kinetic and morphological studies of crystal growth in a surface-active environment.

The Tubular silicate group of Ira V. Rozhdestvenskaya (ivrozhdestvenska@mail.ru) works on alkali calcium silicates with tubular radicals, including studies of such exotic silicate minerals as frankamenite, canasite, miserite, tokkoite, tinaksite and agrellite found in charoitite rocks of the Murun massif, western Aldan Shield, southeastern Siberia. They are layer structures that consist of alternating structural modules: walls of Ca-, and Ca, Na-polyhedra with silicate anions located between the walls. The silicate anions form tubes or bent ribbons in wide channels.

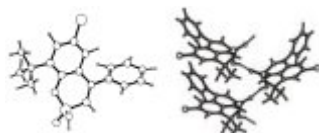
X-ray Laboratory, Dept. of Inorganic Chemistry, Faculty of Chemistry. The laboratory is involved in studies of inhomogenous crystals (solid solutions decomposition, nucleation) and nonstoichiometric compounds (R.A. Zvinchuk, Head of the lab); structures of modified steroid estrogens exhibiting selective biological activity and analysis of structure-property relationships for creation of new drugs (CCDC 164249-164261) (G.L. Starova, starova@VK4829.spb.edu); short-range order in complex stoichiometric 'disordered' oxides with heterovalent isomorphism and Rietveld refinement (Yu.E. Smirnov); and construction of derivative structures on the basis of non-characteristic crystallographic orbits and cyclotomical Patterson's sets.

Contacts have been included in the text.

Laboratory of Diffraction Methods in Kazan



In 1996 the Centre of Physical Methods of the Russian Foundation for Basic Research was established at the A.E. Arbuzov Institute of Organic and Physical Chemistry of the Kazan Scientific Centre of the RAS in the Volga region. The laboratory has two CAD4 Enraf-Nonius diffractometers and a scientific staff of two doctors of chemical sciences, four PhDs and several post graduate students. The laboratory performs X-ray diffraction studies for the Arbuzov Institute, for universities and institutes of Kazan, as well as for research institutes of the Volga region, Ekaterinburg, Ufa, Irkutsk, and St Petersburg. About 200 structures of organic, phosphorus-containing, organoelement and metalloorganic compounds are studied annually.



Geometry of substituted 5,6-benzo-1,2-oxaphosphorin-3-ene and the system of hydrogen bonding in the crystal.

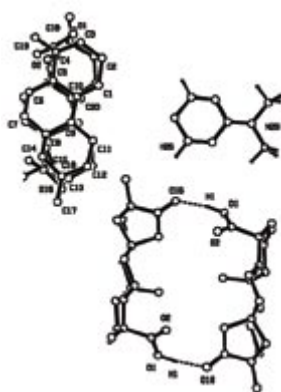
Major research at the institute involves the synthesis and structures of phosphorus compounds, macrocyclic and cage organic compounds, and supramolecular chemistry using a variety of physical methods. I.A. Litvinov conducted a series of experiments on cyclic phosphorus-containing compounds including unsaturated 6- and 7-membered phosphaheterocycles. It was shown that a model of hyperconjugative stereoelectronic interactions can describe the position of the substituents on the phosphorus atom, as well as the variations of molecular geometry in the absence of strong

intermolecular interactions, such as hydrogen bonding.



Schematic representation of localized hydrophilic and hydrophobic regions.

Recent studies have revealed a pattern of localized hydrophobic and hydrophilic domains in supramolecule systems. Analysis of a variety of crystals revealed 4 types of packing depending on the ratio of hydrophilic and hydrophobic regions; homogeneous, spherical, cylindrical, and lamellar. The type of packing correlates with the symmetry of a crystal. Lamellar packing is observed only for low symmetry (triclinic - orthorhombic) rod type crystals and for low symmetry tetragonal and trigonal crystals, while homogeneous and spherical domains can be observed in all crystals, even in cubic systems.



Geometry of molecular complex of isosteviol with dimethylaniline.

A series of investigations of molecular complexes of isosteviol revealed that isosteviol forms isostructural tetragonal crystals in molecular complexes with aromatic compounds having a guest-host ratio of 1:2. This phenomenon may be used to separate spatial isomers of aromatic compounds.

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Kurnakov Institute of General and Inorganic Chemistry, Moscow



Researchers in the Laboratory of Crystal Chemistry of Coordination Compounds.

In the Kurnakov Institute of General and Inorganic Chemistry, there are two laboratories that study crystal structures of different classes of compounds, namely, the Laboratory of Crystal Chemistry of Coordination Compounds and the Laboratory of X-ray Structure Analysis. The Laboratory of Crystal Chemistry of Coordination Compounds was founded in 1945 by G.B. Bokii, who led the laboratory until 1959. In 1959-1990, the laboratory was headed by M.A. Porai-Koshits, and since 1990 it has been headed by V.S. Sergienko.



Researchers in the Laboratory of X-ray Structure Analysis.

For many years the wide variety of interests of M.A. Porai-Koshits determined the compounds studied in the laboratory: complexes of Group V-VII metals with multiple metal-oxygen bonds; isopoly- and heteropolycompounds; binuclear complexes of Rh and other Group VIII metals containing metal-metal bonds; d-metal complexes with organic chelating O-, N-, and S-donating and macrocyclic ligands; heterometal clusters; optically active Pt complexes with amino acid ligands; mono-, di-, and triaminocarboxylates and their mono- and diphosphonate analogues; isoquinoline derivatives and their complexes; amino- and phosphoryl-containing podands; and mixed-ligand Au(I) and Hg(II) complexes.

New projects at the laboratory include:

- (1) Investigation of a wide spectrum of secondary interactions (traditional hydrogen bonds, C-H... π contacts, proton-hydride, attractive, agostic, and stacking interactions, etc.) that play an important role in the formation of crown-ether styryl dyes, charge-transfer complexes based on π -donating bis(18-crown-6)stilbenes, diaryl esters exhibiting liquid-crystalline properties, and dipyrityls and their coordination polymers with Ag(I).
- (2) The trans effect of multiply bound peroxo ligands in pseudo octahedral $\text{VO}(\eta\text{-O}_2)\text{L}_4$ complexes (where L is a donor atom of a monodentate and/or polydentate ligand) was characterized and the structures of the Group IV-VI metal (Ti, Ta, V, Nb, Mo, W) oxoperoxo complexes of this type were analyzed;
- (3) Specific features of regioselective acid-catalyzed substitution of exo-polyhedral hydrogen atoms in the decaborate anion $\text{B}_{10}\text{H}_{10}^{2-}$ were studied. The main product of these reactions is the equatorially monosubstituted derivative.
- (4) Structures of $\text{LaL}_3(\text{Phen})_n$ mixed-ligand complexes (where L is dipivaloylmethanate or hexafluoroacetylacetonate) were determined to establish correlations between the structure and luminescence properties.



The Soviet postage stamp devoted to the 50th anniversary of foundation of the Kurnakov Institute of General and Inorganic Chemistry. The figure on the stamp represents the $[\text{Re}_2\text{Cl}_8]^{2-}$ structure. The Laboratory of X-ray Structure Analysis was organized in 1934 by N.V. Ageev (1934-1953) and N.G. Kuznetsov (1953-1979). Yu.N. Mikhailov has been the head of the laboratory since 1979. The studies performed in the laboratory are related to the interests of other laboratories of the Institute, including the relationships between the composition, structure, and properties of coordination and inorganic compounds. One of the most interesting results was obtained in the laboratory in the 1960s when the first quadruple rhenium-rhenium bond was revealed in the $[\text{Re}_2\text{Cl}_8]^{2-}$ anion. Later, the interpretation of

this bond was confirmed by F.A. Cotton. During the last five years, investigations of monomeric, dimeric, and trimeric compounds of rhodium, iridium, molybdenum, and platinum at different oxidation states have been performed along with the studies of polynuclear compounds containing complex metal-metal bond systems.

Important results have been obtained for nontransition p elements at low oxidation states. Mixed-ligand polymeric compounds of tin(II) with nitrogen-containing organic cation supramolecular compounds having large hollows and channels. Some uranyl complexes with fluoride ligands and tetradentate bridging oxalate ions contain infinite channels approximately 7-10 Å in cross-section. Channels that are formed by cyclic dioxygen anions were found in the structures of some boratobismuthates, the high-temperature modification of $\text{Na}_2\text{B}_4\text{O}_7$, and double potassium-bismuth citrate.

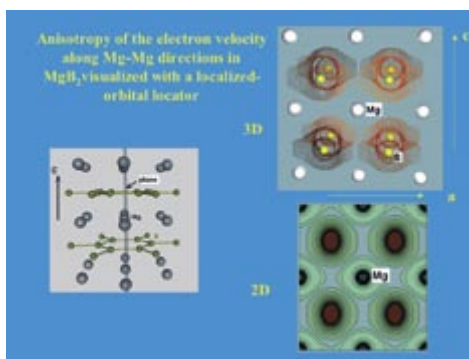
A large series of mixed-cation RE compounds with condensed anions (phosphates, phosphatoborates, phosphatogermanates, germanates, oxophosphatovanadates) have been studied to reveal the effect of cations on the structure of the anionic group. It was found that in the noncentrosymmetric ultraphosphates, the $(\text{P}_8\text{O}_{23})^{6-}$ anion contains isolated oligomers consisting of three connected six-membered rings. These compounds are candidates for quantum electronics. In the last three years, efforts have involved structural studies of polymeric coordination compounds of tin, bismuth, and uranyl with bridging oxo anions, polymeric complexes of silver with nitrogen-containing organic compounds, supramolecular compounds of doubly and triply charged metals with hexamethylenetetramine and different aminopolycarboxylic acids. Crystal structures of these compounds contain large hollows and channels that can be used for preparation of molecular sieves and ion exchangers. Over 150 crystal structures a year are determined by two groups using two Enraf-Nonius CAD-4 diffractometers.

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Electron density at Mendeleev University

Research in the Mendeleev University group, headed by Vladimir Tsirelson, deals with describing bonding in solids in terms of electron density and electrostatic potential, as well as related functions describing local energies and potentials. Early studies were based on the Bader's quantum mechanical topological theory, which was applied to experimental electron density for the first time by Tsirelson and Streltsov in 1985. This approach is now widely accepted. The extensive range of compounds studied spans simple and binary crystals, perovskites, spinels, garnets, silicates, and molecular crystals. The materials were studied using topological theory to quantify atomic and molecular interactions and to elucidate the physical nature of the spatial architecture of crystalline systems.

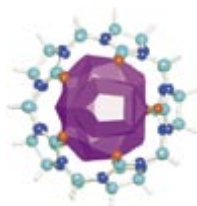
Other studies are devoted to topological analysis of the electrostatic potential in molecules and crystals. It has been demonstrated that the nuclei of neighboring atoms are separated in the inner-crystal electric field by surfaces of the zero-flux potential gradient, inside of which the nuclear charge is completely screened by an electronic cloud. These electrically neutral bonded pseudoatoms define the regions in a crystal dominated by a charge of one or another nucleus, no gradient lines connecting the anions were found. The results led to a physically reasonable description of the Coulomb field features in a system, which is a key point in the development of corresponding models for force field.



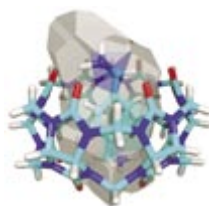
Most recently fundamental research has been performed in collaboration with the X-ray laboratory of the Karpov Institute to combine experimental electron density with the formulae of density functional theory to calculate kinetic energy, potential energy, and exchange energy, etc. It has been shown that maps of kinetic and potential energy densities explicitly reveal features of electronic energy resulting from the molecule or crystal formation, while the integral values of these functions over the atomic basins yield the components of the electronic energy for the bounded atoms. Becke *et al.*'s electron localization function and localized-orbital locator (see figure) and Parr *et al.*'s local temperature and local internal entropy of electron gas have also been approximately expressed in terms of electron density and its derivatives. As a result, X-ray diffraction experiments have been extended to provide detailed descriptions of atomic and molecular interactions in a crystal in a form compatible with a quantum mechanical picture.

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Inorganic structure in Samara



(a)



(b)

Molecular Voronoi-Dirichlet polyhedra (a) of a cavity and (b) of a substrate molecule inside a cucurbit[6]uril molecule.

At Samara State University (SSU) crystallographic and crystallochemical research is conducted in the department of inorganic chemistry. Basic areas of study include: (i) development of computer methods for crystallochemical analysis (V.N. Serezhkin, V.A. Blatov, A.P. Shevchenko), (ii) structure and properties of uranium complexes (L.B. Serezhkina) and compounds containing atoms with lone pairs (D.V. Pushkin).

At present SSU researchers are working on a unique program package (TOPOS) for multi-purpose crystallochemical analysis (a user manual and demo and beta versions are available at www.topos.ssu.samara.ru/). TOPOS is an integrated interactive shell that supports a relational crystal structure database. To analyze crystal structure information TOPOS uses methods based on the quantitative characteristics of Voronoi-Dirichlet polyhedra that avoid using crystallochemical radii or a

priori assumptions concerning the nature of interatomic bonds. The methods provide for unified analysis of crystalline substances at the atomic, molecular and supramolecular levels. Commercial and non-commercial versions of TOPOS are installed in a number of institutes of the RAS and in universities in France, Japan, Italy, Spain, and the UK.



Bottom row (left to right): L.B. Serezhkina and V.N. Serezhkin; back row: D.V. Pushkin, V.A. Blatov, and A.P. Shevchenko.

TOPOS was created to (i) implement and combine computer methods of crystallochemical analysis within a unified data-analytical system; (ii) provide resources for the complex automatic analysis of large groups of chemical compounds to search for common crystallochemical features; and (iii) maintain objectivity while performing crystallochemical analysis.

TOPOS provides the user with tools to (i) calculate coordination numbers of atoms or molecules, (ii) assess a number of geometrical characteristics of atomic and molecular domains; (iii) estimate stereo effects caused by lone pairs or by the Jahn-Teller effect; (iv) analyze the far coordination spheres of atoms or molecules, and study the topology of atomic and molecular packing; (v) search for topological relationships between chemically and stoichiometrically different crystal structures, perform crystallochemical classification; (vi) estimate sizes of voids, cavities and channels in crystals, reveal agostic contacts and non-valence interactions; and (vii) predict stability of coordination compounds and supramolecular aggregates.

Various aspects of TOPOS have been demonstrated by analyzing different inorganic and coordination compounds including σ - and π -complexes, minerals, superionic conductors, zeolites, etc. Using uranium(VI) compounds as an example it was shown that the solid angles by which the faces of a Voronoi-Dirichlet polyhedron are 'seen' from the nucleus of a complexing atom can be used to evaluate the electron-donor capabilities of oxygen-containing ligands with the 18 electron rule. It has been proven that using the ligand electron-donor characteristics obtained with crystal structure data, one can predict the directions of stepwise complexation in water solutions as well as the composition and structure of resulting uranium(VI) complexes.

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MEETING REPORT

Reminiscences from ECM-22

Budapest, Hungary, August 25-31, 2004

Mariusz Jaskolski

The 22nd meeting of the European Crystallographic Assn, ECM-22, attracted nearly 700 people in Budapest, the capital of Hungary. It was a memorable meeting. The charm of Budapest, accentuated by the bridges on the majestic Danube River, the hills, and the impressive architecture, will live in the memories of the participants, as will the smooth organization of the conference. This we owe to A. Kálmán, Chair of the Organizing Committee, and to his young adjutants, L. Fábián and P. Bombicz. Special thanks are due to Petra who, as the Secretary, took quiet and efficient responsibility of almost everything. The months leading to ECM-22 had been very busy, but the work was well organized, a source of satisfaction for those involved. Everything was handled electronically, even the deliberations of the Bursary Committee. Thanks to generous help from the IUCr and the ECA, nearly 100 young scientists were awarded financial support. And youth was surely visible in all the activities of the meeting. This is a good prognostic for the future of our discipline in Europe. A sad point is, that over 20 of the awardees did not show up or withdrew at the last minute.

The meeting's home was the new Convention Centre and the Chemistry Dept. of the Eötvös Loránd U. There were some small inconveniences (such as overflowing seminar rooms and poor audio facilities in the main hall) but they are already forgotten, suppressed by the overwhelmingly positive impressions. The participants and exhibitors were particularly happy with the central location of the exhibition area. It became the favorite place for chatting with friends and for scientific discussions. People were crowding at the booths with instruments and supplies but also, I'm glad to note, around the two book stands. The printed word seems to be doing fine in the era of electronic information.

The scientific program covered virtually all aspects of crystallography and a spectrum of interdisciplinary subjects, from materials sciences to molecular biology. There were 42 microsymbiosia and 16 plenary lectures. A quick look at the keynotes shows topics covering new phasing and statistical methods, new materials, data mining and molecular recognition, biological membranes and virus structure, extreme and special techniques, and even robotics and escience! Perhaps the spectacularly dynamic field of macromolecular crystallography was somewhat underrepresented, but this view may be biased by my personal interests.

In retrospect, I have the most vivid memories from the opening ceremony. It was then that the ECA presented for the first time the Max Perutz Prize. It went to George Sheldrick, an Englishman working in

Germany. The ECA has already awarded a number of prestigious Prizes (Ada Yonath 2000, Jochen Schneider, 2001, Carmelo Giacovazzo, 2003), but Max Perutz became their patron only this year, on the initiative of the Executive Committee. Max Perutz, who passed away two years ago, was one of the giants of European crystallography. An Austrian working in England, Perutz pioneered an entirely new area in crystallography. It is, therefore, very befitting to honor the most creative European crystallographers with a diploma bearing the name of Max Perutz. The title on the diploma handed to George Sheldrick by Hartmut Fuess, President of the ECA, and Anders Liljas, Chair of the Prize Committee, reads: 'for seminal contributions to the development of direct methods and for converting the theory into straightforward procedures for solving small and large crystal structures'.

In his address following the presentation ceremony, George remarked that he had overlapped with Max Perutz for 18 years at Cambridge without ever having any direct contact! George was then in inorganic chemistry at the University Chemical Lab, and Max Perutz was in the Medical Research Council. George offered three interesting thoughts. (1) Crystallography is a truly interdisciplinary science. While this is quite obvious, the corollary is that rigid administrative constraints should not impede migration of crystallographers to new areas. George is the best example of what benefits such migrations bring: started in inorganic chemistry, went to crystallographic methodology and organic crystal chemistry, to finally make very important contribution to protein crystallography. (2) Crystallographers should talk to each other; for example, small molecule crystallography can learn a lot about model bias from macromolecular crystallography, and macromolecular crystallographers do not have to re-discover things that are only too familiar in the small molecule field (for instance C-H and π hydrogen bonds). (3) Openness and sharing is more beneficial to everybody than not sharing. He illustrated this with computer programming practices. In the early days, open code was the only way to distribute programs. Today, the situation has changed. But is it for the better?

With a note of pride, Hartmut Fuess emphasized that European crystallographers, as a community and as individuals, have definitely contributed to the integration of the Old Continent and perhaps even to the enlargement of the European Union. From the conception of the ECM's, the idea has been to bring the East and West together, to avert isolation. In this sense, the fact that the first ECM after May 1, 2004, was held in Budapest, one of the new capitals of the enlarged European Union, is symbolic in itself. It must be noted here that the geography professed by crystallographers is different than in popular atlases. For example, Israel has been a member of our community 'since always', and the continent of Africa is in fact part of crystallographic Europe. The previous ECM was held in Durban, on the southern hemisphere! But despite all the integrative efforts, there are still white patches even on the map of Old Europe. To overcome this, the Executive Committee is extending invitations to join the ECA to all those countries where there is at least some crystallographic activity. In this spirit, we had, as observers, colleagues from Belarus and Moldova.

When Bill Duax, President of the IUCr, took the podium during the opening ceremony, he unfolded an even more global view. In a unique way, crystallography has formed a clear international community, and has contributed to understanding and contacts between people all over the world. This fantastic role of uniting people around common intellectual ideas may have far-reaching effects; perhaps it can contribute to fighting terrorism in a better way than many political actions.

Alajos Kalman, with typical Hungarian hospitality, presented the guests of honor with souvenirs at the opening session. 'Bill Duax will not be present at the closing ceremony', he said. Bill, who is known as a strong advocate of full conference participation, responded quickly and firmly, explaining that, under very exceptional circumstances, he had to leave on the eve of the last day.

In Budapest, I was accompanied by my son Maciej (17). To my despair, his interests are far from scientific, but I took him to the opening ceremony, to one of the scientific sessions, and to the closing

ceremony on the Danube cruise. And he was very impressed. Not by the scientific content, but by the atmosphere and the general family-like feeling. He thought that everybody knew everybody in Budapest. And we were quite a crowd!

Mariusz Jaskolski, ECA Vice President

MEETING REPORT

ECM-22 Satellite

Crystallography at the start of the 21st century: mathematical and symmetry aspects

Budapest, Hungary, August 2004

The XXII European Crystallographic Meeting, held in Budapest, Hungary, was preceded by a three-day Satellite Meeting on *Crystallography at the start of the 21st century: Mathematical and Symmetry Aspects*, which represented the first public activity of the MaThCryst International Workgroup (www.lcm3b.uhp-nancy.fr/mathcryst/), created to draw the attention of researchers working in crystallography and related fields to the fundamentals aspects too often neglected in favor of a blind machine-based solution strategy (IUCr Newsletter, **10**(3), 2003 2).

The Satellite, supported by the Hungarian National Committee of the IUCr and the ECM-22 Organizing Committee, and sponsored by the Commission on Inorganic and Mineral Structures of the IUCr (IUCr-CIMS), was attended by 51 participants with different backgrounds (chemistry, physics, mineralogy, mathematics...) from 20 countries. There were six thematic sessions on diverse topics such as geminography (twinned crystals), OD structures, normalizers of space groups, graph theory, chirality and achirality in crystal structures, higher dimension crystallography, tilings and ball packings, modulated structures, and quasicrystals. The proceedings will soon appear in a special issue of *Zeitschrift für Kristallographie* and will include peer-reviewed original and review articles by the lecturers, as well as contributed peer-reviewed articles by the participants.

The satellite also included poster sessions. The electronic version (PDF file) of the abstracts is available for download from the website (www.lcm3b.uhp-nancy.fr/mathcryst/satellite.htm). The next public activity of the MaThCryst group will be a Summer School on 'Mathematical and Theoretical Crystallography' at the U. Henri Poincaré in Nancy, France, June 20-24, 2005, under the auspices of the IUCr and of the ECA (see announcement in IUCr Newsletter, Vol. **12**, No. 3, page 22 or visit www.lcm3b.uhp-nancy.fr/mathcryst/nancy2005.htm).

Massimo Nespolo, Organizer, ECM-22 Satellite Meeting