

INTERNATIONAL UNION OF CRYSTALLOGRAPHY

IUCr Newsletter

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Original article text and available illustrations

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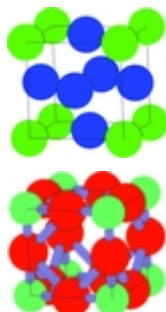
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On symmetry classes of crystal structures

***Acta Cryst.* (2009). A65, 456–465 (doi.org/10.1107/S0108767309030116)**



The structures of AuCu₃ (top) and ReO₃ (bottom) lead to the same symmetry class but represent two different structure types.

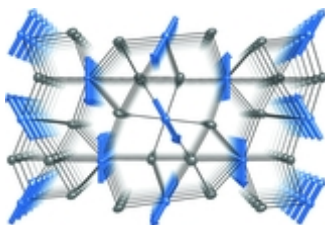
The paper deals with an open-ended classification scheme for crystal structures based on classes of Wyckoff positions (Wyckoff sets) by special supergroups of the space groups (affine normalizers). These classes are free of metrical and geometrical considerations. The multiplicities of Wyckoff positions used in a structure description are added up and lead to a number of sites for atoms. Wyckoff sets combined with these numbers lead to classes of crystal structures. A symmetry class of crystal structures contains all members of a structure type, example *cf.* figure.

H. Burzlaff and H. Zimmermann

Magnetic behaviour of synthetic Co₂SiO₄

Magnetic behaviour of synthetic Co₂SiO₄

***Acta Cryst.* (2009). B65, 664-675 (doi.org/10.1107/S0108768109042499)**



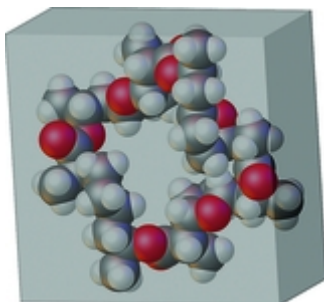
Many studies have provided abundant data on the olivine-type oxides, especially at or above room temperature and at high pressure, because of the fundamental importance for geologists and mineralogists. However, there is still a lack of information on the magnetic properties below room temperature for such materials. In the present paper, the detailed symmetry analysis of the Co-olivine, Co₂SiO₄, magnetic structure was performed based on non-polarized and polarized neutron diffraction experiments.

A. Sazonov, M. Meven, V. Hutanu, G. Heger, T. Hansen and A. Gukasov

IUCR JOURNALS NEWS

Pseudomerohedrally twinned monoclinic structure of unfolded 'free' nonactin: comparative analysis of its large conformational change upon encapsulation of alkali metal ions

Acta Cryst. (2009). C65, o521-o524 (doi.org/10.1107/S0108270109033083)



The elusive structure of anhydrous 'free' nonactin has been unambiguously established and for the first time its precise bond distances and angles were determined. Nonactin crystallizes in a pseudomerohedrally twinned primitive monoclinic cell with two similarly sized twin components. It possesses an unfolded and more planar geometry than its complexes with encapsulated Na^+ , K^+ , Cs^+ , Ca^{2+} or NH_4^+ cations that exhibit more isometric overall conformations. The geometries were analyzed with a new routine that computes the dimensions of the smallest parallelepiped superscribing a molecule.

I. A. Guzei, C. Wang, Y. Zhan, O. V. Dolomanov and Y.-Q. Cheng

IUCR JOURNALS NEWS

Dwelling in the dark: procedures for the crystallography of phytochromes and other photochromic proteins

***Acta Cryst.* (2009). D65, 1232-1235 (doi.org/10.1107/S0907444909034106)**



Schematic and photographic view of the simple infrared set-up to observe and freeze phytochrome crystals

Photoreceptor crystals are sensitive to light irradiation and tend to diffract poorly. An efficient and inexpensive method is presented to manipulate photochromic proteins in complete darkness using infrared equipment. The proposed set-up, in combination with the use of appropriate cryo-protection and cryo-annealing, allowed not only the structure solution of the complete sensory domain of a cyanobacterial phytochrome at 2.45 Å resolution, but also the potential to be easily adjusted to accommodate the demands of other light-sensitive proteins.

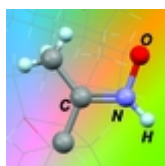
J. Mailliet, G. Psakis, C. Schroeder, S. Kaltoven, U. Dürrwang, J. Hughes and L.-O. Essen

IUCR JOURNALS NEWS

The zwitterion (23'E)-(23R,25S)-23-[1-(oxidoiminio)ethyl]-5 β -spirostan-3 β -yl acetate

The zwitterion (23'E)-(23R,25S)-23-[1-(oxidoiminio)ethyl]-5 β -spirostan-3 β -yl acetate

Acta Cryst. (2009). E65, o2954-o2955 (doi.org/10.1107/S1600536809044651)



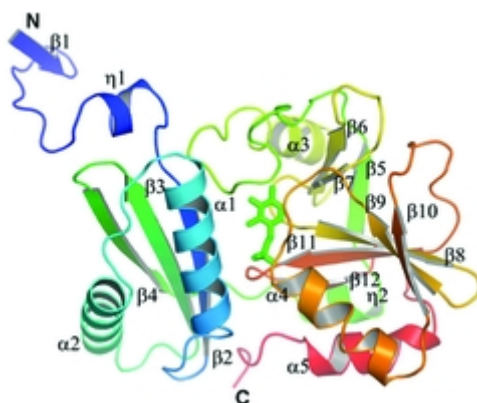
The structure of the zwitterionic oxime group as observed in the solid state by XRD.

Steroids are responsible for important biological functions in living beings. Any modification in their skeleton, functional groups, or stereochemistry may alter dramatically their biological activity. When working on the oximation of a sarsasapogenin derivative, we were aware of a non-conventional behavior of the product in acid media. Single-crystal XRD data revealed that the oxime group is indeed found in the rather rare zwitterionic form, as a consequence of the transfer of a proton from the O atom to the N atom.

M.-G. Hernández Linares, J. Sandoval Ramírez, S. Meza Reyes, S. Montiel Smith and S. Bernès

RIKEN - Daresbury - Liverpool Structural Genomics

Acta Cryst. (2009). F65, 1200-1203 (doi.org/10.1107/S1744309109043772); 1204-1208 (doi.org/10.1107/S1744309109043814); 1209-1213 (doi.org/10.1107/S1744309109044935); 1214-1217 (doi.org/10.1107/S1744309109044923); 1218-1221 (doi.org/10.1107/S1744309109046302); 1222-1226 (doi.org/10.1107/S174430910904651X); 1227-1233 (doi.org/10.1107/S1744309109047046); 1234-1239 (doi.org/10.1107/S1744309109050052)



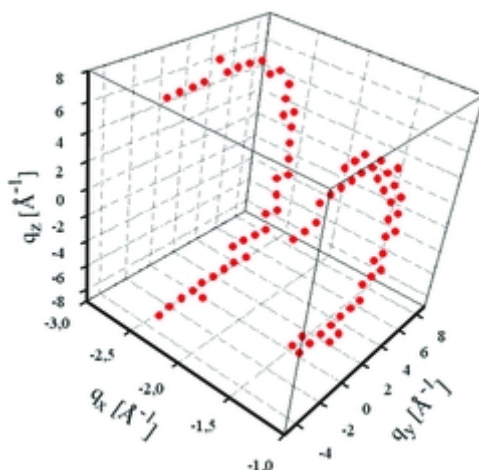
Pyridoxal 5'-phosphate (green sticks) bound to the active site of a putative 4-amino-4-deoxychorismate lyase from *Thermus thermophilus*.

On 20 January 2009 an MOU was signed between RIKEN SPring-8 Center (Japan) and the University of Liverpool (UK). In December *Acta Cryst. F* published the first fruits of this collaboration, which is extended to include the RIKEN Systems and Structural Biology Center: eight crystal structures from four organisms. A common theme in these structures was the presence at active sites of co-factors, substrate and product molecules, such as NAD^+ , NADP^+ , lactate, pyruvate and pyridoxal 5'-phosphate, all of which were endogenous to the protein expression systems.

Y. Agari, S.V. Antonyuk, Y. Bessho, A. Ebihara, M.J. Ellis, S.S. Hasnain, H. Iino, Y. Inoue, S. Kuramitsu, A.D. Malay, B. Padmanabhan, A. Shinkai, R.W. Strange, N. Watanabe and S. Yokoyama

Energy-dispersive Laue diffraction by means of a frame-store pnCCD

J. Appl. Cryst. (2009). 42, 1139-1146 (doi.org/10.1107/S0021889809039867)

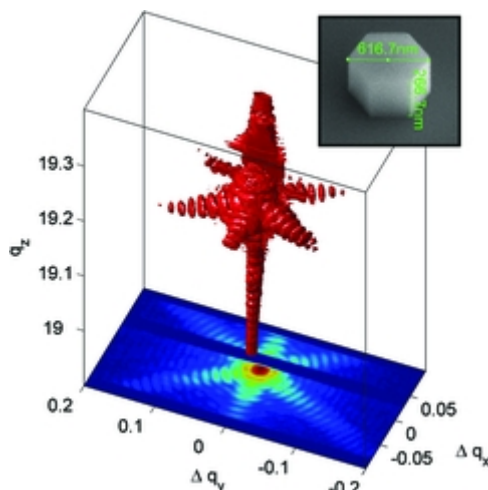


Laue pattern of γ -LiAlO₂ recorded by means of the frame-store pnCCD after conversion into reciprocal space coordinates. The measured Laue spots (red dots) occupy the reciprocal lattice of γ -LiAlO₂. Up to now the Laue diffraction method has been used to determine the symmetry of crystals based on the knowledge of Laue spot positions on a screen. However, crystal structure analyses are not possible due to the unknown energies of the Bragg reflections. The frame-store pnCCD as the first energy-dispersive CCD detector allows a simultaneous spatially and energy-resolved detection of Laue spots. As a demonstration the unit-cell parameters of a γ -LiAlO₂ crystal are determined using a single Laue pattern.

S. Send, M. von Kozierowski, T. Panzner, S. Gorfman, K. Nurdan, A.H. Walenta, U. Pietsch, W. Leitenberger, R. Hartmann and L. Strüder

Individual GaAs nanorods imaged by coherent X-ray diffraction

J. Synchrotron Rad. (2009). 16, 796-802 (doi.org/10.1107/S0909049509032889)



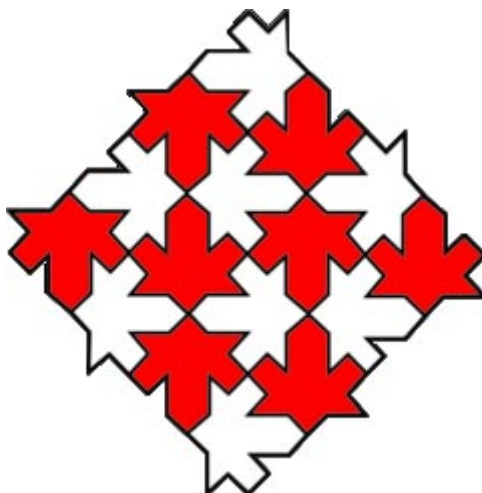
Three-dimensional coherent diffraction pattern of a single GaAs nanorod. Size, shape and strain of the individual object (inset) can be derived from the intensity distribution.

Coherent diffraction imaging in combination with a nano-focused X-ray beam was used to identify both shape and strain state of individual hexagonally shaped GaAs nanorods within a periodic nanorod array. From the three-dimensional intensity distribution around a Bragg peak in reciprocal space, differences in shape and strain of different nanorods could be resolved using phase-retrieval algorithms. The method is promising for the destruction-free analysis of nanoobjects.

A. Biermanns, A. Davydok, H. Paetzelt, A. Diaz, V. Gottschalch, T. H. Metzger and U. Pietsch

SPECIAL REPORT

Crystallography in Canada



'Feuille d'érable - p4g - Maple Leaf' by François Brisse: Logo of the Canadian National Committee for Crystallography (CNCC). This logo is based on a crystallographic artwork produced by François Brisse (then, as now, of the Department of Chemistry at the University of Montreal). This motif was also originally used for the 1981 IUCr 12th Congress in Ottawa [Brisse, F., (1981) *La Symétrie Bidimensionnelle et le Canada. Canadian Mineralogist*, Vol. 19, pp. 217-224].

We saw this Crystallography in Canada edition of the IUCr newsletter as a valuable opportunity to obtain articles of Canadian crystallographic history written by 'those who were there': scientists involved in the crystallography of olden time starting in the early 1940s to 1950s. Much of this history is not described in print and would potentially be lost if not written down or recorded in some form now or in the near future. We were more successful in this than we thought possible and obtained more articles than can be published in a single edition of the IUCr Newsletter.

While the IUCr newsletter editors requested a good balance of current *versus* historical Canadian Crystallography, it was found that historical articles had a higher submission rate. 'C'est la vie.' Information on current Canadian Crystallography can always be obtained by browsing the web, but unless historical memories and history are somehow published, it can be lost. The IUCr newsletter is an appropriate forum to help ensure much informal crystallographic history is not lost. Many of the personal accounts describe extensive education, training and research outside Canada.

- 'Some early Canadian crystallographers' by Carol P. Huber
- 'Larry Calvert (1924-1993): metals, accuracy, powder method and structure databases; a lasting heritage' by Yvon Le Page
- 'Mineralogical crystallography in Canada' by Frank C. Hawthorne
- 'For the love of computers in the 1950s' by Farid R. Ahmed
- 'Crispin Calvo' by Abraham Clearfield
- 'Early days of protein crystallography in Canada' by Michael James
- 'Eric Gabe' by Eric Gabe
- 'Osvald Knop' by T. Stanley Cameron
- 'QTAIM: quantum theory of atoms in molecules' by Richard F. W. Bader with 'Comments on QTAIM' by T. Stanley Cameron
- 'A life in science' by Andrew D. Booth
- '19th century basics spearhead 21st century progress: some autobiographic details' by Yvon Le Page
- 'Veritas vos liberabit: code of ethics in scientific work' (reprint) by Joseph (José) Désiré Hubert Donnay (1902-1994)

- 'Stanley C. Nyburg' by Stanley C. Nyburg
- 'I. David Brown' by I. David Brown
- 'A. C. Larson in Canada' by Allen C. Larson
- 'An interesting interlude with pyrolytic graphite' by Sandy Mathieson

By their variety, we hope these articles will be found informative and interesting, both to the state of current Canadian crystallography (some of it at least), and to its history. During the IUCr 2008 Congress in Osaka, we received more suggestions as to possible historical articles but these needed deferring to a later time. However, as the Montreal IUCr Congress approaches in 2014, we hope to publish more Canadian crystallographic history (currently hidden and undescribed) into the scientific light *via* the IUCr newsletter.

The Canadian National Committee for Crystallography (CNCC)

(www.canadiancrystallography.ca/)

Dedicated to Professor Louis T. J. Delbaere (1943-2009)

(CNCC committee chair, June 2005 to October 2009)

Some early Canadian crystallographers



William Howard Barnes

Probably the first Canadian-born scientist to have an important influence on the development of crystallography in Canada was William H. Barnes. Born in Montreal in 1903, the son of a physicist, he was educated at McGill University, receiving BSc, MSc and PhD degrees there, his PhD in 1927. He subsequently spent three years on Fellowships at the Royal Institution in London under the supervision of Sir William H. Bragg. While there he developed his interest in the analysis of crystal structures by X-rays, and carried out pioneer work on the structure of ice. He returned to Canada in 1930 to join the faculty at McGill University. Legend has it that he carried on low-temperature crystallography at McGill by positioning a camera outside his laboratory window in winter. In 1946 he obtained a Guggenheim Fellowship and went off to MIT to spend a year working with Martin Buerger. He came back to Canada in 1947 in order to establish an X-ray Diffraction Section in the Division of Pure Physics at the National Research Council of Canada, and he remained Head of this section until his retirement in 1968. A few years after his arrival, the NRC Post-Doctoral Fellowship program was established, enabling many young crystallographers from abroad to spend a year or two working in his laboratory. Some of them returned subsequently to their own countries to carry on crystallography there, most notably David Phillips (later Lord Phillips of Ellesmere), who went back to Britain and later became Prof. of Molecular Biophysics at Oxford. Several people from Barnes' lab later set up new crystallographic research or teaching centres in Canada, and so his influence spread across the country.

Dr. Barnes played a major role in persuading the National Research Council in 1948 to adhere to the IUCr for Canada, and was Chair of the Canadian National Committee for Crystallography from 1948 until 1966. He served very successfully as Chairman of the Organizing Committee for the IVth IUCr Congress in 1957 in Montreal. From 1960 until 1966, he was a member of the IUCr Executive Committee. His main research interests were structural studies of vanadium minerals, the use of powder diffraction to identify narcotics and drugs, and the determination of structures of drug-related compounds.



Maria Przybylska



Larry D. Calvert



Farid R. Ahmed



Al Hanson

Two young women crystallographers from Glasgow, Maria Przybylska and Violet Shore, were the first two people to receive NRC Postdoctoral Fellowships and both of them went to work with Barnes, arriving in December 1949. Both had earned doctorates in the lab of J. M. Robertson. Violet Shore went back to Britain after her fellowship years, while Maria Przybylska set up a new lab in the Division of Applied Chemistry at NRC and a few years later moved to the Division of Pure Chemistry, also at NRC, and started up another group. Larry Calvert came from New Zealand to the Barnes lab in 1952, and after his fellowship was completed, set up his own group in the Division of Applied Chemistry. This group later included Eric Gabe and Yvon LePage. Farid Ahmed joined the Barnes group in 1955 from England, where he had received his doctorate in Durward Cruickshank's lab. Barnes could foresee the important role that general-purpose digital computers could play in crystal structure determination and refinement, and encouraged Farid to develop suites of computer programs for the most advanced computers accessible to the group. Over the years, programs were written for the Ferranti FERUT, the IBM 650, IBM 1620, the IBM System 360, and the IBM 3090. Farid Ahmed remained a member of the original group until the groups in Pure Physics and Pure Chemistry merged in 1968, and eventually he was appointed head of the merged group. He served in that capacity until his retirement in 1990. Al Hanson, a Canadian who had received his PhD in Manchester, was also an early member of the Barnes group, arriving in 1955 and staying until 1978, when he left to start a structural lab in NRC's Atlantic Regional Laboratory in Halifax.



Jim Trotter



I. David Brown

Jim Trotter arrived at the Barnes lab in 1957 from Glasgow as a postdoctoral fellow, and in 1960 he went west to Vancouver, and started crystallography at the University of British Columbia. In the ensuing 38 years he trained many crystallographers in his lab there. In 1959 David Brown arrived at McMaster University from the University of London (UK), initially to help Howard Petch, an NMR chemist, build a neutron diffractometer for the reactor at McMaster. David stayed, subsequently built a crystallography group, and is still active there now.



Leonard Berry (right) with A. J. C. Wilson on the left.



Robert B. Ferguson

The 1940s also were the time that the mineralogists became active in Canada. Leonard Berry joined the faculty of Queen's University in Kingston, Ontario, in 1944 as a mineralogist and crystallographer, and remained there for 38 years. Leonard was a member of the Canadian National Committee for Crystallography from 1948 until 1970, and served as its Chairman from 1966 until 1970. Mineralogy was also represented in western Canada by Robert Ferguson who began his career at the University of Manitoba in 1947. Although he was in the Dept. of Geology, he supervised several chemical crystallography students there, including Michael James, Louis Delbaere, and me, in addition to mineralogy graduate students. Upon his retirement in 1985, he was appointed Professor Emeritus and carried on his research for some years.

Even before the days of Berry and Ferguson, J. D. H. Donnay spent from 1939 until 1942 as a professor at Laval University, and thirty years later JDH returned to Canada to be with his wife Gabrielle Donnay when she was appointed Professor of Crystallography at McGill.



Osvald Knop

The first person doing crystallography in the Maritime provinces was probably Osvald Knop, who was born in Czechoslovakia in 1922 and arrived in Halifax in 1957 after stays at Caltech and Laval University.

After 7 years at Nova Scotia Technical College he moved to Dalhousie University, also in Halifax, and remained there for many years, as an emeritus professor after retirement.

Canada can also lay partial claim to two famous crystallographers who ended up elsewhere. A. L. Patterson, although he was born in New Zealand, grew up in Montreal and received his University education at McGill. He completed his PhD work there in 1928. He left McGill, after an additional year as a lecturer, to join Wyckoff's group in New York. A. J. C. Wilson was born in Nova Scotia and received his university education there to the MSc level, which he obtained in 1936 at Dalhousie in Halifax. He apparently did not do any crystallography until his doctoral work at MIT.

I have had to employ an arbitrary cutoff limit, and have probably inadvertently missed some people whom I should have mentioned. If so, my apologies. Now there are crystallography groups in many Canadian cities, so our predecessors' efforts have borne fruit. Thanks are due to the following for providing photos and slides: Farid Ahmed and Margaret Pippy in Ottawa, Margaret James in Halifax, Tony Secco in Winnipeg, Ann Trotter in Vancouver, Marcia Colquhoun in Buffalo and Lachlan Cranswick in Chalk River.

(based on after-dinner remarks July 24, 1997; ACA conference, St. Louis, Missouri, USA)

Carol P. Huber, 1996 President of the American Crystallographic Assn

Larry Calvert (1924-1993): metals, accuracy, powder method and structure databases; a lasting heritage



1952: Larry Calvert's graduation (Courtesy Barbara Campbell).

Lauriston (Larry) Derwent Calvert (Ahmed & Le Page, 1993; Wallace & Mueller, 1993) was the son of Fred Clifford Calvert (1900-1974), a New Zealand mining engineer who worked mostly for tin dredging in Malaysia and for gold exploration or mining at many places in the southern hemisphere, registering his son at boarding schools where possible. Larry's pre-university education was accordingly garnered at a number of different schools, most of them in New Zealand, but it also included the *Suva Boys Grammar School* where he befriended sons of Fijian chiefs. At school recess time, he found employment in ordinary manual jobs, one of them cutting meat at a sheep meat-packing factory. He graduated in Philosophy from the University of New Zealand in Auckland. Awarded an MSc and then a PhD in chemistry in 1952, with Dame Kathleen Lonsdale as external examiner, Larry Calvert became the first New-Zealander crystallographer. In that year, he came to the Division of Physics of the National Research Council of Canada (NRC) as a research associate with William Barnes (Ahmed *et al.*, 1981). Two years later, as a staff assistant research officer, he started an X-ray lab at NRC's then new Division of Applied Chemistry, from which he retired in 1985 as a principal research officer. Larry had two children, Alistair and Margaret, with his first wife Marjorie (d. 1980). He re-married in 1982 with Barbara (now Campbell). They retired at Lakes Entrance in the state of Victoria, Australia where he died in 1993, not very far from Tasmania, where he was born.

Larry was a man of substance and few words, with gentlemanly education and manners. He had a very strong sense of ethics and duty. A keen and sharp observer of his surroundings, he was not a person to be fooled by fast speech or pointless authority. Always friendly in his attitude, he was very cautious and concise in his written or spoken words, often carefully selected tongue-in-cheek words, to the extent that his wise replies and sound advice might at times have to be decrypted. He would never engage in a protracted argument or volunteer unsolicited advice or harsh comments. For example, if asked privately

about an unconvincing presentation, he would use words like 'jazzy' to indicate a great presentation effort combined with modest substance, or 'jazzed-up' when the speaker had basically re-discovered the wheel. Results already presented several times before might be qualified with 're-hash'. 'Gum-flapping' would similarly convey his general evaluation of an unnecessary meeting without wasting words. If asked publicly, he would rather retreat behind a pretended 'loss of hearing due to old age' than volunteer negative comments.

As a person, Larry was characterized by a combination of a strong literary background, a curiosity for anything culturally different, a love for the southern hemisphere where he had grown up, and a sound judgment based on personal experience, on timeless wisdom and on biographies of which he was a keen reader. His approach to science and to life in general was always firmly based on facts or on time-proven observations, never far from verifiable facts. Larry was very knowledgeable about trees, plants, birds *etc.* of the Ottawa area, but his concept of a common tree remained a eucalyptus or a gum tree all along. Larry knew every illustration and every description in *The Birds of Brewery Creek* (Macdonald, 1947), but his concept of a bird was a kookaburra, a kiwi, or a parakeet. From his secondary school years in Fiji and New Zealand, he had acquired and retained all along a great interest and deep respect for the aboriginal cultures of the South Pacific. Larry also relished the company of any person from a different culture or a different background and loved to converse with them, mostly listening while sharing a brew or two, or introducing them to Koonunga Hill wines or Malay curry dishes. To settle an argument, or to show what the right direction was without wasting words, he would easily quote a Latin proverb or maxim with no ostentation: the point that the item being discussed and its time-proven solution were nothing new was instantly made. This came to him very naturally because his undergraduate education had been so strong in languages, literature and philosophy. Although he never mentioned it, Larry was a very regular blood donor as he left behind a Red Cross pin awarded to people who had given blood more than fifty times, still on its cardboard support bearing his name.



1977: Larry Calvert (below) and Bryan Taylor poring over a Debye-Scherrer pattern at the Chemistry Division of NRC in Ottawa. (Courtesy Bryan Taylor)

Scientifically, Larry focused on the basics of a few key topics, most of which are still under development today. He maintained for example up-to-date files about precision and accuracy in diffraction-intensity and diffraction-angle measurement, about absorption and extinction corrections, about incident-beam polarization, about correction for polarization by the sample, about twinning, about standard materials and similar topics. While also practicing single-crystal diffraction, Larry was a staunch and enthusiastic proponent and practitioner of the powder method. The Rietveld least-squares full profile analysis (Rietveld, 1967) was for him one of the great inventions of the 20th century that would finally give justice to the then obsolescent powder-diffraction methods. He was eventually shown right and vindicated. He triumphed when the lecture room allotted to powder methods at the XIIth IUCr Meeting in Ottawa in 1981 was chock-full with standing attendance, many from industry, marking a turnpoint in the renaissance of powder methods. Over the years, he accumulated a collection of thousands of photographic powder-diffraction reference patterns of intermetallic materials that is still kept at the NRC. He contributed nearly 1,300 new experimental powder patterns to the JCPDS, and then to the ICDD Powder Diffraction File, most of them for intermetallic materials.

Larry was an extremely careful, even meticulous experimenter, always using internal standards for example, developing his own equipment with specially designed incident-beam or diffracted-beam monochromators, focusing geometry, evacuated diffraction chambers or multi-sample holders. He spent a lot of time fine-tuning all this, never fully satisfied with the results. He designed and built an evacuated small-angle scattering camera for fiber-diffraction studies with a remarkable collimation system that is still in use at the NRC.

Larry was not a big supporter of the then-prevailing 'publish-or-perish' approach to scientific research administration. He was instead patient and very thorough in everything he undertook, with meticulous attention to detail. In spite of this, he authored or co-authored 87 quality publications, spanning from 1954 to 1992, many on intermetallic materials, in particular rare-earth pnictides or CaNi_5 -type hydrogen-storage alloys (Calvert *et al.*, 1985), way ahead of the current frenzy. His most frequent or noted co-authors were Bryan Taylor, John Murray, Don Heyding, Sandy Mathieson, Yu Wang, Eric Gabe, and Allen Larson *etc.* His models for significant research were people like E. H. Swanson from NBS, now NIST, and A. McL. Mathieson from CSIRO. Larry remained active in his retirement, as he co-edited with Pierre Villars the huge volumes of *Pearson's Handbook of Crystallographic Data for Intermetallic Phases* (1985, 1991) and wrote several articles, the last one in 1992.

Larry was Co-Editor of fourteen *Structure Reports* volumes from 1959 to 1981. The goal of those Reports was to collate succinct *edited* numerical primary data and graphical results for all crystal structures printed world-wide for that year, yet would nevertheless be so thorough that going back to the original paper would be unnecessary. Patiently cross-checking cell data, atomic coordinates, and Wyckoff positions as well as distances and angles was a monster job, often requiring querying authors internationally by steampost about typos in cell data or coordinates. A large majority of authors answered his polite and down-to-earth requests. As a consequence, his *Structure Reports* sections are actually more valuable than the original papers because they are free from typos. In other words, Larry's *Structure Reports* were the paper equivalent of edited crystal-structure database entries in those pre-internet days where even in-lab computers were a rarity.

Although not truly fluent in computer programming, Larry was always an enthusiastic supporter of quality crystallographic software, adding Eric Gabe and later myself, and then John Rodgers to the staff of the Chemistry Division's X-ray Diffraction lab. Larry frequently invited Allen Larson from Los Alamos for extended visits. This led to the automation of structure cross-checks for *Structure Reports* and to a pioneer version of crystal-structure databases, as a by-product of the automation of crystallographic data collection and its efficient and correct processing. The effort also allowed the creation of the *NRCCAD* and *NRCVAX* packages at the Chemistry Division in Ottawa, which are still in use today.

Larry assembled the Metals Data File (MDF) from the pre-1969 data that Don Cromer from LASL in Los Alamos, Bill Pearson from the University of Waterloo and himself had gathered in different formats. He integrated them and carried on with the whole post-1969 task until 1984. The initial programming by Allen Larson from LASL included such key routines as SPGP, a very compact routine to generate the general position of any space group from its Hermann-Mauguin short symbol, in standard orientation or not. This MDF software package was then much expanded by Eric Gabe, Yu Wang and Florence Lee in collaboration with Allen Larson. Data collection, extraction and input was performed by John Byron, while Larry edited it. In 1984, the MDF contained 5,600 entries, representing essentially all intermetallic crystal structures solved from 1913 to 1984. The MDF later became NRC's CRYSTMET relational database, developed with the help of John Rodgers, Pierre Villars and Gordon Wood. In those pre-internet days, where email was the fastest way to communicate, CRYSTMET was automated to answer email queries. Nowadays, CRYSTMET is a Windows package that runs on PCs and contains over 128,000 edited entries. It is up-to-date for intermetallic compounds and has been expanded to include inorganic entries as well. Not much of this would exist today without Larry's vision and relentless pioneering efforts.

Larry intensely participated in professional activities. An active member of the ACA, he served on its Nominating and Fankuchen Award Committees. He was Local Chair of the Ottawa ACA meeting in 1970 and a member of the Committee on Standards for the Publication of Powder Patterns. Internationally, he was a member of the Editorial Board of *Zeitschrift für Kristallographie*. At the IUCr level, he participated in the CNC/IUCr from 1970 to 1976, the last three years as its secretary. In addition to his involvement with *Structure Reports*, Larry was deeply involved with ICDD. He went on attending their semi-annual meetings until 1993, as Chair of the Metals and Alloys Subcommittee (Wallace & Mueller, 1993).

Larry was Local Chair of the XIIth IUCr Congress and General Assembly at Carleton University in Ottawa in 1981, with Farid Ahmed as Program Chair. This was before the times when congress centers that could professionally handle all daily activities around the meeting sprouted in most big cities. This was also before Internet and even before FAX or email became widespread internationally. Most registration items accordingly had to be handled by steampost, and all accounting was on paper. All accompanying members' activities had to be arranged months ahead of time, with a deposit. With seed money from the IUCr and from NRC, and the support of Ken Charbonneau from NRC's Congress Office, everything went smoothly. The larger-than-anticipated attendance at the Carleton University site even left a very sizeable surplus in the hands of the CNC/IUCr after returning the seed money.

With the CNC/IUCr, Larry went through the patient paperwork of creating a tax-exempt charity CNC/IUCr Trust Fund with the sole function of awarding Student Travel Bursaries to IUCr meetings from the tax-free interest gathered by the IUCr XII surplus money in the fund. This fund has since helped Canadian students to attend nearly all IUCr meetings, usually one, two or sometimes three each time. This fund was renamed the 'L. D. Calvert CNC/IUCr Trust Fund' by a unanimous vote of the CNC/IUCr shortly before Larry died in 1993.



1993: Larry Calvert retired in Australia. (Courtesy Barbara Campbell)

Retrospectively, Larry Calvert was a man with vision, attracted by the steady and often thankless effort of huge tasks with lasting value, and preaching only by example. A most careful experimenter with encyclopedic knowledge of metals and alloys, he will probably be remembered scientifically mostly for rare-earth pnictides, for his pioneering crystal-structure database efforts, for his staunch involvement in ICDD and for his role as Local Chair of the XIIth IUCr Meeting in Ottawa in 1981. A new pnictide mineral species, $\text{Cu}_5\text{Ge}_{0.5}\text{S}_4$, has recently been very appropriately named *calvertite* (Jambor *et al.*, 2007) after Larry. He left a scientific heritage of lasting value that has helped put modern crystallography on its tracks. At the personal level, he is remembered as a reserved man with a sharp sense of observation, gentlemanly education and manners, caring for others without ostentation. He will undoubtedly go on doing that for many years to come through the L. D. Calvert CNC/IUCr Student Travel Bursaries he created and funded.

Yvon Le Page

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Mineralogical crystallography in Canada

Canada has a long tradition in Mineralogical Crystallography, and several founding members of the *Canadian Mineralogist*, a major international mineralogical journal, were crystallographers. Minerals are complicated materials, and work on single-crystal diffraction has gone hand-in-hand with microbeam chemical analysis, spectroscopy, and work on the theoretical aspects of the solid state. Here, I will focus on the last 20 years with some reference to historical provenance.



Mike Fleet, U. of Western Ontario.

Mike Fleet is a geochemist, an import from Manchester (England), who has recently retired from the University of Western Ontario. Mike is interested in basic rocks which host platinum-group metals, the distribution of REES (rare-earth elements) in apatite and synthetic analogues, EXAFS and XANES of sulfide minerals, and the structure of glasses. Although much of this work is not directly single-crystal diffraction, Mike does extensive structure work to provide an atomistic perspective to his geochemical interests.



Joel Grice, Canadian Museum of Nature.



Bob Ferguson, U. of Manitoba.

Joel Grice (after whom griceite, LiF, was named) is a research scientist with the Canadian Museum of Nature in Ottawa. Joel, a native of Ontario, did his PhD with Bob Ferguson at the University of Manitoba on the structures of the Ta-Nb-oxide minerals. He made major contributions to our understanding of borate and carbonate minerals, producing structural hierarchies for the latter two groups of minerals that provide the basis for a structural and paragenetic understanding of the behaviour in rocks. Joel was Chair of the International Mineralogical Association's Commission on New Minerals and Mineral Names for eight years, and was among the top ten most highly cited geoscientists for the decade 1997-2006.



Lee Groat, U. of British Columbia.

Lee Groat is professor of mineralogy at the University of British Columbia. Lee did his doctoral work with Frank Hawthorne at the University of Manitoba on the crystal chemistry of vesuvianite. Lee's principal current interest is in the properties and occurrence of gemstones. He has organized many symposia and short courses, and has been closely involved with the current exploration for gem deposits in Canada.



Frank Hawthorne, U. of Manitoba.



Joel Grice and Frank Hawthorne, discussing crystal structures.

Frank Hawthorne [$\text{Cu}^{2+}\text{Te}^{6+}\text{O}_4(\text{OH})_2$] is Canada Research Chair in Crystallography and Mineralogy at the University of Manitoba. Frank, originally from Bristol (England), did his PhD with Doug Grundy at McMaster University on the crystal chemistry of the amphiboles. He is interested in the relation between bond topology and energetics in minerals and has developed the idea of structural hierarchies in minerals based on bond topology, and its relation to sequential crystallization of minerals, particularly from aqueous solutions. He has worked extensively on rock-forming minerals (amphiboles, staurolite, tourmalines, micas) using both single-crystal diffraction, microbeam analysis (EMPA, SIMS) and a wide variety of spectroscopic methods (infrared, MAS NMR, Mössbauer etc.), and has shown that short-range order is a major feature that controls much of the behaviour of these minerals.



Grant Henderson, U. of Toronto.

Grant Henderson, professor of mineralogy at the University of Toronto, did his undergraduate work in New Zealand and came to Canada to do his PhD with Mike Fleet at Western. Grant has worked extensively on the surface properties of minerals using atomic force microscopy, and is now involved primarily in the structure of amorphous materials, particularly glasses and liquids. He uses EXAFS, XANES, MAS NMR and Raman spectroscopies to elucidate the structures of germanate, silicate and aluminosilicate glasses to derive information on speciation, coordination and linkage.



Andy McDonald, Laurentian U.

Andy McDonald is professor of mineralogy at Laurentian University. He did his PhD with George Chao at Carleton University on the minerals of the Mont Saint-Hilaire intrusion in Quebec, and has worked on the structures of accessory minerals in alkaline rocks, describing several new minerals. Recently, he has been working on the structures of the platinum-group minerals, a broad group of economically important minerals that are the economic source of platinum-group elements, and on the crystal-chemistry of tourmaline-group minerals associated with economic mineral deposits, with a view to developing exploration indicators for platinum-group elements and high field-strength elements (e.g. Ta, Nb).



Ron Peterson, Queen's U.

Ron Peterson, professor of mineralogy at Queen's University, obtained his PhD at Virginia Polytechnic Inst. with Jerry Gibbs. Ron is interested in minerals that occur in mine waste, particularly natural and synthetic hydroxy-hydrated transition-metal sulfates. These structures are particularly sensitive to temperature and ambient humidity, and Ron has constructed apparatus to examine their stability and dehydration/hydration processes as a function of these variables.



Elena Sokolova, U. of Manitoba.

Elena Sokolova (sokolovaite, $\text{CsLi}_2\text{AlSi}_4\text{O}_{10}\text{F}_2$) is a research professor at the University of Manitoba. Elena was born in Moscow, Russia, and did her PhD with N. V. Belov at Moscow State University on the structures of synthetic borate compounds. Her primary interest is in the structures of Ti-silicate minerals where she has imposed order on a bewildering variety of very complex structures. She has worked extensively on the structures of new minerals from granitic pegmatites, particularly from Dara-i-Pioz in Tadjikistan, and the peralkaline rocks of the Kola peninsula. She also heads a major collaborative project with the Inst. of the Geology of Ore Deposits: Petrography, Mineralogy and Geochemistry of the Russian Academy of Sciences on the crystal chemistry of minerals from the Kola Superdeep Borehole, the deepest hole (12.2 km) ever drilled in the Earth's crust.

Frank C. Hawthorne

For the love of computers in the 1950s

Farid R. Ahmed, Leeds, UK, 1953



Farid R. Ahmed, and Durward William John Cruickshank, U. of Leeds, UK, 1953.

After graduating in 1950 with a BSc Honours in mathematics from the University of Leeds, I wanted to continue my study for a PhD in mathematics. However, the Mathematics Dept. told me to see E. G. Cox, head of the Inorganic and Physical Chemistry Dept. Cox introduced me to Durward W. J. Cruickshank. They explained that they would be interested in having me pursue a PhD degree in crystallography. Durward would be my supervisor, and the title of my thesis would be *Development of Mathematical Methods for the Determination of Molecular Structures by X-ray Analysis*. I agreed to give it a try.

The first year was spent learning about crystallography and Hollerith equipment (business punch-card machines in the UK) and their use for crystallographic calculations as described by Cox, Gross & Jeffrey (1949). Our use of the Ferranti Mark II electronic digital computer at the University of Manchester started soon after its installation in the summer of 1951. The article by Ahmed & Cruickshank (1953) gives some details of the computer and the crystallographic programs that we devised in that period. They were for the calculation of structure factors, differential syntheses with correction for finite summations as described by Booth, and for the estimation of errors according to Cruickshank. For maximum efficiency, those initial programs were space-group specific. However, sections of the programs could be altered easily to adapt them for other space groups.

The photograph of Durward and me was taken in 1953 after the farewell party that the Chemistry Department held before my return to Egypt. For the next two years, I taught mathematics to engineering students at the University of Alexandria.

Before 1955, the University of Toronto installed a Ferranti Mark II computer just like the one we had used at Manchester. The X-ray diffraction group at the National Research Council of Canada (NRC) decided to use that computer for their crystallographic calculations. W. H. Barnes and David C. Phillips met in Ottawa with Peter J. Wheatley, who was a crystallographer at the University of Leeds. They decided that crystallographic computing in Ottawa would benefit by bringing in someone with my experience and I received an offer to join the NRC as a postdoctorate fellow for one year. I accepted gladly because of my love for crystallography and computers. My wife Jean and I then immigrated to Canada in 1955.



A Ferranti Model No. 1 computer of the 1950s installed at the U. of Manchester, UK.

The NRC crystallographers got permission to use the Ferranti computer at the U. of T. (FERUT) as much as they needed. At that time, crystallography in Canada was already well established at a few universities and government laboratories. They did powder diffraction and single-crystal analyses of organic and inorganic compounds as well as minerals and metal complexes. Their crystals had varied space groups. It was therefore essential to devise generalized crystallographic programs, which could handle all space groups, and economize on the calculations by taking full advantage of the space group symmetries, and the corresponding structure factor and electron-density expressions, given in the *International Tables for X-ray Crystallography* (1952). To achieve that, the appropriate equations for the space groups were represented by a simple pseudo code, which directed a short interpretive routine to make the necessary changes to the main program. The article by Ahmed & Barnes (1958) gives some details of the NRC generalized crystallographic programs.



Co-authors of the NRC FORTRAN package of programs: (top left) William Howard Barnes, second Head of crystallography at the NRC (circa 1966); (top right) Sydney R. Hall (circa early 1970s); (bottom left) Carol P. Huber (photo taken in April 1997) and (bottom right) Margaret E. Pippy (photo taken July 29, 1988).



F. R. Ahmed taken on his 73rd birthday, November 21, 1997.

After FERUT was retired we used computers in Ottawa, starting with the IBM 650 then the IBM 1620, and in each case we used the symbolic codes of those computers. Finally in 1965 the NRC Computation Center installed an IBM 360. This time all our crystallographic programs were written in FORTRAN IV, by F. R. Ahmed, S. R. Hall, C. P. Huber & M. E. Pippy. That was the last rewrite of the NRC generalized crystallographic programs. These programs served us till about 1986. By that time our crystallography group became part of the NRC Institute of Biological Sciences, under the new name of Protein Crystallography Group. Our main research was now directed to the study of macromolecular structures. Computer programs for these studies were imported from other laboratories. The program *FRODO* for computer graphics was in daily use by our group.

The programs that we wrote in machine or symbolic language lasted for an average of 3.3 years, while the FORTRAN programs lasted for 20 years. The FORTRAN package was also more versatile for distribution to other laboratories in Canada and abroad. The technical advances of the last half century including powerful X-ray beams, automatic data collection with diffractometers/area detectors, and computers with phenomenal speeds, have benefited most areas of crystallography. I consider myself lucky to have witnessed these interesting developments.

I am grateful to Lachlan M. D. Cranswick for inviting me to write this article, and for his help with the Figures.

Farid R. Ahmed

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Veritas vos liberabit: code of ethics in scientific work

1. Observe conscientiously. Record faithfully. Reason coldly. Infer prudently. State your conclusions courageously.
2. When the investigation is finished, be brave and write it up. Do not 'rush into print', but know when the job is done.
3. Write clearly, with the determination of making yourself understood, not in the hope of impressing people! Look for elegance in simplicity: shun verbiage and pomposity.
4. Be honest in every statement you make. Avoid double talk!
5. Do not bluff your way out of difficulties. Keep your scientific integrity at all times.
6. Do not stop reworking your manuscript until you are sure you can no longer improve it. Watch details too: anything worth doing is worth doing well. Above all: do not expect the referee to clear up your mess for you!
7. Give credit where credit is due. Acknowledge all help you received. Give thanks with dignity. The important point is to say what the person did do for you. (A perfect example: 'Miss Ann Pletinger took all the X-ray photographs.' Bad form would be: 'I am immensely grateful to the gracious and distinguished Miss So-and-so for the invaluable help she gave me in the course of this investigation.')
8. Do not hesitate to ask your professional friends for criticism and advice, as you yourself should be willing to help them at any time. Scientific research is a beautiful co-operative adventure, not a cut-throat business. Professions in which people help one another rate high in the community, in the Nation, and in the World.
9. If you sign a joint paper, you must check the whole paper (repeat every calculation, etc.). If you are not willing or able to go through this checking, then you should refuse co-authorship, and you should sign only that section of the paper for which you are willing to be responsible.
10. When the referees' reports come in, squash your feelings, suppress your passion. Weigh and appraise. Let only your reason decide.
11. Reject the argument of authority (for ten good people can be wrong), but accept the authority of the argument (for one single judicious remark may clinch it)!

12. Have plenty of scientific humility to draw on when you start revising your manuscript. In every instance, make sure that the referee's argument is right, then accept his criticism gratefully, correct the flaw, and sincerely resolve never to make the same mistake again.
13. Be a perfectionist when reading galley proof. If the editor is willing, insist on seeing page proof too, and check that your corrections have been executed.
14. After your paper has been published, an error may be detected in it. Either you find it yourself, or someone else finds it for you. Perhaps a friend of yours writes you a nice letter about it, or a stranger publishes a note to set things straight. In any case, you should be thankful the error was discovered. You must, of course, immediately send an Erratum to the editor, in which you recognize the validity of the criticism. If somebody suggested the correction to you, it is usual to mention him by name. Avoid explaining the mistake away: your fellow scientists are not interested in excuses (they are humans like you, and they too make mistakes); all that matters is the final corrected statement. Remember that your stature as a scientist is not diminished, but actually increased, by your sending in the Errata.
15. Let a cheap politician worry about his 'image'; as a scientist, your only concern is TRUTH.

[Donnay, J. D. H. (1995). Canadian Mineralogist (33/2), inside back cover.]

(reprinted with permission of Canadian Mineralogist editor, Robert F. Martin)

Joseph (José) Désiré Hubert Donnay, born on June 6, 1902 in Grandville, Belgium, passed away peacefully at home, on the flanks of Mont Saint-Hilaire, Quebec, on August 8, 1994. The above, reprinted here with permission of the family, was first circulated to students and colleagues at the Johns Hopkins University in 1965-1966. All of us who worked closely with José know how well he lived by the above Code of Ethics. It does not seem at all out of place to reprint it thirty years later, as a memento to his high standards, and in particular to his sustained efforts in the production of this journal.

Crispin Calvo (1930-1977)



C. Calvo presents a prize (a case of Charrington Toby ale) to 'Miss Solidification' who was featured at the open house. Photo from page 86 of the Annual Report of The Institute for Materials Research, McMaster University, Hamilton, Ontario, Canada, April 1970; courtesy of Jim Britten.

Cris and I were classmates at Rutgers in the chemistry department doctoral program. Cris' project involved the synthesis and structures of polyvanadates and polymolybdates. Although he obtained many crystals, they were too complex to be solved by crystallographic methods of the time (early 1950s). We had no computers and all data were obtained by film methods. His PhD dissertation was on the crystallography of the polyvanadates. I believe that he was then a postdoc at Cornell University working on gas phase electron diffraction. He then was appointed as an assistant professor at McMaster University where he spent his entire career.

Cris' major contribution was in doing the crystal structures of pyrophosphates and phosphates of transition metals. There were high and low temperature phases and in special cases plagued with disorder and thermal anomalies in the literature associated with these compounds. Cris solved many of

their structures and later refined his own and literature data by full matrix least squares methods. Thus, he was able to clear up many anomalies and to explain the luminescence of these compounds. He systematized the field and was able to predict and synthesize new phases.

Cris and his wife Georgia had five children but unfortunately he contracted Hodgkins' disease. He refused to take the required medication because it restricted his ability to think, a condition he could not tolerate.

Cris was one of the most intelligent individuals it was my privilege to know. He could read J. Willard's Gibbs *Thermodynamics* in the original as though it was a novel and explain it to you on the spot. In character he was the 'salt of the earth'. His untimely death was a tragedy for his family, friends, and his beloved crystallography.

Abraham Clearfield

To be continued in Volume 18, Number 2.