

NIST Centennial Celebration - Crystallographic Highlights

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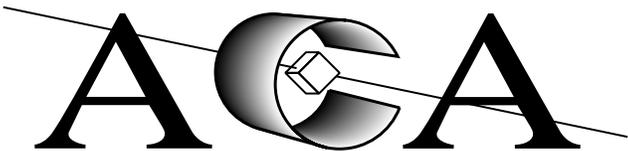
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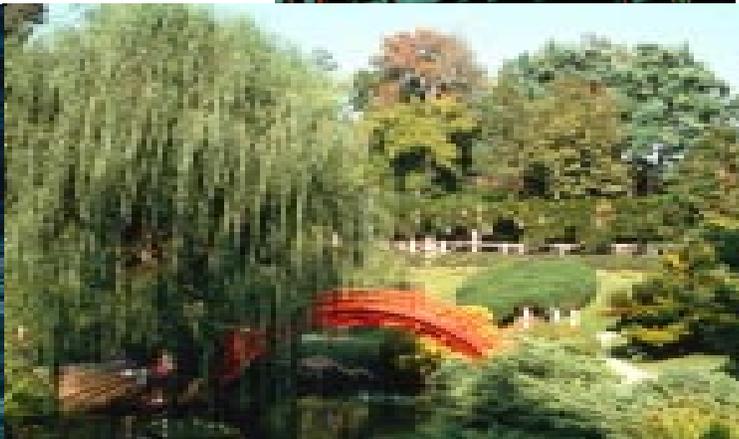
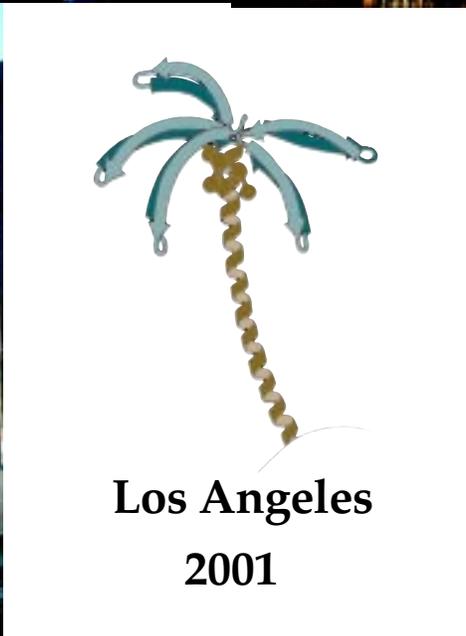


Table of Contents

President's Column 2
 News of Crystallographers 3
 Harry Brumberger (1926-2000) 3
 News from Canada 4
 Call for Award Nominations 5
 ACA Election Results 6-7
 ACA 50th Anniversary Meeting - St. Paul 8-16
 Battery Symposium 8
 Travel Award Reports 10-14
 Ortep of the Year 14
 Lyle Jensen's Buerger Award Address 15
 Protein Data Bank Update / Announcing Acta E 17
 ACA Fund Donors 18
 ACA Corporate Members 19
 What's on the Cover / Contributors to this Issue 19
 Paul Award Graphics 19
 11th Annual Southwest Macromolecular Symposium 20
 Physics Today / ECM Election Results 20
 USNCCr Meeting 21
 NIST Centennial Celebrations 22-31
 Positions Available 32
 Meetings - Schools - Workshops -Calendar 32
 Beamtime Available / Oxford Comes to North America 33
 ACA "no chad" Electronic Balloting 33
 Reduced Rate Subscriptions to JSB 33

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Articles by e-mail or on diskettes are especially welcome. Deadlines for newsletter contributions are: February 1 (Spring), May 1 (Summer), August 1 (Fall) and November 1 (Winter). Matters pertaining to advertisements, membership inquiries, or use of the ACA mailing list should be addressed to:

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President's Column



The best thing about this past year was the opportunities to interact with so many exceptionally nice people. No wonder the ACA can depend on volunteers for so many tasks.

The fall meeting of the ACA Council was held in Los Angeles so that we could look over the 2001 meeting site and talk with Local and Program Chairs. Duncan McRee is well along in planning the program, which looks exciting and very well-balanced. As for the site, the Westin Bonaventure Hotel is almost incredible to one who remembers staying in college dormitories during ACA meetings. The architecture of many of the buildings in the downtown area near the hotel is quite interesting, but the contrast between the contemporary elegance of the Bonaventure and the splendor of the Regal Biltmore, where the Awards banquet will be held, is especially striking. Art lovers can visit the nearby Museum of Contemporary Art and the excursion to the Getty Center.

In other business, the Council considered pros and cons of instituting an ACA Fellows designation to honor distinguished members, an idea that I hope may come to fruition within a year or two. Another initiative that I fervently hope will succeed in time is that of developing the ACA website as a resource for crystallographic education and research. This subject has been discussed in more detail in previous columns, but since this is my last column as President, I'm going to put in one more plug. The Council would be pleased to fund a student project to work on this and any suggestions about people who might be willing to coordinate such a project would be most welcome.

Finally, I want to express heartfelt thanks to so many of you who cheerfully said yes when I asked you to serve on committees or to help out in other ways. I knew the newsletter editors worked very hard, but I did not fully realize how much the Local and Program chairs do. Or what a tremendous boon to the smooth functioning of this organization our Buffalo office is. The ACA Council has been a cooperative and congenial group to work with and I am happy that I will continue to be a part of it for one more year. We are all especially grateful to Jane Griffin who has served ably as ACA Treasurer since 1995. She will retire from the Council next June next to allow for overlap with the incoming Treasurer. Although we will not have the pleasure of Abe Clearfield's company at Council meetings next year, he is not quite off the volunteer hook yet, because he has agreed to organize the *Transactions* symposium "Structure Solutions from X-ray Powder Data" at the San Antonio meeting in 2002. Finally, I wish to welcome to the Council our new Vice President, Charlie Carter, and new Treasurer, Doug Ohlendorf. I hope you both will enjoy this new challenge, and that you will find, as I have, that there are many personal rewards.

Connie Chidester

Crystallographers in the News

The Biophysical Society has announced the Society Awards Winners for the year 2001. The winners will present lectures at the 2001 Annual Meeting and will be recognized at an Awards Ceremony on Monday evening, February 19, 2001. The following crystallographers are among the 2001 Awardees:

Frederic M. Richards: Distinguished Service Award for service in the field of biophysics.

Jane Richardson: The Emily Gray Award for significant contributions to education in biophysics.

Helen Berman was named as one of six scientists honored by the Biophysical Society as 2001 Society Fellows. There are currently about 6,000 members of the society and, to date, only 45 have been selected as fellows.

The Rutgers' Board of Governors has named Helen M. Berman a Board of Governors Professor of Chemistry. "The Board of Governors Professorship is the highest academic honor that Rutgers can give a member of its faculty," said Joseph J. Seneca, university vice president for academic affairs. "This is an affirmation of Professor Berman's international reputation for scholarship and recognizes her dedication to the missions of teaching and research at Rutgers."

Professor John R Helliwell was recently awarded the first 'Professor K Banerjee Endowment Lecture Silver Medal' of the Indian Association for the Cultivation of Science (IACS). It was presented to him after his lecture entitled "New Opportunities in Biological and Chemical Crystallography" given at the IACS in Calcutta on the 19th September 2000.

2000 MSC Future Investigator Award Winners

Molecular Structure Corporation proudly announces the recipients of the first annual MSC Future Investigator Awards. These awards are designed to reward outstanding and promising researchers early in their careers before they have become independent investigators. The inaugural MSC Future Investigators were chosen from numerous applicants based on their scientific accomplishments in the field of structural biology, their publications, and the expectation that they will continue to contribute positively to the field of macromolecular crystallography in the future. Each of the four will receive an unrestricted \$2,500 cash award. The four winners are:

Melanie J. Bennett (California Institute of Technology) for her work on the hereditary haemochromatosis protein HFE and

its complex with transferrin receptor (TfR) that may help understanding of this iron-overload disorder. Melanie is a former Fellow of the Damon Runyon-Walter Winchell Foundation.

Jeffrey C. Boyington (LIG/NIAID, National Institutes of Health) who has recently determined the structure of the complex of the immunoglobulin-like natural killer (NK) cell receptor KIR2DL2 with its ligand, the class I MHC protein HLA-Cw3. The cytolytic activity of NK cells is tightly regulated by both activating and inhibitory NK cell receptors. This particular receptor inhibits NK cell-mediated lysis upon recognition of certain class I MHC molecules on the surface of healthy host cells. The KIR/MHC interaction is strikingly different from the interaction observed between T-cell receptors and MHC molecules and reveals a mechanism of allotypic specificity.

Patrick Cramer (Stanford University School of Medicine) who, along with colleagues solved the structure of yeast RNA Polymerase II, the central enzyme of gene expression that synthesizes all messenger RNA in eukaryotes.

C. Erec Stebbins (Yale School of Medicine) for his work on many structures including the ternary complex of the tumor suppressor VHL bound to Elongin B and Elongin C; the complex of Hsp90 with its inhibitor, geldanamycin, an antitumor antibiotic; and the bacterial GreA transcript cleavage factor, involved in regulating the processivity of RNA polymerase. He is a Fellow of the Damon Runyon-Walter Winchell Foundation

Harry Brumberger (1926-2000)

It is with deep regret that we inform you that Harry Brumberger passed away on Friday, November 10th. Harry's contributions to the small-angle scattering field over many years were of course enormous. He was one of the founders of the small-angle scattering community both in this country and abroad. Harry remained active to the end as the SIG's Member-at-Large, providing much help and wisdom in the organization of this year's SAS sessions in St. Paul. Tributes of greater length to Harry will appear later. For now, we state simply that he will be greatly missed by all who practice the art of small-angle scattering, and his family and friends will be in our thoughts and prayers.

His family has suggested that anyone wishing to make a donation in his memory should send it to the Jewish National Fund, 42 E. 69th St., New York, NY 10021, USA.

Tom Rieker, ACA SAS SIG Chair
Andrew Allen, ACA SAS SIG Vice-Chair

Canadian Institutes for Health Research (CIHR) (www.cihr.org/index.shtml)

On June 7, 2000, the Canadian Institutes for Health Research (CIHR) was officially created to replace the Medical Research Council of Canada (MRC) as the primary funding source for health research in Canada. The objective of CIHR is to excel in the creation of new scientific knowledge and its translation into improved health for Canadians, more effective health services and products and a strengthened health care system.

The Institutes will encourage interdisciplinary, integrative health research and forge a health research agenda across disciplines, sectors, and regions. Each Institute will include fundamental bio-medical research, clinical research, and research respecting health systems, health services, the health of populations, societal and cultural dimensions of health and environmental influences on health. Each mandate profiles the research areas around which the Institute can focus, develop and evolve.

List of Institutes and Description of Research

1. *Institute of Aboriginal Peoples' Health* - will support research to address the special health needs of Canada's Aboriginal people.

2. *Institute of Cancer Research* - will support research to reduce the burden of cancer on individuals and families through prevention strategies, screening, diagnosis, effective treatment, psycho-social support systems, and palliation.

3. *Institute of Circulatory and Respiratory Health* - will support research into causes, prevention, screening, diagnosis, treatment, support systems, and palliation for a wide range of conditions associated with the heart, lung, brain, blood and blood vessels.

4. *Institute of Gender and Health* - will support research to address how sex (biological factors) and gender (socio-cultural experiences) interact with other factors that influence health to create conditions and problems that are unique, more prevalent, more serious or different with respect to risk factors or effective interventions for women and for men.

5. *Institute of Genetics* - will support research on the human genome and in aspects of genetics related to human health and disease, including interaction of genes with physical and social environments.

6. *Institute of Health Services and Policy Research* - will support research to address the need for health systems, technologies and tools to promote health, and to prevent disease, and to deliver healthcare effectively for all sectors of the Canadian population.

7. *Institute of Healthy Aging* - will support research to promote healthy aging and to address causes, prevention, screening, diagnosis, treatment, support systems, and palliation for a wide range of conditions associated with aging.

8. *Institute of Human Development and Child and Youth Health* - will support research to enhance maternal, child, and youth health and to address causes, prevention, screening, diagnosis, treatment, short- and long-term support needs, and palliation for a wide range of health concerns associated with reproduction, early development, childhood, and adolescence.

9. *Institute of Infection and Immunity* - will support research to enhance immune-mediated health and to reduce the burden of infectious disease, immune-mediated disease and allergy through prevention strategies, screening, diagnosis, treatment, support systems and palliation.

10. *Institute of Neurosciences, Mental Health and Addiction* - will support research to enhance mental health, neurological health, vision, hearing, and cognitive functioning and to reduce the burden of related disorders through prevention strategies, screening, diagnosis, treatment, support systems, and palliation. Associated research will advance our understanding of human thought, emotion, behaviour, sensation (sight, hearing, touch, taste, smell), perception, learning and memory

11. *Institute of Musculoskeletal Health and Arthritis* - will support research to enhance active living, mobility and movement, and dental health; and to address causes, prevention, screening, diagnosis, treatment, support systems, and palliation for a wide range of conditions related to bones, joints, muscles, connective tissue, skin and teeth.

12. *Institute of Nutrition, Metabolism and Diabetes* - will support research to enhance health in relation to diet, digestion, excretion, and metabolism; and to address causes, prevention, screening, diagnosis, treatment, support systems, and palliation for a wide range of conditions and problems associated with hormones, the digestive system, kidney and liver function.

13. *Institute of Population and Public Health* - will support research into the complex interactions (biological, social, cultural, environmental) which determine the health of individuals, communities, and global populations and how that knowledge can be applied to improve the health of both populations and individuals.

Dr. Alan Bernstein has been named as the first President of CIHR and Chair of the nineteen-member Governing Council. A Scientific Director and an Advisory Board for each Institute should be determined by January 2001. Very significant increases in health research funding are expected from CIHR, as compared to that provided previously by MRC.

2002 Patterson Award

The next presentation of the A. L. Patterson Award will be made at the San Antonio, TX, meeting of the American Crystallographic Association in May, 2002. The A. L. Patterson Award, established in 1980, is to recognize and encourage outstanding research in the structure of matter by diffraction methods. This may include one or more of the following: (i) significant contributions to the methodology of structure determination, (ii) innovative application of diffraction methods, (iii) elucidation of biological, chemical, geological or physical phenomena using new structural information. There are no restrictions as to nationality, race, sex, religion, or ACA membership. The award is given in memory of Arthur Lindo Patterson, Senior Member, Institute for Cancer Research, Philadelphia, whose most important contribution to crystallography was the function named after him. Previous recipients were: Wayne A. Hendrickson, 1981; Jerome Karle and Herbert Hauptman, 1984; David and Liselotte Templeton, 1987; Michael Woolfson, 1990; George Sheldrick, 1993; Christer E. Nordman, 1997; and Gérard Bricogne, 1999. The monetary portion of the Award has been as much as \$3000.

Nominations are invited from throughout the crystallographic community. The major contributions of the nominee should be identified, with supporting documentation. The closing date for nominations is March 31, 2001; earlier nominations would be welcomed. The members of the 2002 Patterson Award Committee are:

Phillip Coppens, Dave Duchamp, Jane Griffin, Andy Howard

Nominations and supporting documents should be sent, in writing, to the ACA office in Buffalo, 73 High Street, Buffalo, NY 14203-0906.

2001 MSC Future Investigator Awards

Molecular Structure Corporation has created a new and exciting award designed to promote and foster excellence in research from young structural biologists. To be eligible to participate, the entrant must be currently employed as a postdoctoral fellow in the field of macromolecular crystallography with the intent to remain in the field on a permanent basis. Participants must also have received a Ph.D. no earlier than 1996. The awards are open to applicants from anywhere in the world.

Four unranked MSC Future Investigator Awards will be announced shortly following the ACA meeting in 2001. Each award carries an unrestricted cash prize of \$2500. To apply for the award, send a cover letter, CV, letter of recommendation, and copies of two recent publications to Dr. James Pflugrath, Molecular Structure Corporation, 9009 New Trails Drive, The Woodlands, TX 77381 USA. The deadline for submission is April 1, 2001. A committee will select Future Investigators based a number of criteria, including the applicant's potential to make a positive impact on the field of structural biology. Information can also be found at our website: <http://www.msc.com/msc/awards.html>

The European Crystallography Prize

The European Crystallographic Association (ECA) invites nominations for the second European Crystallography Prize to recognize a significant achievement or discovery in crystallography in the past 5-10 years. Nominees should be affiliated or identified with the European crystallographic community, as broadly defined in the charter of the ECA (see the ECA-news site <http://www.ba.cnr.it/eca/>).

The prize, including a monetary award and certificate of recognition, will be awarded at the opening ceremony of the 20th European Crystallography Meeting (ECM-20) to be held in Krakow, Poland, 25-31 August, 2001.

The first prize was awarded to Professor Ada Yonath of the Weizmann Institute of Science in Israel at the ECM-19 in Nancy, France in August, 2000 for "her pioneering achievements in structural studies on the ribosome".

Nominations for the prize should include a statement of the contribution for which the prize is to be awarded, a 2-3 page curriculum vitae of the nominee and the signature of at least three additional nominators, preferably with supporting nominating letters. They should postmarked no later than February 28, 2001 and should be sent to:

Professor Davide Viterbo, Dip. di Chimica IFM, Università di Torino, Via P. Giuria 7, I-10125 TORINO, Italy. Fax: +39-011-6707855, E-mail: viterbo@ch.unito.it

ICDD Grant-in-Aid Program

The International Centre for Diffraction Data (ICDD) has increased grant-in-aid funding for the 2000-2001 grant cycle in order to allow more grants to be supported.

The ICDD is interested in obtaining high quality experimental powder diffraction patterns to add to its internationally renowned database, the Powder Diffraction File (PDF). The ICDD's Grant-in-Aid Program is designed to give limited financial support to those institutions interested in supplying new patterns. A grant can be used most effectively as supplement to existing funded projects involving the preparation and characterization of new materials using X-ray powder diffraction.

There are two grant cycles with proposal deadlines of:

Cycle I 31 January

Cycle II 31 July

For more information, please review the guidelines found on the ICDD web site, <http://www.icdd.com>, or contact: Ms. Shelley Wolkov Grant Coordinator International Centre for Diffraction Data 12 Campus Boulevard Newtown Square, PA 19073-3273 U.S.A., (610)-325-9814, Fax: (610)-325-9823 E-mail: wolkov@icdd.com

ACA Election Results*New Council Members***Vice-President** *Charles Carter***Treasurer** *Doug Ohlendorf**New Standing Committee Members***Communications***John Sack (3 year term)**Jeannet Krause-Bauer (4 year term)***Continuing Education***Winnie Wong-Ng (3 year term)**Philip Fanwick (4 year term)**Special Interest Groups***Biological Macromolecules**

Chair-elect

*Cele Abad-Zapatero***Small Molecule**

Chair-elect

*John Bollinger***Fiber Diffraction**

Chair

Secretary / Treasurer

*Dan Kirschner**Barry Farmer***Young Scientists**

Chair-elect

Secretary / Treasurer

*Nicholas Silvaggi**Johanna Mazlo***General Interest**

Chair-elect

Member-at-large

*Gloria Borgstahl**Judy Flippen-Anderson**Canadian Division Election Results***Neutron Scattering**

Chair-elect

Secretary

*Simon Billinge**James Richardson***Chair***Gary Enright***Secretary***Brian Patrick***Service Crystallography**

Chair-elect

Secretary

*Paul Boyle**Michael Carducci***Small Angle Scattering**

Chair-elect

Secretary / Treasurer

*Paul Butler**Peggy Cabe*

New ACA Vice-President – Charlie Carter



This year's newly elected vice-president is Charlie Carter. Charlie received his B.A. degree in Molecular Biophysics at Yale and his Ph.D. degree from the University of California at San Diego. There he solved the structure of the high potential iron protein in Joe Kraut's laboratory. After doing a postdoc at the MRC in Cambridge in Aaron Klug's laboratory, he returned to the US and took a position at the University of North Carolina at Chapel Hill in 1974.

I first met Charlie in 1988 when I was a student at the University of Paris. He was on sabbatical at the LURE synchrotron and he quickly convinced me that aminoacyl-tRNA synthetases were fascinating enzymes, and that I had to join his group to work on the structure determination of the tryptophanyl-tRNA synthetase (TrpRS). This structure proved to be particularly tough to crack. The key to the structure determination relied heavily on the novel use of phase permutation and Bayesian phase determination methodology, which was done in collaboration with Gérard Bricogne and Chris Gilmore. This process implemented Gérard's Bayesian paradigm for solving macromolecular structures, and rescued phases for the TrpRS structure from an otherwise crippling lack of isomorphism.

Charlie's research has been on the cutting edge of method development and the TrpRS project exemplifies his crucial contributions to the crystallographic community. They range from the use of incomplete factorial designs to quickly identify crystal growth conditions, and improve on them when crystals have been obtained, to the use of maximum entropy solvent flattening and Bayesian statistics to obtain better macromolecular phases. I have very fond memories of dinners with Charlie and Gérard, where invariably by the end of dinner numerous equations had been jotted down on a multitude of paper napkins.

Charlie has been a member of the ACA for 25 years and he has a long record of service. He was program chair of the 1994 ACA in Atlanta. He has been a member of the US National Committee for Crystallography. Also, he and Bob Sweet co-edited two volumes of *Methods in Enzymology in Macromolecular Crystallography* that quickly became classics. Charlie embraces the study of matter in the broadest sense. His enthusiastic love of science encompasses macromolecular and small molecule crystallography, as well as enzymology and molecular biology. We can look forward to reinforced links between the ACA and other professional organizations, such as the IUCr, as Charlie really is a scientific citizen of the world. The ACA is indeed very fortunate to have him as its new Vice President. Please join me in welcoming him to this new position.

Sylvie Doublié

New ACA Treasurer - Doug Ohlendorf



Doug Ohlendorf is a faculty member in the Department of Biochemistry, Molecular Biology and Biophysics at the University of Minnesota in the Twin Cities. He was program co-chairperson for the year 2000 ACA meeting in St Paul. Doug has always been an active ACA member and is currently on the editorial board of the *J. of Applied Crystallography*.

Raised in southern Illinois, he won a scholarship to CalTech and spent his freshman year in Pasadena. Doug left there after his 1st year having missed the mid-western climate. For the remaining years as an undergraduate he studied in the Physics Department at Washington University in St. Louis, Mo. In four years, Doug graduated Summa cum laude with both a B.S. and an M.S. His master's thesis was in crystallography dealing with the use of computations to simulate molecules and living processes. He then joined the graduate program in biochemistry at Washington University. A true physicist, at that point he had taken but a single course in biology. His Ph.D. thesis involved image reconstruction of a crystalline lipoprotein.

Doug did post-doctoral studies at the Molecular Biology Institute - University of Oregon. In addition to his work in protein crystallography, Doug used model-building techniques to propose one of the earliest models for protein:dna interactions. He then became a Senior Research Scientist at the Genex Corporation in Maryland. One of the earliest biotech companies, the group was one of the first in protein engineering. They were also involved in the pioneering development of software for one of the early area detectors. He later moved to take a principal investigator position at DuPont in Delaware. At DuPont he solved the crystal structure of protocatechuate dehydrogenase 3,4-dioxygenase and hence began his interest in metallo-enzymes. Wanting to return to academics, he joined the faculty at the University of Minnesota in 1991 and is currently a full professor. Carrying on his structural studies of metalloproteins, he also developed a research effort aimed at understanding the structure/function relationships of super antigens and other virulence factors in gram positive pathogens.

In addition to his faculty position, he is a fellow of the Minnesota Supercomputer Institute. He is an active member of a number of professional societies and has served as a reviewer for several granting agencies.

Doug Ohlendorf is a talented musician playing the bassoon, sax, piano and organ. He is married to a scientist (Dr. Cathy Earhart). He is an avid skier - a product of those good old Keystone meetings. In Minnesota besides his science and crystallography, he has taken to being an active party member in a certain (unspecified) political party.

Len Banaszak



ACA Presidents (past and present) gather to cut the ACA 50th Anniversary cake during the river cruise in St. Paul - July 2000. Back row: Penny Coddling (1998), Connie Chidester (2000), Jenny Glusker (1979), Charlie Bugg (1987), Helen Berman (1988), Jon Clardy (1997), Judy Flippen-Anderson (1991) Bill Busing (1971), Bill Duax (1986), Carroll Johnson (1977) and Bryan Craven (1989). Front row: Betty Wood (1957), David Templeton (1984), Robinson Burbank (1975), Sidney Abrahams (1968) and Abe Clearfield (1999).

Symposium 1.01: Battery Materials: Amorphous Carbons & Polymer Electrolytes



Symposium Speakers

The motivation for research in battery materials lies in the expanding consumer demand for compact, high-energy density power sources for portable electronic devices, and environmental issues such as global warming and air pollution that have provided the impetus for mass transportation by electric vehicles.

The Battery Materials Symposium, chaired by Jacqueline Johnson (ANL) focused on three topics: the structure and electrochemical properties of new and existing electrolytes, devices for fabricating and investigating thin films, and large-scale computer simulations.

The symposium opened with a presentation by Rex Gerald

(Argonne) on a recently invented device for *in situ* investigations of batteries using nuclear magnetic resonance. It is well known that the transference number for Li ions in polymer electrolytes is well below the optimum value of unity. A salient consequence of a low transference number for the electroactive ion is the formation of a thin film depleted of ions at the reducing electrode, which is equivalent to an internal open circuit in the battery. He showed a series of magnetic resonance images that followed the evolution of the ion depletion zone from which transport numbers of specific species could be obtained. Nuclear spin relaxation measurements, related to molecular dynamics of the anions, were recorded at different positions within the ion concentration gradient of the depletion zone. These measurements revealed a direct correlation between localized molecular dynamics in the polymer electrolyte and the local concentration of ions.

Joop Schoonman (Delft University) described several methods for preparing and analyzing thin films made of solid electrolytes. These methods included chemical vapor deposition, electrostatic spray deposition, and the Solufill process. Thin film technology is critical for reducing cell resistance, increasing conductivity in ceramic electrolytes, and extending cell life at elevated operating temperatures. Some of the most interesting results presented showed a lithium-ion battery comprised of electrode and electrolyte layers wound and compressed by several compacting methods developed and used by Schoonman. Dynamic compacting by munitions and magnetic forces were compared. The substantially higher impact forces afforded by munitions do not seem to alter the initial nano-structure of the composite materials, and the performance of these materials is superior to composites fabricated by other approaches.

Aiichiro Nakano discussed large-scale (10 million to 2 billion

atoms) computer simulations of polymer and ceramic systems. A unique strain curve was produced for PEO along with the simulation of oxidation. An overview was given of a DOE Cooperative Research 2000 program, in the initial stages, that was set up to pursue these atomistic simulations. Details were given for standard computational methods, and the atomic motions in a cube of a silicon nitride (Si_3N_4) based composite of side length 0.28mm were shown following point impacts to show future possibilities for this system.

Doug MacFarlane (Monash University) described conductive plastic crystals based on pyrrolidinium imides. A conductive matrix is made of alkylmethylpyrrolidinium imide cations and bis(trifluoromethane sulfonyl) imide anions doped with lithium cations from a lithium salt with the imide anion in common. The plastic crystal behavior is thought to arise from thermal rotation of the pyrrolidinium cation about an axis that bisects the pyrrolidinium ring. Longer alkyl chains on the pyrrolidinium cation correlate with larger ionic conductivities in a lithium-doped matrix. This can be several orders of magnitude larger than the undoped matrix and can reach values near $10^{-2} \text{ S cm}^{-1}$ at 100°C . The fraction of ionic current carried by the lithium cations in the plastic crystal matrix is not known. It is presumed to be large based on the observation that the ^7Li NMR linewidth decreases with increasing temperature.

Joseph Pluth (U of Chicago) presented his recent crystallographic studies of Pb compounds found in the ubiquitous lead-acid battery. He showed the structures of tribasic lead sulfate ($3\text{PbO}\cdot\text{PbSO}_4\cdot\text{H}_2\text{O}$) and tetrabasic lead sulfate ($4\text{PbO}\cdot\text{PbSO}_4$). Striking structural similarities noted between the reactant (-PbO) phase and the phases of the sulfate products were attributed to the substitution of Pb atoms by sulfate groups. Lead atom vacancies in PbO_2 were studied in a series of samples that were cycled by conventional and rapid charge methods. Changes in the structural parameters of (-PbO) were correlated with the cycle life of the battery, and evidence was given for substitution of Pb^{+4} by 4H^+ at the center Pb atom in the (-PbO) cell. These investigations were conducted to obtain basic knowledge of the chemistry and phase changes in lead-acid battery materials in order to improve battery performance for potential applications in electric vehicles.

Austen Angell (Arizona State Univ) discussed the general problem of electrolyte polarization in Li-ion battery systems with cation transference numbers less than unity. He explained the use of polyanions, which have a lithium ion transference number of unity, but cautioned that the room temperature conductivities of known systems are too low. Alternatively, polymer-in-salt electrolytes are based on a predominant salt component, such as LiAlCl_4 , with a small amount of polymer to provide a rigid host matrix. The salt is designed to have a large cation-decoupling index. That is, the cation is weakly bound to the anion and moves independently in the host matrix. He also discussed giant anions, which effectively increase the Li+ transference number because of the very slow anion mobility. He did caution that there was a limit to the effectiveness of this approach due to the synthetic complexity of very large anions.

Steven Greenbaum (Hunter College) provided an introduction to NMR interactions that are useful for investigations of lithium-

ion battery materials. Analysis by NMR is nuclear specific, probes local environments and dynamics, and is non-destructive. He discussed ^7Li NMR results of a solid electrolyte system composed of LiI dissolved in PEO. The crystalline phase in this electrolyte was suppressed by incorporating a small amount of Al_2O_3 . The ^7Li NMR spectra of a LiI/ Al_2O_3 mixture were recorded to elucidate the role of Al_2O_3 in the electrolyte, and showed two lithium environments. One resonance corresponded to LiI and the other was assigned to Li^+ at the Al_2O_3 interface. Notable interest was generated by a series of ^7Li NMR, x-ray diffraction, and conductivity measurements on a uniaxially stretched sample of $\text{LiClO}_4/\text{PEO}$ electrolyte. The conductivity parallel to the elongated dimension was enhanced by a factor of 5-16 depending on temperature. NMR spectroscopy revealed local order about the Li^+ ions, and X-ray diffraction showed long-range order in the sample. This evidence suggests that the PEO helices are aligned in the direction of greatest conductivity. The immediate implication of these results is that the helices may be ion mobility directors.

Work on oriented polymer electrolyte samples is ongoing. Yuri Andreev (U. of St. Andrews) gave a historical overview of a number of crystal structures of polymer electrolytes solved using XRD in the group of Peter Bruce. The Rietveld method was discussed in the context of solutions to crystal structures for $(\text{PEO})_3:\text{NaClO}_4$, $(\text{PEO})_3:\text{LiCF}_3\text{SO}_3$, $(\text{PEO})_4:\text{MSCN}$ ($\text{M} = \text{NH}_4, \text{K}, \text{Rb}$). In some cases, solutions of these crystal structures by the Rietveld method were not satisfactory, and for this reason Andreev and coworkers developed the simulated annealing method, an approach based on first principles. The simulated annealing method was successfully used to solve the crystal structures of $\text{PEO}:\text{NaCF}_3\text{SO}_3$, $(\text{PEO})_3:\text{LiN}(\text{CF}_3\text{SO}_2)_2$, $\text{PEO}:\text{KCF}_3\text{SO}_3$, and the most complex structures to date, $(\text{PEO})_6:\text{LiAsF}_6$, $(\text{PEO})_6:\text{LiPF}_6$, $(\text{PEO})_6:\text{LiSbF}_6$. The latter complexes have an unusual structure that consists of individual PEO chains that form half-cylinders. A pair of half-cylinders encircles a chain of hexafluoro arsenic anions. The lithium cations are penta-coordinated by two PEO half-cylinders leaving a single oxygen atom nearby that is not involved in the immediate coordination sphere. The conductivity of the crystalline phase of $(\text{PEO})_6:\text{LiSbF}_6$ at room temperature is larger than in the amorphous phase of the same composition. The last speaker of the symposium was Peter Papanek (U. of Pennsylvania). He spoke about various disordered carbon materials used as anodes in Li-ion batteries. These materials are composed of randomly stacked, flat graphene sheets with hydrogen-terminated edges, and it has been widely reported that Li uptake increases with hydrogen content in disordered carbons. Intercalated lithium is reversibly bound to both sides of the graphene sheets and at the edge carbons. He also described his inelastic neutron scattering studies of carbon materials derived from pyrolyzed epoxy novolak resins. His data supports the graphene sheet model and is also consistent with calculations of interior and edge carbon atoms in pyrene that chemically reacted with lithium.

Rex E. Gerald

ACA Travel Awardees

We (Ron and Judy) asked recipients of ACA travel awards to send in a short note telling what receipt of the grant meant to them. It's obvious from the following comments that donations to the ACA student travel funds are wise investments.

Joanna Clark, University of Nebraska



Every day of the meeting held either interesting talks or fun events to attend. The sessions related to use of synchrotron facilities around the country were very informative. It was useful to learn about what is happening in terms of small molecule studies at synchrotrons, and find out how prospective users can obtain access to the beamlines. I enjoyed the poster presentations at the meeting, although my poster presentation was on the night of the boat ride, so there was a somewhat slim audience to discuss it with. Another nice aspect of the meeting was that I met several people from both academia and industry, and was able to discuss the differences in the two career environments. I was strongly encouraged to try proteins instead of large (small) sugar molecule complexes (by many protein crystallographers)! I'm thinking about it. The employment outlook session put on by the Young Scientist SIG was also full of valuable information. Speakers that were selected for the panel were from various diverse workplaces.

Minnesota is a nice state and was a good location for the meeting. I'm a Peanuts lover and got to see several different Snoopy statues around town. For me, the highlight of the meeting was the banquet. I enjoyed hearing from such impressive speakers—those so important to the entire field of crystallography. I am always surprised by the friendly people at ACA meetings in particular, because it's not like that at every meeting you go to. Overall, I really enjoyed this meeting and I am grateful to the ACA for helping with the expenses.

Bruno Deme, Institut Laue-Langevin, Grenoble

The grant helped my participation for it exactly covered my travel expenses from Europe, which I tried to reduce as much as possible for that period of the year. The waiving of the participation fees by the chairman of the session where I was invited was also appreciated.

I found very interesting parallel sessions 8.01, 8.02, and 5.01 on Sunday 23 and Monday 24, which are close to my research and instrumentation activities. The organization was perfect and the time given to speakers was well balanced. The River Centre in St Paul is a good place to welcome meetings of that size.

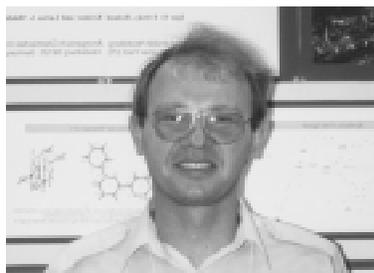
So I personally enjoyed the meeting and would like to thank the ACA for giving me this grant.

Florian Schubot, University of Georgia

I thought that accommodation and lecture halls were excellent. The wealth of extracurricular activities, especially the boat trip, was impressive and provided plenty of opportunities to meet people. My section on the solution of problem structures was to me the most interesting session. Since the common denominator of the ACA meeting is crystallography rather than molecular biology, I think there should be more sessions on that aspect of people's research.

The poster sessions were well organized and I enjoyed the more in depth discussion with the people that were presenting their work. Overall the meeting had an inspirational effect on me and made me eager to go back to work and apply some of the new ideas to my own research. Thank you again for your generous support, which allowed me to attend this meeting.

Igor Fritsky, Universität Heidelberg, Germany



It was my first ACA meeting, and my first participation in an American meeting at all. A grant generously rendered by the ACA really helped me to come to Minnesota; it covered nearly all travel expenses.

In St Paul I presented a poster. As for organization and scientific program, I think it was excellent without any reservations. I was quite surprised by the extent of macromolecular crystallography in the meeting. I found only few lectures and posters dedicated to small molecules. It is not like in Europe: in August 1998 I participated in XVIII European Crystallographic Meeting in Prague, and there was only a section (covering maybe one fourth of total amount of presentations) dedicated to protein crystallography. For me personally, it was very important to participate in such a meeting, for I am "on the way" from small to large molecules. My current research in the field of Bioinorganic Chemistry is related to hydrolases (in particular, alkaline phosphatase) and their low molecular weight structural and functional models.

I should also mention the very high level of artwork in both lectures and posters in the meeting. The meeting exhibitions provided direct contact with companies working in the field. I found, in particular, very interesting and helpful information about new software from Cambridge Crystallographic Data Centre, the program 'Conquest'. It has a lot of new features as compared to older software, and after the Meeting I started to use it successfully at my home university. The ACA meeting provided a lot of opportunities to communicate with people and to make new contacts. I was very happy to meet American scientists and

to communicate with them personally. Some of these acquaintances could be helpful in my future research collaboration: in particular, I met Dr. Ewa Skrzypczak-Jankun, Research Assistant Professor from the University of Toledo, OH, and Dr. Philip J. Squattrito from the Central Michigan University. I hope to collaborate with these people in the nearest future. I also met young scientists - grantees like me and understood that the ACA grants facilitated their coming as well.

In conclusion, I should mention that it was my first visit to the USA and my first participation in an American meeting. Honestly, I am fascinated by this country and by very high level of research in the field of crystallography presented by the American scientists. I came back from Minnesota filled with new ideas and new knowledge. I hope this will be helpful in my future research.

I thank once again the ACA for its generous support, which made possible my participation at this wonderful meeting.

Jeff Habel, University of Toledo



Once again the ACA was able to orchestrate another excellent summer meeting. Thankfully I was able to attend the entire meeting solely because of the travel award given to me by the ACA. Without the award I would have had a very difficult time presenting my new research, chairing my sessions, and presiding over the always enjoyable YSSIG social gatherings. The meeting was filled with so many interesting talks this year that I sometimes had a difficult time bouncing back and forth between rooms. My personal favorite was Wim Hol's amazing drug design talk. I hope that everyone had a chance to hear that talk in particular. Even with all the amazing structure presentations going on, part of me was missing some more pre-diffraction pattern sessions that maybe weren't geared for high throughput screening. All in all, it was an amazing meeting filled with seeing old friends and making new ones. I hope that everyone enjoyed it as much as I did. Thank you again ACA for making this trip possible for me.

Audray Harris, University of Alabama



I am truly grateful that I was able to attend the national ACA meeting in St. Paul, Minnesota for this year of 2000. I was delighted to be present at the 50th anniversary of the organization. Without the generous student travel grant that I received it would have not been possible for me to travel to the meeting. Not only did I enjoy the scientific meetings and talks, but also I enjoyed meeting and speaking with other scientists.

Faculty Advisor Virginia Pett and Andrea Jorjorian, College of Wooster



I can not express in words what a wonderful opportunity this was for me. As an undergraduate student, the travel grant provided me with a rare opportunity to attend a professional meeting. Due to monetary constraints, I would not have been able to attend without the aid of the travel award. The feedback that I received from my poster presentation was incredible. Everyone was very supportive of my research efforts and offered valuable suggestions for future research. Also, prior to attending the ACA meeting I had been planning to apply to medical school. However, when I returned from the meeting I realized that I was not yet ready to give up research and I am now planning to pursue some sort of graduate work in the biomedical field when I graduate this spring. I also want to mention that I found the workshop on the Cambridge Database very informative and useful. We have just setup the database at our school and I now feel fully capable of using the database to its full potential. I can not thank the ACA travel award committee enough for the opportunity they provided me. My experiences at the meeting were invaluable and my recent decision to continue with research as opposed to attending medical school was greatly influenced by my attendance at the ACA meeting. Everyone that I met was friendly and genuinely excited about their work. The atmosphere was highly conducive to learning. I hope that this brief note gives you some insight into my experiences at the ACA meeting and once again thank you for the travel award.

D. Kumaran, Brookhaven National Laboratory



I presented some interesting results in the ACA meeting (Poster/Abstract # P116). Many of the participants showed interest in this poster.

The talks were fascinating and very interesting. I enjoyed all the lectures delivered by experts, particularly in Genomics, Drug Design, Hot New Structures, Ultra-High Resolution Macromolecular Crystallography, Enzyme Mechanism, Synchrotron Data Collection, High Throughput Crystallization and HHMI contributions to macromolecular science. I learned a lot from this meeting.

The meeting was arranged very nicely. I really enjoyed the conference location and Annual Cruise. Thanks a lot to the ACA organizers and experts. I hope we will meet in Los Angeles.

Yongjae Lee, SUNY, Stony Brook



It wasn't until late March that I decided to attend the ACA2000 annual meeting in St. Paul. I had been involved in some "interesting" powder diffraction experiments, and some of the results started to be shaped for the presentation by that time. I submitted the abstract during the extended abstract submission term, and even requested for the student travel grant, explaining why I did this after the deadline for the submission. Fortunately and surprisingly, both my abstract and request for the student travel grant were accepted, and close to the meeting date, I got invitation letters for 5-min poster presentation and 10-min talk in New Science Using New Neutron Sources and Instruments session.

This meeting was the second of my ACA meeting experiences. In both meetings I could find only few colleagues dealing with the similar topic, zeolites and dynamic powder diffraction (<http://sbmp32.ess.sunysb.edu/yongjae.html>). This of course has advantages as well as disadvantages. At first glance, I may find myself in a wrong place where macromolecules dominate "the crystallography" (and some people complain about this). On the other hand, it is a nice chance to be stimulated in another environment where I can design my version of experiments/analyses (through the SHELX workshop and programs in General Interest sessions, in particular). I believe many young scientists, including myself, also enjoyed the flavor of the variety of crystallography through the Poster Preview session, and thank Jeff Habel for organizing this session so well.

At the end of this meeting, I was invited to the annual banquet via the Pauling Poster Prize and saw the history of the ACA. This meeting was more than generous to me, and now I'm working to meet you again in next year's meeting in LA.

Raquel Lieberman, Northwestern University



As a graduate student who is relatively new to the field of crystallography (I solved my first structure a few days short of the new millennium), I was particularly struck by the breadth of scientific research that utilizes the technique of crystallography. From small-molecule to macromolecule crystallography, through technical advances in synchrotron radiation, I was pretty stunned by the number of different lenses through which scientists see crystallography (and a little overwhelmed). I mostly attended lectures dealing with macromolecular crystallography, where I not only learned more about the techniques, but also got a sense of the hot structural biology research interests, especially relating to drug design and cell biology. Who would have predicted that a crystallography conference could double as a 5 day crash course in biology?

I was pleasantly surprised by the number of people who were interested in my poster. This was the first time I presented a poster at a national meeting, so I was a little unsure about what would happen. My poster, which described the crystal structure of a red copper protein, attracted blue copper enthusiasts, who were excited to be introduced to a protein that may be a long-lost structural cousin of blue copper proteins.

I thank the ACA for their generous travel grant, which permitted me to attend and present my poster in St. Paul, as well as the honorable mention I received for my poster. It was an important step in my scientific development as a macromolecular crystallographer.

Jeff Lovelace, University of Toledo



I enjoyed the 2000 ACA meeting. If it were not for the travel grant I don't think I would have been able to attend. The conference provided a lot more than an interesting collection of presentations. For example, unlike most I got to see Dr. BC Wang's rendition of YMCA at the MSC dinner.

Shhh! Don't tell anyone but I escaped from the confines of the macromolecular world and I spent the evening with the small molecule group at the Mentor/Mentee dinner. I was a little disappointed that the ACA conference did not even get one protestor as compared to the Biotech conference in Minneapolis.

Johanna Mazlo, University of Nebraska



I would like to send my sincerest appreciation for being able to attend ACA2000 in St. Paul, Minnesota. It was a great experience on many levels. Being my first visit to St. Paul, I enjoyed the Snoopys located throughout town. Scientifically, I was able to meet many colleagues in my field. This allowed me the opportunity to discuss the latest research in the various labs. For example, I obtained advice that has definitely accelerated the completion of my project. Thank you Dr. Stanfield and Dr. Greasley. The talks also acquainted me with the types of research occurring in the various areas of crystallography. It definitely provided me with a sense of crystallography's future. Personally, I enjoyed meeting so many wonderful people. Several of us would gather for dinners. I would especially like to say "HI!" to the dinner group that trekked one way by foot for 40 minutes to the Greek restaurant only to find out that it was closed for maintenance.

Lastly, I gained insight into the workings of the American Crystallography Association by attending the various meetings. I hope to become more involved in the future. This has been a very enriching experience.

Eliud Oloo, University of Saskatchewan, Canada

My attendance and participation at the ACA Golden anniversary meeting in St. Paul was made possible by a generous student travel award.

For me, being at the ACA meeting was a unique experience in the sense that it was my very first opportunity as a graduate student to present my work to a group comprised of first-rate scientists in my field of study and research

I derived great benefit from the Shelx workshop and from the many other interesting sessions that I attended in the days that followed. I was particularly keen on talks exploring the direct applications of X-ray crystallographic methods in the design of drug molecules since that is where my career ambitions lie.

It was also quite an inspiring experience to meet, listen to and talk to people who I believe would belong to the crystallographic association Hall of fame - if such a hall were in existence. People I had previously only read about in books, manuals and journal articles. I was particularly pleased to meet Dr. Lyle Jensen whose beautifully written introductory text "X-ray structure determination", co-authored with George Stout, I do credit with helping me to understand a good number of somewhat abstract concepts in X-ray crystallography.

I came out of the meeting with a good idea of what the current hot research areas in crystallography are and having a clearer picture of what avenues I could take to direct my academic and career interests exactly where I want to go with greater precision.

I would like to extend my sincere gratitude to all donors who made this year's awards available to young scientists like myself.

Sean X. Ouyang, Texas A&M Univeristy

Going back to the hot summer days in College Station, Texas, made me really miss the St Paul breezes.

Going to the meeting gave me lots of learning opportunities. I enjoyed very much my trip during the six day stay in St. Paul. I attended the Shelx and Twinning Workshop and listened to many excellent talks for exciting new results and hints/tricks for problem structures. I had a nice chat in a mentor/mentee diner with Dr. B. C. Wang, and had a reunion pictures time with ACA summer school teachers and classmates. I especially enjoyed the discussions with twinning experts Dr. Bob Sparks and Dr. Vic Young about my quadruply twinned crystal problems.

I would like to express my thanks to ACA and all the supporting organizations/companies/members for contributing to my travel award for the St. Paul meeting.

Thomas Proffen, Michigan State

Having received my Ph.D. in crystallography back in Germany, crystallographic meetings are always close to my heart, so I was naturally very happy to receive the ACA travel grant allowing me to attend the meeting in St. Paul. I especially enjoyed the sessions on battery materials and on "New Science using New Neutron Sources and Instruments". Attending the meeting gave me a great opportunity to meet other US scientists employing crystallographic methods allowed me to make new contacts for future collaborations. I would like to take the opportunity to thank all people that helped to make this meeting so enjoyable.

Akhilesh Tripathi, SUNY, Stony Brook

The travel grant was very timely and helpful. It gave me 100% surety to attend ACA-2000 at St. Paul. I had an enriching experience with some of the latest developments and software additions in single crystal structure determination. The lectures by Dr. Victor Young, Dr. Robert Sparks and Dr. Regine Herbst-Irmer on twinning were particularly invigorating for me.

Amy Wernimont, Northwestern University

I definitely enjoyed the meeting and thought it very informative and entertaining. The new techniques and topics made me think in a new way about my projects while the problem structures lectures made me feel better about my own problems.

The travel grant was helpful in that it paid for hotel accommodations, food and gas for the car my colleagues and I drove. We didn't worry about how much everything was going to cost us and instead were able to focus on learning new subjects and meeting fellow crystallographers. All in all, it was a very good experience and I thank the ACA committee for their generosity.

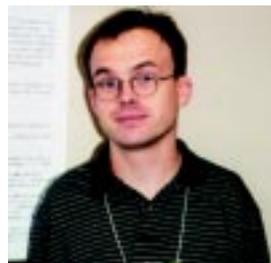
Chris Bunick, Vanderbilt University

Having just graduated from college in May 2000, the ACA travel award offered a young student like myself the chance to travel to St. Paul without incurring a huge personal debt. Because of the ACA travel award, I was able to attend the meeting and present my undergraduate crystallography research done with Dr. Gerald Stubbs at Vanderbilt

University. After battling through the gauntlet of exhibitors, notable crystallographers did make it to my poster and offered insights into improving my future results. I entered my poster with the hope for helpful comments, and with the ACA travel award, I was able to actually show up in St. Paul to hear such advice—from people such as Alexander McPherson, Robert Sweet, Bob Cudney, Don Voet, Lisa Edberg, and Kris Tesh.

And while great progress was made on the research end, the ACA meeting was also filled with endless fun—booze and dancing at the MSC dinner (Somebody out in the crystallography community has a picture of BC-Wang doing the YMCA dance at the MSC dinner), greeting Snoopy around every corner, harassing the genetics activists, and reminiscing about Howard Hughes. Of course, following around the Oak Ridge Boys, Gerry Bunick, Joel Harp, and Leif Hanson, could only lead to trouble.

But when all was said and done, the learning and the fun over, one thought still lingers without completion...is anyone going to answer Wim Hol's question to Tom Steitz? "Mr. Steitz, how do you think life was created in light of your ribosome structure?" Guess we'll all have to show up in Los Angeles to find out the answer.

Wolfram Tempel, University of Georgia

First I would like to thank the ACA for the financial support I received for my attendance. It was my first ACA meeting and I am grateful to have had the opportunity to attend one so shortly after my entry into the field of crystallography. I became interested in protein crystallography during the time of my graduate work on the synthesis of enzyme inhibitors,

incidentally in an area that was represented by the talk on rhinovirus inhibitors. In the meeting announcement I was pleased to see that macromolecular crystallography would have such a prominent position during this year's meeting. Many interesting talks on protein structures were offered, sometimes overlapping in time so that it was difficult to choose. The session on Problem Structures at the end of the meeting was a true highlight. In fact, it made me wish that most presentations on protein structure at future meetings should be presented as Problem Structures. This subject group is, in my opinion, particularly well suited to the format of a meeting that focuses on experimental method, such as a meeting of crystallographers.

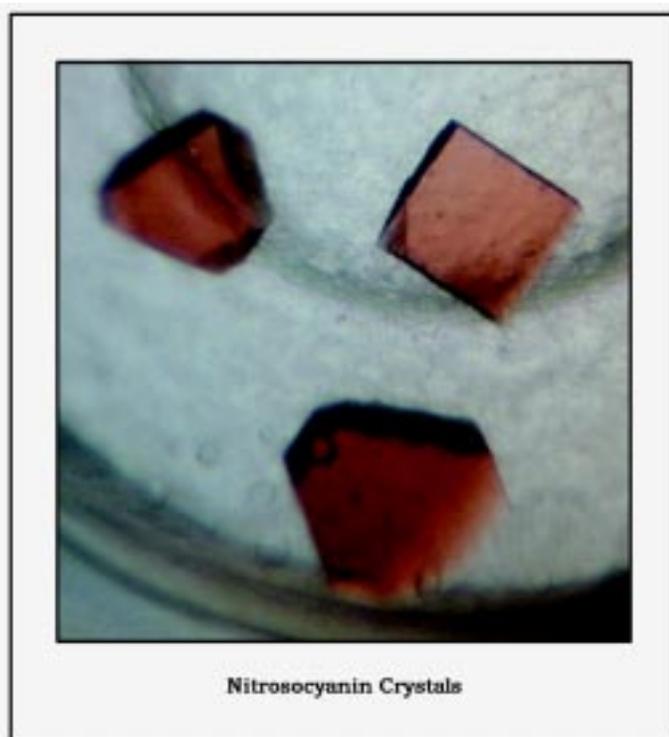
I was surprised to see so many known faces at the conference. I got to know many of them at the ACA summer course in Athens, Ga. where I was a student in 1998. I was impressed with the progress they have made during the short time that has passed since. I look forward to my next ACA meeting.

ORTEP of the Year - 2001

The R. Harlow Foundation for Disabused Crystallographers will, as usual, be presenting a check for \$200, at the Los Angeles ACA Meeting in July 2001, for the best example of how an ORTEP drawing was used to spot and resolve a "problem" structure. Any published structure in a refereed journal is eligible for submission, but the submitter must clearly demonstrate the manner in which ORTEP (or more generally, any thermal-ellipsoid plotting program) was used to indicate that the structure as published was incorrect and must provide an ORTEP of the "corrected" structure. Structure determinations where the ORTEP drawing failed to indicate a "problem" structure are also eligible; these too are educational. Entries should be addressed to the Foundation at 7 Shull Dr., Newark, DE, 19711, USA. You do not have to be present at the LA Meeting to win (but it sure would be nice to see you there).

I would also like to point out to the crystallographic community two other Foundation initiatives, the Hydrogen Challenge and the Low-temperature Prize. Details of these awards can be found under the ACA Service SIG web page at <http://www.pitt.edu/~geib/aca.html>. In addition, I am looking for examples of mis-printed stereo ORTEPs. i.e. stereo pairs reversed, same image twice (no stereo effect), etc. For entries in this category, I will send you a small token of my appreciation.

R. Harlow



Excerpts from Lyle H. Jensen's Buerger Award address: Then and Now: Reflections on a Mature Discipline, July 24, 2000.



(In his opening remarks Lyle described his memories of M.J. Buerger and his early reliance on the work of Prof. Buerger:)

"It was September of 1939 when I enrolled as a graduate student at the University of Washington (UW) in Seattle. At that time, crystallographic research on the diffraction of X-rays by crystals was well established at a number of U.S. academic institutions such as Cal Tech, MIT and Harvard University. In fact, a local group of crystallographers in the Boston/Cambridge area, mostly mineralogists, met in the autumn of 1939 to organize a professional society, the Crystallographic Society of America (CSA) with M.J. Buerger, Prof. of Mineralogy at MIT, as president.

The following year, Prof. Buerger was appointed to the Committee on X-ray and Electron Diffraction in the Division of Chemistry of the National Research Council in Washington D.C. In 1941, that Committee agreed to establish a second organization, the American Society for X-ray and Electron Diffraction (ASXRED). Because of the disruptions caused by World War II, only a very few meetings of either society were convened. In 1948/49, members of both the CSA and the ASXRED voted to merge and on January 1, 1950, the American Crystallographic Association became a reality. Thus at this meeting here in St. Paul, we celebrate the 50th anniversary of our organization.

In retrospect, I find it an intriguing coincidence that just as I entered graduate school at the UW, the CSA, was being organized. But that was continent away - !

In graduate school, course requirements were relatively heavy and with the added responsibilities of a Teaching Fellowship, I was not ready to begin my thesis problem until completing the qualifying examinations in the spring of 1942. The timing was fortunate because a young faculty member, Ed Lingafelter, from UC Berkeley had joined the UW Chem. Dept. and had ordered equipment to initiate X-ray crystallographic studies.

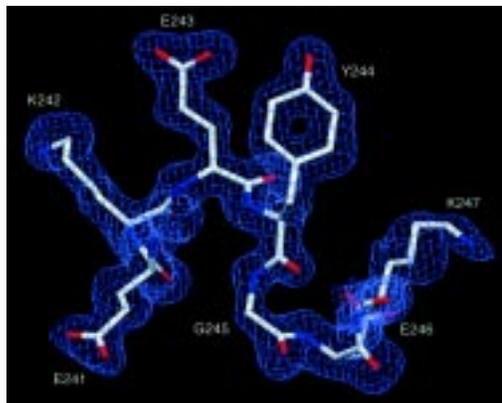
As the first graduate student to choose a thesis problem in that area, I found my first responsibility was to set up the new equipment which consisted of a cold-cathode, demountable X-ray tube, a high voltage transformer and control unit, and a rotation/oscillation camera.

At the same time, I needed to learn the theoretical basis of diffraction by crystals and how to record and interpret the data. In 1942, few books on the theory and practice of X-ray diffraction by crystals were available, so most of my initial efforts involved searching the literature. Although this was a rewarding activity, and I developed a profound appreciation for the contributions of the English crystallographers, searching the literature was time intensive. And then a new book appeared entitled "X-ray Crystallography" by M.J. Buerger. Here was just what I needed, everything from the theoretical basis of diffraction and crystal symmetry to various ways of recording the data.

Setting up the equipment was without incident, except that the first diffraction photographs showed only weak white radiation streaks with no evidence of characteristic reflections! On disassembling the X-ray tube, we found the windows to be of a thick rubberized material that absorbed most of the radiation. Thus, we fashioned new windows from 0.005" Be metal sheet and pronto - an intense beam and excellent diffraction pattern.

Incidentally, we had no sensitive electronic detector to monitor radiation, only a small fluorescent screen and the X-ray film itself. A small piece of film enclosed in a black paper envelope with a superposed coin served as a convenient radiation detector. I set this to intersect any possible secondary radiation from the face of the pinhole system. After several weeks of X-ray time, the film showed only the faintest evidence of any shadow from the superposed coin. Although this did not provide an absolute value for the radiation level, I was assured that it was very low.

My thesis project involved a series of sodium 1-alkane sulfonates, $C_nH_{2n+1}SO_3Na$ with even n from 8 - 18. These structures have detergent properties and were being intensively studied in the Chem. Dept. at that time. Since the graduate students involved in these studies synthesized large quantities of these structures, they were willing to provide the small amounts I needed for growing diffraction quality crystals. One of these students showed me the glistening, extremely thin, sheet-like crystals that precipitated from aqueous media in the purification step of his prep. I soon found that crystallizing from mixed solvents produced thicker, tabular crystals which could be improved by adjusting the dielectric value and viscosity of the crystallizing media. In this way, at temperatures from 40 - 55°C, satisfactory crystals could be grown in a few days, although the record shows that one batch was allowed to grow for 330 days!



During the course of my thesis research, I became quite optimistic about growing crystals. I remember thinking that if paraffin chain molecules could be crystallized, then any pure substance could be crystallized - it was just a matter of finding the right conditions.

Rotation and oscillation photos showed the crystals to be monoclinic with a and b axes for the six structures $\sim 16.8 \text{ \AA}$ and $\sim 10.1 \text{ \AA}$ respectively. But the long c axes turned out to be a tough problem, ranging as they do from $\sim 55 \text{ \AA}$ to $\sim 106 \text{ \AA}$. Thus extreme accuracy is required in aligning the crystals for oscillation photos about the c axis. A procedure was finally developed involving Laue photos and gnomonic projections. The layer lines with odd l were extremely weak, suggesting that the structure is composed of molecular bilayers, c corresponding to the thickness of two bilayers, approximately four chain lengths.

Indexing reflections on the basis of oscillation photos is a tedious task of graphically reconstructing the reciprocal lattice level by level and matching the distance of each reflection from the rotation axis. The extinctions turned out to be h0l for h and l odd and hkl for k+l odd, indicating the space group as Aa or A2/a. In all this part of my thesis research, Buerger's book proved indispensable.

As a final check that my indexing was correct, I followed Buerger's description of one of the moving film methods for recording diffraction data, the Sauter method. But in practice it can be made even simpler by substituting a one rpm synchronous electric clock motor to drive the circular film. Only a few hours was necessary to construct the modification. The result was the h0l level of reciprocal space for $C_{14}H_{29}SO_3Na$. For this moving film photo, indexing becomes simply a matter of inspection and counting, requiring only minutes instead of the intense effort over many, many weeks with the oscillation photos.

Subsequent work showed that the crystal form I had initially discovered was a 1/8 hydrate. Thus we had 4 units of $(RSO_3Na)_8 \cdot H_2O$ per unit cell. This meant that even for the C_{10} structure, we had 121 nonhydrogen atoms per asymmetric unit, far beyond our computing capability at the time. Nevertheless, low resolution, 3-dimensional, eye-estimated data were collected, mainly from a axis oscillation photos from a small crystal of $C_{10}H_{21}SO_3Na$. Three line Patterson functions, $P(1/2, y, 0)$, $P(0, 0, z)$ and $P(1/4, y, 0)$, were evaluated by the use of trig tables and showed the relative positions of the molecules in the unit cell.

While I was intensely absorbed in my thesis project, Ed Lingafelter acquired a little book that caught my attention: *The Philosophy of Physical Science*, authored by the eminent British Astronomer, Sir Arthur S. Eddington. He began this book with a story about an ichthyologist who, in his study of fish, recorded among other observations the length of each specimen netted. One day in scanning his records, he noted that no fish measured less than 2" in length. And then he realized that his net with a grid spacing of 2" would not retain any fish less than 2" long. Eddington went on to show how physical data collected by various techniques are limited by the instrumentation. Through the years this story keeps reminding me that what is missed by one technique can be supplied by another, and we gain the most

complete view of nature by applying a diversity of techniques."

(Lyle went on to describe his early work at Ohio State University, his return to the University of Washington Chemistry Dept. and his subsequent switch to life sciences. Lyle started with biologically interesting small molecules and his interest in the accurate refinement of small molecules strongly influenced his later conviction that the same principles could be applied to proteins. His studies on the beautiful crystals formed by rubredoxin provided a good basis for his eventual demonstration of the validity of this conviction.)

"As the idea gained credence that protein models could be refined, improved programs were developed, which included energy minimization, constraints or restraints on known molecular parameters, and other features. Synchrotron sources of radiation have greatly increased the rate of data collection, and along with area detectors, have improved the quality of the data and increased its range. Data have also been improved by holding the crystal at very low temperature, reducing radiation damage by the X-ray beam. Through the years the capacity and speed of computers have increased enormously so that the vast amounts of data from macromolecular structures can be processed efficiently. And now we hear that IBM is developing a computer that is projected to be 500 times faster than anything now existing. According to the account I read, the machine is being developed for the sole purpose of modeling how a protein chain folds into the particular shape that we know as the active protein. But it is just that shape we determine when we solve protein structures. Now I have a question: Will Blue Gene, as the machine has been dubbed, displace protein crystallographers? I doubt it; but whatever the outcome, byproducts of the enormous development effort are likely to be useful in unforeseen ways. Could Blue Gene produce a "trial structure" that we could refine?

So now we live in a technological age, computers of unprecedented capacity and speed, and almost instantaneous communication with colleagues anywhere in the world. But I have a question: When do we have time to think? One can only applaud the improved efficiency of modern equipment, but the frenetic pace of so much research today results in undue stress that can be detrimental in human terms. In a recent book on the genetic basis of cancer, I came across an extreme example of research under pressure. The author cites the case of a crash effort involving 24 hour work days by one group to beat a competing group to a deadline. Such an extreme course of action seems to me unworthy of science and has led to some questionable ethics. A saying I encountered years ago suggests a useful response to a competitive situation: "Compete with your possibilities and not with your neighbor".

In closing my remarks, I wish all members of the ACA success and tranquility in your crystallographic efforts – as well as your lives in general."

(The full text is available from either of the Newsletter editors.)

Update on Protein Data Bank Activities

An increased number of depositors and users has kept the staff of the PDB busy in the year 2000. The main PDB server receives an average of 90,000 hits per day — more than one hit per second, 24 hours per day, seven days per week.

During the period of January 1, 2000 through October 31, 2000, 2493 structures have been deposited to the PDB. Of these structures, approximately 57% are deposited with an “hold until publication” release status, 23% are deposited with a “release immediately” status, and 20% are put on hold until a given date.

A popular PDB feature is the ADIT Validation Server, that allows depositors to check a structure at any time during structure determination and refinement. It checks the format consistency of coordinates during the Precheck step, and creates validation reports about a structure before deposition during the validation step. Structures can then be deposited to the PDB using ADIT (<http://pdb.rutgers.edu/>). Once deposited, these entries are processed to completion, returned to the author for review, and released on the PDB site (<http://www.pdb.org/>) and its mirrors.

During this past year, the PDB staff has continued to enhance and upgrade the capabilities of the PDB searching and reporting tools. As part of the Data Uniformity project, PDB members have curated the R-factor, resolution data, and primary citation data for all entries in the PDB, and have incorporated this information into the database. These fields are available for improved searching, and the updated data is available via database reports.

Other developments in query and reporting include expanded ligand searching and reporting capabilities, improved access to dynamic links using the Molecular Information Agent (<http://mia.sdsc.edu>), the accurate query of enzymes, the incorporation of cross-links to sequences databases, and improved graphics options. The PDB can now be queried based on source, by number of chains, and by the availability of experimental data. After internal testing and before incorporation into the main Web server, query and reporting enhancements are made available at <http://beta.rcsb.org/pdb/> for public testing.

At the ACA Annual Meeting in St. Paul, MN (July 22- 27), the PDB hosted an exhibit booth and a User's Meeting. It was a great experience to meet so many of our depositors and users in person, and we look forward to seeing you in Los Angeles in 2001!

Questions and comments should be sent to info@rcsb.org. Updated news is available from the PDB home page.

Christine Zardecki

E is for Electronic

- and exciting, and enterprising, and edifying, and perhaps even entertaining! But certainly ESSENTIAL.

January 2001 will see the first issue of the IUCr's new electronic-only journal, Acta Crystallographica Section E: Structure Reports Online. It is being launched by the IUCr in collaboration with the Cambridge Crystallographic Data Centre (CCDC) in order to provide rapid and efficient publication of

organic, metal-organic and inorganic crystal structures. As such it replaces the ‘electronic papers’ published in Section C recently, and seeks to build further on our experience with that medium.

The section editors are Bill Clegg (University of Newcastle) and David Watson (CCDC).

A number of important enhancements are being made in the introduction of Section E.

* All communication between authors and the journal, from web-based submission through checking and review, to proofs and publication, is entirely electronic.

* Author services provided will include a new free easy-to-use CIF editor, and we are developing tools for incorporating output of popular word-processing and chemical drawing software in the submission, to make life easier: no more crude text-editing of CIF's!

* Published articles, available to readers in PDF and HTML formats, can include graphical illustrations of many kinds; even multi-media supplements are possible. Extensive supplementary material is also provided, including the complete CIF, diffraction data, and output of the automatic checking procedures.

* The length of textual comment by the author is flexible and it can be quite brief in many cases.

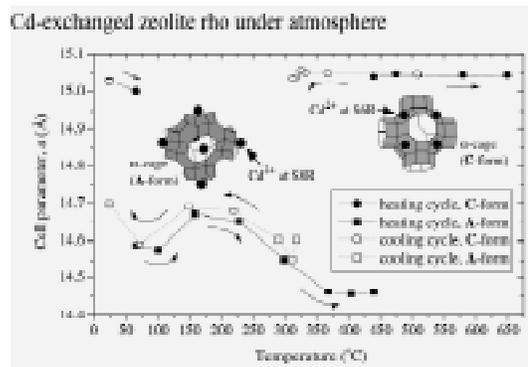
* Although the journal will not publish fundamentally flawed work, and is not going to act as a repository for poor quality results, acceptance criteria are more flexible than hitherto in Section C. The publication of more difficult and challenging structures is encouraged, with appropriate comment by authors on the problems encountered.

* Each published report (of a single structure) includes a set of ‘key indicators’ for precision, completeness, and validation of the results, enabling readers easily to make their own assessment of how these might be used appropriately in their own work.

So here is your chance to use a new and fast publication method and perhaps make an impression on those piles of unpublished structures that the world should hear about. Can we catch up on the flow of data from modern diffractometers?

For further information on the journal and how to submit and subscribe, see the IUCr web page for Crystallography Journals Online, at <http://journals.iucr.org>

Bill Clegg



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What's on the Cover

Basic ribbon palm tree design by Daniel Anderson, final production by David Goodsell. Patti Coley added the meeting information. All photos are by Michele and Tom Grimm and are used courtesy of the Los Angeles convention and visitors Bureau and Picture LA.com. Upper left: Downtown at dusk. Upper right: Westwood movie premier. Middle left: the Westin Bonaventure (the meeting site). Middle right: Los Angeles city hall. Lower left. Surfer. Lower right: : Japanese garden at Huntington Gardens.

Pauling Award Winners

Scattered throughout the Newsletter are graphics selected from the posters of some of the Pauling Award winners at the ACA meeting in St. Paul. Jennifer Padilla (page 43): Symmetry makes possible the construction of large macromolecular assembly from identical subunits. In principle, protein cages, filaments, tubes, layers, and crystals can all be constructed by creating a fusion of two proteins that form simple oligomers such as dimers or trimers. If the symmetry elements associated with each oligomer can be held in fixed orientations that match one of the point, laer, or space groups, then self-assembly of the symmetric complex results. This design principle has been used so far to crete a protein cage and protein filaments. Satinder K Singh (page 18): A 2 |Fo| - |Fc| map for Isocitrate dehydrogenase from *Bacissul subtilis*. It's contoured at 1 s and it typical of the electron density observed throughout the structure and illustrates the dramatic atomic detail that can be visualized at a resolution of 1.55 Å. Raquel L. Lieberman (page 16): Crystals of nitrosocyanin, a novel red copper protein from *Nitrosomonas europaea*. Akhilesh Tripathi (page 13): Polyhedral reprersentaton of the structure of an octahedral molecular sieve projected along [3-21]. The Na1 and Na2 polyhedra in yellow link the strands of octahedrally coordinated Ti/Nb atoms in blue. Yongjae Lee (page 20); Graph showing the temperature driven relocation of Cd in the zeolite RHO.

Contributors to this Issue: In additon to the contributors listed with by-lines the Editor would like to thank Patti Coley, Louis Delbaere, Sue Duncan, Guiseppi Filippini, Marcia Evans, Sally Lunge, Bill Stallings, and Ron Stenkamp. Graphics were provided by Pauling Prize winners Yongjae Lee, Raquel L. Lieberman, Jennifer Padilla and Satinder K. Singh. Photos were provided by Judy Flippen-Anderson, Bill Duax, Edgar Meyer, and Winnie Wong-Ng.

11th Southwest Macromolecular Symposium Texas A & M, November 18, 2000



Speakers include: Top row: Matther Baker (Rice), J.J. Almrud (UT Austin), Bob Fox (Galveston), and Kris Tesh (Molecular Structure Corp.). The second row: Shahram Khademi (Texas A&M), Chetfen Crossnoe (University of Houston), and Jon Robertus (UT Austin). The front row: Mark White (Galveston), Angel Criswell (Rice), Kara Bortone (UT Austin), Sheng Ye (Dallas) and Hong Zhang (Dallas).

Sunbelt Saturdays are not supposed to be wet, but hearty crystallographers from all over Texas braved the elements to gather for the eleventh SouthWest Macromolecular Symposium on Saturday, Nov. 18., in the Biochemistry Building at Texas A&M University. Sessions and posters ran all day, interspersed with coffee breaks and lunch. Titles and abstracts are available at: <http://www.tamu.edu/struct/SWMS/SWMSchedule.html> The program was sponsored by the Nonius Company and by the Molecular Structure Corporation.

The highlight of the meeting was the presentation by Bob Fox of the schedule for the Gulf Coast Synchrotron MAD facility: Crystals should be diffracting by next May and happy users processing data routinely by next summer. The host synchrotron (CAMD: <http://www.camd.lsu.edu/>) provided a 7.5 Tesla wiggler, which gives us a potential flux comparable with frequently used beamlines, but this one is next door. The beamline is sponsored by LSU, NSF and NIH funding. Watch this spot for updated information.

Edgar Meyer

Physics Today Increases Online Presence

Physics Today magazine has expanded its web site to include full editorial content from the magazine. The new redesign includes repackaging the articles into a more readable format for the web. "Over the next year we hope to expand the amount of material on physicstoday.org to include papers from the magazine's archive," says Paul Guinnessy, Physics Today's Online Editor. "For example, the December online edition of the magazine will include a collection of arms control articles from over the past few decades." The site is currently developing new services and restricted access controls for launch next year. Access will remain free to subscribers and individual members of ACA and other societies connected to the American Institute of Physics. <http://physicstoday.org>

Paul Guinnessy, Online Editor Physics Today

The European Crystallographic Association Election Results for 2001

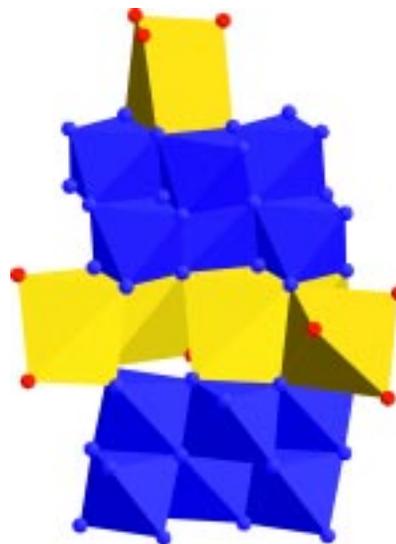
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RapiData 2001

Rapid Data Collection and Structure Solving at the NSLS: A Practical Course in Macromolecular X-Ray Diffraction Measurement will be held at the Biology Department and NSLS Brookhaven National Laboratory April 22-27, 2001.

Synchrotron radiation has become an indispensable tool for macromolecular crystallography groups. Beam time is such a scarce commodity that it pays for some workers to enhance their skills by participating in an intensive hands-on training program.

To provide such an educational experience, we have designed a course in Rapid Data Collection and Structure Solving: http://www.x12c.nsls.bnl.gov/rr_course_2001/course_announce.html. It will be sponsored in part by a grant from the National Center for Research Resources of the National Institutes of Health. In this course we will develop the ideas behind conventional methods for diffraction data collection (crystal freezing, data-reduction programs), will emphasize aspects unique to the synchrotron experience (MAD phasing, rapid structure solving), and will give students practical experience in work on their own specimens.

The course should accommodate 48 students total: 24 students, each with an experimental problem to be solved, and 24 who will gain equivalent experience as observers and helpers. We invite applicants for this course {starting after about 2 January 2001}: http://www.x12c.nsls.bnl.gov/rr_course_2001/applic.html

Robert M. Sweet (sweet@bnl.gov)

The US National Committee for Crystallography

Front row, left to right, Marv Hackert, Connie Chidester, Cynthia Stauffacher, Bob Sweet, second row, Jane Griffin, Jim Kaduk, John Clardy, Ian Robinson, Gary Gilliland, back row, Charles Prewitt, Alex Chernov, Bill Duax, Bill Stallings, Peter Busech, John Parise, Bing Jap, Howard Einspahr.

The US National Committee for Crystallography (USNCCr) is the body which represents the United States to the International Union of Crystallography (IUCr). Administratively, it falls within the Committee on International Organizations and Programs of the National Research Council. Its funding (and that of other U.S. national Committees) is provided on a proposal basis by NSF.

Under new Chair Marv Hackert, Vice Chair Jon Clardy, and incoming Secretary-Treasurer Winnie Wong-Ng, the USNCCr is refining its goals and objectives, to concentrate on benefitting crystallography in the U.S. and worldwide. Suggestions from members of the community are welcome.

A theme of the next three years will be "outreach" - both to the general public and to crystallographers in the developing world. The "Africa Initiative" described by IUCr President Henk Schenk in the most-recent *IUCr Newsletter* suggests that the ACA (as Regional Affiliate) and the USNCCr might consider the opportunities for outreach to Latin America. Both groups are considering how they best work together with Latin American crystallographers. USNCCr Chair Hackert earlier this year attended the first-ever meeting of the Chairs of various U.S. National Committees. We can expect more such interactions, and joint projects among National Committees for the general good of science.

One way for US crystallographers to have a voice in the international crystallographic community is to provide input to the IUCr. The USNCCr has been asked to suggest members for the Jerusalem Program Committee, and additional suggestions are welcome. We have also been asked to suggest candidates for members of the IUCr Executive Committee and Chairs and

Members of Commissions. Suggestions for Program Committee, Executive Committee, and Commissions should be sent to the newly elected Secretary-Treasurer Winnie Wong-Ng (wongng@hotmail.com) before 1 February 2001. Suggestions for chairs and topics for microsymbiosia, plenary, and invited speakers for Jerusalem should be sent to Howard Einspahr (howard.einspahr@bms.com), also by 1 February.

A vital concern of the USNCCr is crystallographic education. The Education Subcommittee (Cyndy Stauffacher (cyndy@gauguin.bio.purdue.edu), Ian Robinson (ikr@uiuc.edu), and Charles Carter (carter@med.unc.edu)) is attempting to compile an inventory of crystallography courses and education resources in the U.S. Input from members of the community is especially welcome. An ongoing concern of the USNCCr is to ensure that necessary crystallographic expertise is available when and where needed.

As for the Glasgow meeting, the USNCCr expects to have available ~\$40-45,000 to support the travel of young scientists to the Jerusalem IUCr meeting. Details of the grant program will be available at the Los Angeles ACA meeting.

The USNCCr attempts to represent the breadth of crystallographic interests and views in a relatively-small body. Nominations (including self-nominations) for candidates in next year's election are welcomed by the Nominating Committee (Charles Carter (carter@med.unc.edu) and John Parise (john.parise@sunysb.edu))

Jim Kaduk, USNCCr Secretary-Treasurer

NIST Centennial Celebration Crystallographic Highlights

The Centennial Celebration of the National Institute of Standards and Technology (NIST), formerly known as the National Bureau of Standards (NBS), takes place in the year 2001. NIST has a long history (> 50 years) of crystallographic research, and has made significant contributions to the advancement of the fields of crystallography, biology, and materials science. Many scientists from various divisions in diverse disciplines have contributed to this history. The research has been carried out as a fundamental part of the overall mission of NBS/NIST and has become an integral part of many aspects of its research programs. The following articles are designed to give a brief overview of the role of crystallography at NIST — past, present, and future. The articles cover a broad spectrum of topics including structural databases, high-pressure crystallography, neutron crystallography, structure determination via neutron and x-ray diffraction, quasicrystals, reduced cells, magnetic structure determinations, polymer crystallography, electron diffraction, materials properties databases, protein crystallography, novel biomaterials, biological minerals, crystallography of construction materials, and synchrotron radiation. An article on the protein databank is not included as this topic is discussed elsewhere in this newsletter. Finally, more detailed coverage of a few of the articles will be given in a one-day symposium, dedicated to the celebration of this special occasion, at the ACA meeting in Los Angeles.

Winnie Wong-Ng and Alan D. Mighell

High Pressure Crystallography

Crystallographic studies began two years after the invention of the diamond anvil cell (DAC) at NBS in 1958. The instrument, developed initially for infrared spectroscopy, was modified in 1960 by Charles E. Weir and Gasper J. Piermarini for x-ray powder diffraction studies. Powder patterns were obtained on materials such as AgI, Bi, Tl, and several alkali halides and rare-earth elements. Compression data were obtained and many materials were shown, for the first time, to exhibit pressure-induced polymorphism.

In 1962, Alvin Van Valkenburg developed a gasket method to confine liquids in the DAC by placing a thin metal sheet containing a small hole with the liquid between the anvil faces. As a result, single crystals could be compressed in a hydrostatic environment in a gasketed DAC. This was very important to crystallography because it led Block, Piermarini and Weir in 1964 to develop a high pressure single crystal x-ray diffraction precession camera technique which utilized a DAC fabricated from Be metal to minimize x-ray absorption effects. For the first time, x-ray intensity data were obtained at high pressures from single crystals of ice VI and ice VII in equilibrium with water at room temperature. An analytical procedure was developed to calculate absorption corrections for the Be cell and it became possible to determine unknown structures at high pressure. The crystal structure of a high pressure polymorph of benzene (C₆H₆ II) at 2.5 GPa was the first determined by this method. This work was of great importance because it proved that single crystal structure analysis was possible at high pressures utilizing a DAC. The method served as the foundation for later more advanced work with the introduction of automatic diffractometers and miniaturized DACs. In 1971 the group working with the

DAC at NBS invented the ruby fluorescence method of pressure measurement. These achievements, combined with many others at NBS, were, indeed, milestones, for together they ignited and fueled an explosion of activity, not only in crystallography, but in high pressure research in general, by laboratories throughout the world, an activity that continues unabated today.

Gasper Piermarini and Stanley Block, Materials Science and Engineering Laboratory

Neutron Crystallography at NBS / NIST

Neutron diffraction at NBS / NIST started soon after the NBS Reactor went critical in the summer of 1969. The first instruments designed for crystallographic work were a low-resolution, single-counter powder diffractometer using radiation from a 220 Cu monochromator with variable take-off angle, mounted on the BT-1 beam port, and a single-crystal four-circle diffractometer at the BT-8 beam port. The development of the Rietveld method of profile analysis increased the range of complexity of the structures soluble with high-resolution powder diffractometers, and decreased the need for single crystals. This trend was felt at NBS / NIST, and the original powder machine was replaced, in rapid succession, first by a five-counter diffractometer of intermediate resolution, and then by the present day 32-counter diffractometer, whose resolution can be varied from low to high.

In parallel with these technical developments, significant improvements were made in the techniques of profile refinement. Revised versions of the original program were written that for the first time included background in the refinable parameters in the model, and that applied general constraints directly (E. Prince, *internal communication*, 1980). In addition, the entire procedure initially proposed by Rietveld (*J. Appl. Cryst.* 2, 65 (1969)) was placed on solid statistical grounds when it was proved that estimates of the parameters and their standard deviations are not significantly different whether the method of refinement uses peak profiles or integrated intensities (E. Prince, *J. Appl. Cryst.* 14, 157 (1981)). The complexity of the crystallographic problems studied at NBS / NIST has increased with time, from early work on simple structures in which light atoms, such as Li, coexist with heavy ones, such as Ta (A. Santoro, R. S. Roth and D. Minor, *Acta Cryst.* B33, 3945 (1977)), to more complicated, multi-phase cases in which the nuclear and magnetic configurations of each phase have to be determined with high precision (Q. Huang *et al.*, *Phys. Rev. B* 61, 8895 (2000)).

Present research focuses on relating crystal structure and physical properties of complex systems analyzed with multiple techniques, ranging from small angle scattering to spin-echo spectroscopy which are available at the NIST Center for Neutron Research. An example which illustrates the advance made possible by the use of modern methods is the determination of the complete crystal structure (including Li in an asymmetric unit of 46 atoms) of RUB-29, a microporous lithosilicate of formula Cs₁₄Li₂₉(Li₁₈Si₇₂O₁₇₂)14H₂O (So-Hyun Park *et al.*, *in preparation*). This material has been analyzed by the combined use of NMR, synchrotron x-ray techniques, and neutron powder diffraction.

A. Santoro, NIST Center for Neutron Research

Magnetic Structure Determinations at NBS/NIST

Magnetic neutron scattering plays an essential role in understanding the microscopic properties of a vast variety of magnetic systems, from the studies of fundamental nature, symmetry, and dynamics of magnetically ordered materials to elucidation of the magnetic characteristics essential in technological applications. From the early days of neutron measurements at NBS/NIST, magnetic diffraction studies have been a central theme involving many universities, industrial and government labs from around the US and worldwide. Such measurements have been used to determine the spatial arrangement and directions of the atomic magnetic moments, the atomic magnetization density of the individual atoms in the material, and the value of the ordered moments as a function of thermodynamic parameters such as temperature, pressure, and applied magnetic field, on single crystals, powders, thin films, and artificially grown multilayers.

There have been hundreds of studies of magnetic structures and ordering at NIST, on wide classes of materials. Early work addressed materials such as spinels and ferrites, followed by rare-earth intermetallics (J. J. Rhyne, *IEEE Magn.* 8, 105 (1972)) and rare earth hydrides. One topic that has sustained interest over the years is the magnetic ordering that occurs in superconductors. The Chevrel-phase superconductors (e.g. RMO_6S_8) and related compounds typically order antiferromagnetically at low temperatures (~1K), which happily coexists with superconductivity. In the rare and more interesting situation where the magnetic interactions are ferromagnetic, there is strong competition with superconductivity that gives rise to long wavelength oscillatory magnetic states and/or reentrant superconductivity (*Phys. Rev. Lett.* 52, 133 (1984)). The cuprate superconductors (e.g. $\text{RBa}_2\text{Cu}_3\text{O}_{6+x}$) offer new and interesting perspectives into our understanding of “magnetic superconductors” (*High Temperature Superconductors*, Springer-Verlag, 1990)). The rare earth ions order at low temperature similar to “conventional” magnetic superconductors, while in the de-oxygenated, insulating state the Cu spins order above room temperature. In the superconducting regime the rare earth spins still order magnetically, while the Cu spins typically don’t order but rather exhibit quantum spin fluctuations which are thought to play an essential role in the formation of the (*d*-wave) superconducting pairs. Nevertheless, there always seems to be an exception to the rule, and indeed (weak) long range spin density wave order of the Cu spins has recently been observed to develop, at the same temperature as the onset of superconductivity (Y. S. Lee, et al., *Phys. Rev.* B60, 3643 (1999)). Other recent work includes the magnetic structures of the rare earth borocarbides (*Phys. Rev.* B55, 6584 (1997)), and the ordering at 136 K of the Ru moment in the superconductor $\text{RuSr}_2\text{GdCu}_2\text{O}_8$ (*Phys. Rev.* B61, R14964 (2000)).

There has been a natural evolution in the complexity of materials that have been investigated; early work tended to be on relatively simple systems, but as the instrumentation has improved and calculational capabilities expanded, ever more complex structures have been successfully tackled. Magnetic neutron scattering will no doubt continue to play a dominant role for many years to come.

J. W. Lynn, NIST Center for Neutron Research

Synchrotron X-ray Characterization of Materials

Synchrotron radiation sources provide intense beams of x-rays for leading-edge research in a broad range of scientific disciplines. The NIST Materials Science and Engineering (MSEL) program to characterize materials by means of this powerful probe began in the early 1980s, with the design, construction and commissioning of a monochromatic x-ray topography station (X23A3) at the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory. This x-ray facility also supported hard x-ray microscopy and parallel-beam x-ray radiography measurements. This early x-ray topography facility was the only dedicated monochromatic instrument of its type in this country. In 1989, an ultra-small-angle x-ray scattering (USAXS) capability was added to X23A3, specifically for the purpose of enabling anomalous ultra-small-angle scattering measurements.

Today, the NIST/MSEL synchrotron radiation program includes the utilization and operation of three additional beam stations at the NSLS. X23-A2 serves a large x-ray absorption fine structure (XAFS), diffraction anomalous fine structure (DAFS), and spectroscopy community; it provides stable scanning of monochromatic x-rays in the energy range from 4.9 keV to over 30 keV. X24A, which is a shared beam station, provides radiation in the 1.2 keV to 6 keV energy range, and is used for standing-wave x-ray measurements. Finally, the U7A station, which is also a joint effort, utilizes XPS and XAS to study the structure and chemical nature of diverse materials from a surface and bulk perspective in vacuum and under atmospheric reaction conditions.

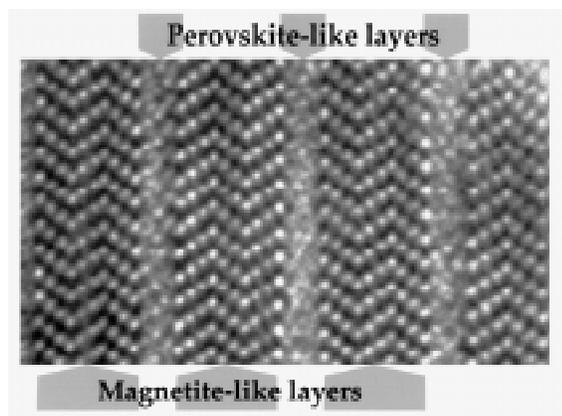
Looking to the future, NIST/MSEL became a partner in 1995 with the University of Illinois at Urbana/Champaign, Oak Ridge National Lab, and UOP, in a collaboration called UNICAT to instrument sector 33 at the Advanced Photon Source (APS) at Argonne National Laboratory. Thus, in 1998, a new NIST USAXS facility was installed and commissioned on the UNICAT undulator beam line, and in 2000 the next-generation x-ray topography experiment was installed on the bending magnet beam line. The emphasis throughout this program is on state-of-the-art measurements on advanced materials. Today, the combined portfolio of NIST facilities at the NSLS and the APS offer measurement capabilities in x-ray microtomography, ultra-small-angle x-ray scattering, high-resolution x-ray topography and XAFS. It also offers opportunities for cutting-edge experiments in structural crystallography and time-resolved structural scattering, surface/interface scattering, diffuse scattering, and magnetic scattering.

G. G. Long, A. J. Allen, D. R. Black, H. E. Burdette, D. A. Fischer, E. Nelson, R. D. Spal and J. C. Woicik

Electron Diffraction via Transmission Electron Microscopy (TEM)

There are three major advantages in using TEM electron scattering for crystallographic studies: (1) an electromagnetic lens can focus electrons to a fine probe (< 10 nm); thus single-crystal diffraction data can be obtained from a very small volume; (2) the scattering cross section for electrons is 10^3 -

10^4 as great as for x-rays and neutrons; thus it is possible effectively to detect subtle changes in a structure (e.g., ordering, distortions, short-range ordering); (3) TEM operates in a complementary image mode which enables direct imaging of structures and defects.



An example of structural imaging of the $Ba_{26}Fe_{20}Ti_{21}O_{98}$ compound. The compound was shown to be an ordered intergrowth between a perovskite-like structure (in its hexagonal form) and a structure related to the magnetite-type block of $Ba_{12}Fe_{28}Ti_{15}O_{84}$ phase.

Robert S. Roth was first at NIST to realize the possibilities of using TEM structural imaging to study structures of complex oxides. This effort was essential in establishing the important structural principles of crystallographic shear and intergrowth. In the second half of the 1980s, TEM studies of rapidly solidified Al-based alloys by Dan Shechtman resulted in one of the most important discoveries of modern crystallography – quasiperiodic structures (see a separate article in this issue). In 1986 the existence of a quasiperiodic decagonal structure, periodic in one dimension and quasiperiodic (10-fold symmetry) in two dimensions, was established by electron diffraction. Other NIST contributions to the new field of “quasi-crystallography” were (1) determining the m35 point group of the icosahedral phase using convergent beam diffraction and (2) finding the existence of “hypertwins”. Systematic use of TEM in studying complex phase transformations by analyzing domain interfaces allowed one to establish transformation sequences in the form of maximal group-subgroup relationships for numerous systems, including aerospace Ti-Al-Nb alloys and microwave dielectrics oxides $Ca(Ca_{1/3}Nb_{2/3})O_3$. With the help of high-resolution structural imaging, initial structural models were established for numerous newly discovered compounds.

L. A. Bendersky, *Materials Science and Engineering Laboratory*

Crystal Structure Determination Studies

Discovery and development of new technologies has created a continuous demand for new and improved materials. Over the past fifty years, the crystal structure of a large number of phases in diverse areas of materials science have been characterized at NIST using single crystal x-ray and neutron powder diffraction methods. These materials include dielectrics, ferroelectrics, superconductors, microwave materials, magnetic materials, and

materials for optical applications.

The transition metal complexes involving five-member heterocyclic ligand compounds such as pyrazole, imidazole, and 1,2,4 triazole were of interest because of their optical and magnetic spectroscopic properties. These compounds contain more than one active site for coordination, hydrogen bonding, or both, potentially yielding novel structures and interesting spectroscopic properties. Another series of compounds containing boron was solved by Block and coworkers. These compounds included $NaB(OH)_4 \cdot 2H_2O$, $BaO \cdot B_2O_3$, and $PbO \cdot 2B_2O_3$. In collaboration with the FDA, another branch of studies included a series of biologically important compounds, including a toxic compound in Agent Orange.

Since 1951, Roth and collaborators have maintained a continuous effort in the study of the structures of a variety of niobate, tantalate and titanate dielectric materials, which have potential industrial applications. Other areas of interest included the ionic conductors and PZT compounds. Examples of such materials included $LiReO_3$, Li_2ReO_3 and $(Ti_{0.75}Pb_{0.25})_4Cl_5$. In recent years, another important area of research emphasizes the search for new microwave dielectrics for component miniaturization in wireless communications. Various crystals studied so far belong to the systems Ba-Ti-Fe-O, Sr-Nb-Ti-O, Ca-Nb-Al-O, and Sr-Ta-O. Attempts to correlate these structures with physical properties are also being pursued.

The 1986 discovery of high T_c superconductors placed a demand on the crystallographic studies of new phases. Compounds of interest mainly are in the Ba-R-Cu-O (R=lanthanides and Y) and Bi-Pb-Sr-Ca-Cu-O systems. Examples included detwinned $Ba_2YCu_3O_{6+x}$, Ca_2BiO_4 , $Bi_{14}(Sr,Ca)_{12}O_x$, and BaR_2CuO_5 . Combined x-ray single crystal and powder neutron studies were carried out to understand the detailed structures, particularly the oxygen content.

W. Wong-Ng, R.S. Roth, S. Block, C. Reimann, A. Santoro, V. L. Karen, C.R. Hubbard, A.D. Mighell and, T.A. Vanderah

Structural Studies of Biological Minerals in Dental Research

The crystallography program at the American Dental Association Health Foundation, Paffenbarger Research Center, National Institute of Standards and Technology was initiated in the early 1970s to provide a broad structural basis for understanding the various chemical phenomena associated with the tooth and bone minerals. Although x-ray powder diffraction was used extensively for the identification of materials and for the study of gross phase/structural changes, the major emphasis was on single crystal structural studies of biological minerals and related compounds associated with or with a potential bearing in mineralization processes. The primary importance was on calcium phosphates, but extended to calcium pyrophosphates, calcium carbonates and calcium bisphosphonates. A large number of highly hydrated magnesium and alkaline earth phosphates and arsenates were also investigated for comparative insights into nucleation phenomena.

Hydroxyapatite, $Ca_{10}(PO_4)_6(OH)_2$ (OHAp), has been

considered as the prototype for the tooth mineral. However, apatites as they occur in biological tissues, mineral formations and laboratory products can incorporate wide variety of impurities and are seldom found in pure stoichiometric form. We have determined the structures of a number of substituted apatites to evaluate the structural changes associated with the substitution. For example, in a lead apatite, an apparent covalent Pb-O bond may account for the incorporation of Pb into bone mineral. Octacalcium phosphate, $\text{Ca}_8\text{H}_2(\text{PO}_4)_6 \cdot 5\text{H}_2\text{O}$ (OCP), is one of the most important calcium phosphates in biological mineralization. The most important role of OCP in biological mineralization is probably that of a precursor to other phases found in biological settings, especially defect OHAp. OCP has a layer type structure, with one layer quite similar to that of apatite and the other, a hydrated layer consisting of more widely spaced Ca, and PO_4 ions and all the water molecules. The closeness of fit in the apatitic layer accounts for the epitaxial, interlayered mixtures formed by these two compounds which may account for the variation in Ca/P ratios in biological apatites. The crystal chemistry of OCP has been extensively investigated to illustrate how OCP affects the growth mechanisms, impurity-defect content, morphology, stoichiometry and other properties of OHAp. Dicarboxylate ions incorporated in the hydrated layer of OCP offers another mode of OCP involvement in biomineralization.

Our structural studies have shown that a number of calcium phosphates fall into class of glaserite compounds. Both α and β - $\text{Ca}_3(\text{PO}_4)_2$ belong to this class and can accommodate Mg ions in their structures affecting their relative stabilities. The mineral whitlockite, found in dental calculus, has a structure very similar to that of Mg-substituted β - $\text{Ca}_3(\text{PO}_4)_2$. The work was supported by NIDR grant DE05030 to ADAHF, and NIST.

Mathai Mathew and Shozo Takagi, ADAHF- PRC, Polymers Div.

Protein Crystallography at NIST

Protein crystallography came to NIST during the mid 1980s, just before the old Bureau of Standards got its new name and broader mission in 1988, and before the Biotechnology Division was established in 1991. NIST's neutron source provided a key initial impetus for protein crystallography, during an era when X-ray sources and detectors were still rare and unreliable. The opportunity for neutron studies of ribonuclease and insulin crystals helped to attract some of the first protein crystallographers to NIST, including Alex Wlodawer (now at NCI), David Davies (NIH), and Gary Gilliland, who was hired in 1986 and now heads the Biotech Division.

Another key development for protein crystallography at NIST was the creation of CARB, the Center for Advanced Research in Biotechnology. With a central concept of industry-driven protein structural research, CARB was born at NIST in 1985. By 1989 CARB had grown into an alliance between NIST and the U of Maryland Biotechnology Institute, with a new laboratory building built on land contributed by the county, 5 km south of NIST. Although an x-ray diffractometer for protein work operated at NIST for a few years during the late 1980s, ever since 1989 the CARB laboratory has housed the equipment for NIST

protein crystallography. This creates a minor inconvenience for researchers driving between the two campuses, but a rich cross-fertilization of ideas among the various NIST, university, and industrial elements.

Macromolecular crystallography is now an essential discipline of biology, and is increasingly important at NIST as groups outside of structural biology see growing industry involvement in biomolecular structure and seek structural knowledge regarding their own projects. NIST's recent involvement in the Protein Data Bank and expansion of its Biomolecular Crystallization Database functions underscores the increasing importance of this field to NIST. It is likely that future developments will include expanded research in areas such as the measurement of quality and accuracy of structures, measurements of basic physical processes underlying crystal growth, crystal freezing, and protein diffraction, and methods of structure determination.

Travis Gallagher, Biotechnology Division

Structural Studies in the NBS/NIST Polymers Division up to the Year 2000

Most scientists, let alone the general public, are unaware that the synthetic polymers (commonly called "plastics") that are ubiquitous in our everyday lives are nanocomposites made up of tiny crystallites embedded in a matrix of amorphous material. The industry that tailors materials properties by using sophisticated processing to define crystallinity and microstructure is a huge one. For this reason the characterization of microstructure has been an important ongoing activity within the NBS/NIST Polymers Division since its earliest times. The level of effort devoted to structural studies has waxed and waned with changing fashions in polymer research.

There have been only a couple of instances in polymeric materials where "conventional" crystallographic methods have been applied to define atomic arrangements within unit cells. Crystal structure has been an important input to studies in spectroscopy and physical properties of a variety of polymeric.

Following upon the heels of the discovery of chain folding in solution-grown polymer single crystals, the 1960s and 70s saw considerable NBS activity aimed at defining the kinetic behavior of the crystallization process and the resulting morphology. In the 1980s x-ray facilities were commissioned to examine structure-property relationships using wide-angle orientation texture and small-angle scattering. At about the same time small-angle neutron scattering emerged as a tool of choice for characterizing phase behavior in block copolymers, semicrystalline polymers, and molecular structure in interpenetrating polymer networks and hyperbranched polymers. More recent times have seen the beginnings of work using synchrotron SAXS. The challenges that will face the polymer materials science community in the second century of NIST's history cannot yet be foreseen, but one can be certain that scattering and diffraction methods will be as vital as ever.

John D. Barnes, Polymers Division

Databases and Computation in Modern Structural Science

Dreams are important in driving new technology, and materials design is no exception. Today advanced computational technology is advancing our dream of designing new materials from first principles. Or is it? I would argue that the dream is not to assemble 10^{22} to 10^{23} atoms of various quantities, and let the computers determine optimal structure and derive properties from that structure. Instead, I believe the dream for the coming decades is to couple computer power with comprehensive structural databases and data exploitation (data mining) methods to develop new leads for materials to be optimized according to the requirements of specific applications

Materials design and development rarely starts with a blank computer screen. The projected application guides us to certain materials classes through experience, broad knowledge of material potentiality and creative thinking. The process almost always involves a general or specific application. Modern material science has opened the range of possibilities considerably over the last thirty years. Composites, advanced processing and finishing, and new manufacturing techniques are examples. At the same time, new approaches, such as materials selector software, open new avenues of interest. The ability to screen materials classes on the basis of a few desired properties is proving to be quite popular. Yet the process of materials design does not stop with the selection of a traditional or new material. Almost always, there is a need to optimize some aspect of a selected material. The considerations can take place at many different levels – atomic, cluster, microstructure, surface, etc. Where do modern structural databases fit in with this scheme? Where does computation support it?

Modern structural databases (e.g., the Inorganic Crystal Structure Database (ICSD)) provide a comprehensive and quality-controlled depository of data on the full structure of crystalline compounds. The ICSD now contains structural data on over 50,000 inorganic compounds that usually fall into a few structure types. Searching for new materials leads is facilitated by investigations of compounds in the same structure type. However, we can go further. Using new data mining techniques, the ICSD may yield additional structure-property correlations beyond the current structure type classifications. The real purpose of both database exploitation and computation is not to create data for every possible structure, but to generate ideas for structures in general, and specific structures as needed. The design and development of new engineering materials is a process of getting new ideas and gradually transforming those ideas into a real material, suitable for an application. Structural databases and structure computation will soon replace experimentation as the major source for the new ideas. Indeed, the real materials of tomorrow will come from the virtual materials of today.

John Rumble, Jr., Standard Reference Data Program

Crystallographic Data in the NIST Standard Reference Data Program

All fields of experimental science generate numerical data in some form. Since the useful lifetime of these data is very long, and the volume of production continues to accelerate, data management is a significant issue. It is wasteful to repeat

measurements out of ignorance of what has already been measured and reported in the literature. Intelligent planning of new research often depends on a foundation of existing data. Thus every field of science should be concerned with the collection and organization of the data it produces.

Crystallographers have been at the forefront of science in this regard. Over the years, many outstanding crystallographers have dedicated portions of their careers to the exacting and unglamorous task of systematizing the data on crystal structures. In particular, the crystallographic community has led the way in applying modern computers to scientific data management. Today, a vast amount of crystallographic data can be accessed quickly and reliably by electronic means.

The National Bureau of Standards (now the National Institute of Standards and Technology) has played a key role in this process. The NBS Crystal Data Center was one of the charter data centers when NBS established the Standard Reference Data program in the mid 1960s. Under the SRD program, a productive collaboration between NBS and the Cambridge Crystallographic Data Centre has continued for more than 30 years. A close collaboration between NBS and the Joint Committee on Powder Diffraction Standards/International Centre for Diffraction Data also continues. NBS/NIST crystallographers have made important contributions to the development of software for evaluation and retrieval of crystal data. The Single Crystal (nonstructural) Database that they maintain is a comprehensive scientific numerical database with crystallographic data on all classes of materials. Most recently, the NIST Standard Reference Data program has joined in collaborative efforts with respect to the Inorganic Crystal Structure Database and the Protein Data Bank. Crystallography has been a key element of the SRD program and will undoubtedly continue to be in the future.

David R. Lide

The Inorganic Crystal Structure Database

Crystallography has a long and successful history of self-organization and was one of the first areas to create numerical scientific databases. Virtually all structure determinations have been archived in databases that allow ready access and complete coverage. Crystallographic databases and computational archives support research on a daily basis for thousands of scientists worldwide. The crystalline structure of inorganic materials is of interest to analysts in areas such as materials design, properties prediction, and compound identification.

The Inorganic Crystal Structure Database (ICSD) contains full structural and bibliographic information for all structures from 1915 through the present. There are more than 50,000 entries in the current version and approximately 2500 new entries are added per year. Data items include bibliographic information, such as the article title, authors' names and literature citation; compound designation, such as chemical name, chemical formula, mineral name; and crystallographic parameters, such as unit cell, space group, element symbol with numbering, oxidation state, multiplicity for Wyckoff position, x,y,z coordinates, site occupation, thermal parameters and reliability index R , among others. The data are evaluated by experts in specific disciplines and by specialized computer programs.

Several types of evaluation are performed, including examination of an individual data item, looking for consistency within a complete entry, and checking the relationship of an individual entry to the entire database.

Over recent years, FIZ and NIST have been building a modern infrastructure for the Inorganic Crystal Structure Database. This has included a complete re-design of the ICSD database structure, conversion and loading of the data into a relational database management system, designing graphical user interfaces to access the data, and creating scientific application modules to analyze the results of a database search. The ICSD is marketed through a variety of computerized media which include both PC and Web-accessible versions of the database. The new Windows-based graphical user interface for the ICSD is tabular in design, allows for searching in five general categories of Chemistry, Crystal Data, Reduced Cell, and Reference Data, and will include enhanced features for the characterization of materials based on lattice and chemistry search modules, and 3-dimensional visualization and powder pattern simulation of inorganic structures.

Anticipating the needs of the materials community in both science and industry, the ICSD is helping scientists use crystallographic data models to visualize, explain and predict behavior of chemicals and materials. With increasing use comes an increasing range of computational techniques to analyze and correlate data, and to help researchers concentrate experimental work in directions that optimize the discovery process. Interoperability with other data sources and software tools appears to be one of the emerging driving forces for innovation today.

The Inorganic Crystal Structure Database is produced cooperatively by the Fachinformationszentrum (FIZ) (Karlsruhe <http://www.fiz-karlsruhe.de/>) and NIST (<http://www.nist.gov/srd/>)

Vicky Lynn Karen, Materials Science and Engineering Laboratory

NIST Materials Properties Databases for Advanced Ceramics

The anisotropy of any physical property of a single crystal must be consistent with the observed symmetry in the physical structure of the crystal. By extension, anisotropy in the properties of polycrystalline materials should be correlated with the degree of texturing in the sintered body. Both crystal structure and texturing have important consequences for the behavior of materials subjected to external stimuli (temperature, pressure, and electromagnetic fields). Furthermore, structural data from crystallographic studies can be used to determine the coefficients of thermal expansion which, in turn, can be used in the evaluations of axial and volumetric derivatives of physical properties. Consequently, crystallography plays a central role in classifying and understanding the physical behavior of solid materials, and the associated data form a useful basis from which to study and pursue the development of new materials.

As a result, the NIST Standard Reference Databases (SRD) for high temperature superconductors (HTS, SRD Number 62) and structural ceramics (SCD, SRD Number 30) include a full complement of crystallographic data (crystal system, space group,

the unit cell parameters, the relative atomic coordinates x , y , and z , and site occupancy numbers), along with the principal thermal (expansion, conductivity, diffusivity, and specific heat) and mechanical (elasticity, strength, hardness, toughness, and creep) properties. The HTS database also includes electromagnetic (resistivity, surface resistance, magnetic susceptibility, penetration depth, Hall coefficient, and thermoelectric power) and critical superconductor (T_c , J_c , H_{c1} , H_{c2} , and H_{c3}) properties.

The crystallographic data serve several functions in these databases. The data sets themselves are, of course, part of the reference information characterizing the materials and may be used for purposes of identification. Conversely, crystallographic data may be specified as part of the criteria when searching the database for properties of particular materials. These data also are used both in data evaluation efforts, to help ensure that only data from comparable materials are being analyzed together, and in developing or applying models of material behavior. Many physical properties depend significantly on the phase compositions of the constituent particles, the interface or grain boundary regions, and the possible surface layers. Similarly, the size, shape, and distribution of pores, which may be treated formally as a secondary phase, can have a dramatic influence on property values. Crystallographic data often provide the key to understanding the effects of phase composition and porosity in terms of structural features in the material.

R. G. Munro, Ceramics Division

Phase Equilibria Research

Since the 1930s, NIST has collaborated with various industries on phase equilibria research. One of the first phase equilibria studies at NIST was by McMurdie et al. on the relation of MgO to Portland cement compositions. This was followed by the study of the PbO-SiO₂ system for its relation to glazes for whiteware.

Roth and his coworkers started phase equilibria studies of dielectric materials in the 1950s, with focus on the tantalate, niobate, titanate and tungstate systems, and their chemistry with rare-earth oxides. Phase relations have also been determined in the technologically important PZT-containing (PbZrO₃-PbTiO₃) system. A number of phases in the Bi₂O₃-TiO₂, Bi₂O₃-SiO₂ systems later became sought-after piezoelectric materials. Since the mid-nineties, the emphasis has been shifted to microwave dielectric materials (Vanderah and Roth). Systems being investigated included BaO-Fe₂O₃-TiO₂, SrO-TiO₂-Nb₂O₅, SrO-Al₂O₃-Nb₂O₅, and CaO-Al₂O₃-Nb₂O₅. Among the new phases discovered, a significant finding pertains to the dielectric properties of compositions prepared between Ca₃Nb₂O₈ and Ca₂AlNbO₆, which have relatively high dielectric constants and quality factors, and can be tuned to having near-zero temperature coefficients.

Since 1986, NIST has been involved with the EPRI and later the DOE programs in phase equilibria research of high T_c superconductor systems. Following Roth's studies of the Ba-Y-Cu-O system, Wong-Ng and Cook investigated the phase transformation of BaR₂Cu₃O_{6+x} (R = lanthanides and Y), and also the phase diagrams in the AO-R₂O₃-CuO (A = Ba, Sr) systems which are related to the development of coated

conductors. To provide basic information for the wire and tape development using Ag powder-in-tube techniques, multi-component phase diagrams of the Bi-Pb-Sr-Ca-Cu-Ag-O system have been successfully studied.

The compilation of published phase diagrams was started in 1933 and it was published by the American Ceramic Society (ACerS). This started a continuing relation between NBS and ACerS and has resulted in the publication (Roth, Cook, Ondik, and Vanderah) of 12 volumes of compilations and several volumes in special areas.

R.S. Roth, H.F. McMurdie, T.A. Vanderah, H. Ondik, L.P. Cook, and W. Wong-Ng

The NIST/CARB Biological Macromolecule Crystallization Database

The Biological Macromolecule Crystallization Database (BMCD) was developed at NIST to assist crystallographers in developing strategies for crystallizing biological macromolecules. The database summarizes the information concerning crystallization that is available in the literature. The BMCD was established in 1989 with assistance from the National Institute of Standards and Technology (NIST) Standard Reference Data Program [Gilliland, G. L. (1988). *J. Crystal Growth* 90, 51-59]. At this time, the BMCD software was developed as an independent program for personal computers (PCs). Access to the data was provided through menu-driven software that allowed browsing and searching of the crystallization conditions of 1025 crystal forms of 616 biological macromolecules. The information, which included most of the crystallization protocols of biological macromolecules in the literature through the end of 1982, had been previously deposited in a formatted ASCII file in the Protein Data Bank.

In 1991 the second version of the BMCD software and data was released. The data included 1465 crystal forms of 924 biological macromolecules. In 1994 the BMCD became the NASA Protein Crystal Growth (PCG) Archive and incorporated data from crystal growth studies supported by NASA. The new software and data were released as Version 3.0 of the BMCD. The number of crystal entries was increased to 2218 for 1465 biological macromolecules.

Version 3.0 of the BMCD was subsequently ported to a UNIX platform to take advantage of the development of network capabilities that employ client-server tools. The first web implementation of the BMCD provided many of the features of the earlier PC versions of the BMCD. The searching capabilities and the data were increased in release 2.0 of the BMCD web software that now includes 3546 crystal forms for 2526 macromolecules. The current web implementation is available at <http://wwwbmcd.nist.gov:8080/bmcd/bmcd.html>. To facilitate the updating process, the current implementation provides a beta-test version of software that users can employ to submit their crystallization data directly to the BMCD. The BMCD's primary goal will continue to be an error-free and up-to-date crystallization database. The availability of the NIST BMCD on the Internet will facilitate the distribution and error correction of the data. Alternative ways for users to search, use and display the

data will continue to be added making it a useful resource for the macromolecular crystallographer.

Gary L. Gilliland, Michael Tung, and Jane E. Ladner

The NBS-JCPDS/ICDD Research Associateship Program

The NBS-JCPDS/ICDD Research Associateship program at NBS/NIST was a long standing (1949-1986) successful industry-government cooperation to produce high quality x-ray powder diffraction reference patterns to be included in the Powder Diffraction File (PDF). Today the JCPDS/ICDD is known as the International Centre for Diffraction Data (ICDD). The PDF is a continuing compilation and the only comprehensive source of reference patterns gathered from many sources, and is widely used for phase identification and materials characterization.

The Associateship was established at NBS/NIST in July 18, 1949 to prepare high quality powder x-ray diffraction reference standards. In the early years, the Associateship was directed by H.F. McMurdie, and in the 70s and 80s by C.R. Hubbard. Their leadership ensured the quality of the patterns, as well development of state-of-the-art computer technology and x-ray diffraction instrumentation. The standard patterns were produced –by M.Morris, E. Evans, J. DeGroot, H. Swanson and others– using reproducible, controlled conditions of specimen preparation and the best recording instrumentation available. The consistent high quality of the patterns produced has been an integral foundation of the Powder Diffraction File (PDF). These patterns were also published in the NBS Monographs (21 volumes), products derived from the PDF, as well as the early issues of the *Powder Diffraction*. The Associateship was also involved in the implementation and testing of the computer software (led by Hubbard, A.D. Mighell, J.K. Stalick and N. Pyrrros) necessary for data collection and reduction, least-squares refinements and editorial processes. Concurrent with the Associateship efforts NBS also certified a number of Standard Reference Materials for use as internal standards and for improving powder diffraction analysis (SRM640, 640a, 640b, 660, 676, and 1874).

With the expanding capability for others to accurately measure and report XRD reference patterns, the mission of the JCPDS/ICDD Research Associate program was accomplished in 1986. A three-year program at NBS to produce high quality x-ray patterns of important ceramic phases followed. Further collaborations continue through the ICDD Grant-in-Aid program (W. Wong-Ng, and J. Kaduk of BP-Amoco), with emphasis on materials for electronic applications.

H.F. McMurdie, C.R. Hubbard (ORNL), W. Wong-Ng

Standard Reference Material™ for Single Crystal Diffractometer Alignment

The importance of the investigation of the accuracy and precision of lattice parameters measured in the industrial, academic and government x-ray laboratories were recognized by the IUCr, ACA, Hauptman-Woodward Medical Research Institute (HWI), and NIST. As a result, two related projects have been developed: an international round robin study of a potential

alignment standard, and the certification of this material as a standard Reference Material™ (SRM).

The international round robin project was launched by distributing to various laboratories 50 sets of a ruby sphere along with a zeolite reference crystal through the Hauptman-Woodward Medical Research Institute. The results of this project confirmed that well-aligned diffractometers are important for obtaining accurate lattice parameters, and that the ruby spheres satisfy the



W. Wong-Ng, T. Siegrist (Lucent), G. DeTitta (HWI), L. Finger (Geophysical Lab), H. Evans (US Geological Survey), E. Gabe, G. Enright (NRC, Ottawa), J. Armstrong, M. Levenson, L.P. Cook, and C.R. Hubbard (ORNL)

criteria required of a SRM. Through extensive international collaborations, the ruby sphere SRM1990 has been certified. The primary technical objective of this SRM was to provide world-wide laboratories with an alignment standard to improve the accuracy of lattice parameter determination. These rubies are rhombohedral, with space group R3c. The certified room-temperature unit cell parameters ($a/\text{Å} = 4.76080 \pm 0.00029$, and $c/\text{Å} = 12.99568 \pm 0.00087$) agree with the round robin data and are also supported by the Guinier-Hägg transmission data. The results of this work are expected to have a significant impact on accurate scientific investigations using single crystal diffractometers. These standards will be available in the near future at the SRM Office of NIST. The Web site for information is <http://www.nist/srm>

A Natural Bone Cement – A Laboratory Novelty Led to the Development of Revolutionary New Biomaterials

In the early 1980s, Brown and Chow of the American Dental Association Health Foundation Paffenbarger Research Center (PRC) at the National Institute of Standards and Technology (NIST) conducted studies on calcium phosphates aimed at developing remineralizing pastes for repairing early dental carious lesions. Based on the solubility properties of calcium phosphates, they formulated mixtures containing tetracalcium phosphate (TTCP) and dicalcium phosphate anhydrous (DCPA) or TTCP and dicalcium phosphate

dihydrate. Upon addition of water, these mixtures would rapidly form hydroxyapatite (HA), the major component of tooth mineral, at body temperatures. It was observed that some of the pastes became a hardened mass when left in test tubes for a few hours. Thus, the PRC scientists inadvertently discovered a new type of self-hardening cements that consisted of only calcium phosphates and formed HA as the only product.

By optimizing the particle size distributions of the cement ingredients, the scientists were able to develop cements with setting times of about 30 min and compressive strengths of 34 MPa (WE Brown and LC Chow, *Cements Research Progress* 1986, pp352 (1987)). From animal studies conducted at Northwestern University, Costantino and coworkers (PD Costantino et al., *Arch Otolaryngol Head Neck Surg* 117, 385(1991)) found that when implanted in bone defects, CPC was gradually resorbed and replaced by new bone. Dense and porous ceramic HA materials, which have good biocompatibility, have been used clinically for repairing bone defects. However, because these materials do not resorb, they must be used in granular forms to allow a blend of soft and bone tissues to form interstitially with the HA granules to achieve stable implant-tissue integration. This has limited the use of ceramic HA to repairing small defects in bone. In contrast, because of its self-hardening and *in vivo* resorption properties, CPC has the potential to be useful in a much wider range of clinical applications.

Continued research conducted by PRC scientists at NIST has resulted in many significant improvements in CPC. In July 1996, a CPC consisting of TTCP + DCPA was approved by the Food and Drug Administration for repair of cranial defects in humans, thus becoming the first material of its kind available for clinical use. CPC has become a subject of great interest to many scientists and clinicians worldwide, and several additional CPC products are now commercially available. With continuing improvements in cement properties and understanding of material-tissue interactions under various clinical situations, different CPC formulations with properties optimized for specific clinical applications are being developed.

Laurence C. Chow and Shozo Takagi, ADAHF-PRC, Polymers Div.

Quasicrystals

When, in 1982, Dan Shechtman produced an electron diffraction pattern from a rapidly solidified alloy with sharp spots that apparently had icosahedral point group symmetry, $m-35$, other members of the NBS staff were skeptical. After all, sharp diffraction is the consequence of periodicity. Conversely, it was thought impossible to have sharp diffraction from any aperiodic structure, and no structure with icosahedral symmetry could be periodic. Shechtman's pattern had to be produced by some peculiar kind of twin. But Shechtman had conducted several other careful experiments that ruled out any possibility that the observations could be explained by twinning. (Unless you believe that, for example, hexagonal close packed structures exist only as a periodic twinning of a face centered cubic structure.)

The level of skepticism was much reduced, within the NBS staff at least, if not in the broader crystallographic community, when it was demonstrated mathematically that there could be an underlying quasiperiodicity in the so-called Fibonacci sequence,

a discrete Fourier transform where the “orders” were not integers, but rather powers of the “golden mean,” $\tau = (1 + \sqrt{5})/2 = 2 \cos 36^\circ$. Further, it was demonstrated theoretically and confirmed experimentally that the aperiodic Penrose tilings would produce optical diffraction patterns with sharp spots having fivefold rotation symmetry. The mathematics of pentagonal or icosahedral quasiperiodicity predicts that the diffraction spots go out in reciprocal space by successive factors of τ (in contrast to what would be expected from twinning) and get stronger, exactly what is observed experimentally. One of the important characteristics of quasiperiodicity is that it can always be described as periodic in a higher dimension, and diffract discretely in that and lower dimensions. The skeptics notwithstanding, thousands of papers, studying more than a hundred different metallic systems, have established not only that quasicrystals exist, but that they occur with other “noncrystallographic” symmetries, including five-, eight-, ten-, and twelve-fold axial systems, and that large stable quasicrystals can be grown with a high degree of perfection.

Many metallic systems exhibit sequences of periodic approximants with ever larger unit cells that converge to the observed true quasicrystals. The Patterson functions of all these structures are quite similar to one another and to those of the true quasicrystals. A number of “quasicrystal structure determinations” have been made, using methods of hyperspace crystallography. A few years ago the IUCr changed the definition of crystal based on discrete diffraction, so that these “quasiperiodic crystals,” or quasicrystals for short, are now included among crystals. There continue to be extensive studies of this fascinating extension of crystallography.

J. W. Cahn and E. Prince, Materials Science and Engineering Laboratory

The Fundamental Role of Reduced Cells in Crystallography

In theory, physical crystals can be represented by idealized mathematical lattices. Under appropriate conditions, these representations can be used for a variety of purposes such as identifying, classifying, and understanding the physical properties of materials. Critical to these applications is the ability to construct a unique representation of the lattice. The vital link that enabled this theory to be realized in practice was provided by the 1970 paper on the determination of reduced cells by A. Santoro and A. Mighell (*Acta Cryst.* A26, 124 (1970)). This seminal paper led to a mathematical approach to lattice analysis initially based on a systematic reduction procedure and the use of standard cells. Subsequently, the process evolved to a matrix approach based on group theory and linear algebra that offered a more abstract and powerful way to look at lattices and their properties.

Application of the reduced cell to both our database work and our laboratory research at NIST was immediately successful. Currently, this cell is widely used: (i) as a standard cell for the reporting of triclinic crystals, (ii) in crystallographic database work (iii) in symmetry determination because of the close link between metric and crystal symmetry (A. Mighell, A. Santoro, and J. D. H. Donnay, *International Tables for X-ray Crystallography*, Vol. 1, 530 (1969)) and *Acta Cryst.* A36, 321(1980)), and (iv) for identifying crystalline materials (A.

Mighell, *J. Appl. Cryst.* 9, 491 (1976)). Today the scientific community routinely uses this identification strategy, as it has been integrated into commercial X-ray diffractometers.

Due somewhat to serendipity, the most significant and lasting value of this work is probably not reduction itself. Rather, reduction has played a key transition role in helping to move the discipline of crystallography in new directions with new insights. The research on reduction proved that there are excellent reasons for looking at the crystal lattice from an entirely different point of view. Consequently, with time, many other lattice-related papers followed, including papers on sublattices and superlattices, composite lattices, coincidence site lattices, and lattice-metric singularities in the indexing of powder patterns. At NIST, the mathematical analysis of lattices was pursued further and evolved to a matrix approach that offered a more abstract and powerful way to look at lattices and their properties. The matrix approach, in particular, has many applications, including for example, symmetry determination (V. L. Karen (Himes) and A. D. Mighell, *Acta Cryst.* A43, 375 (1987)).

*Alan D. Mighell, Materials Science and Engineering Laboratory
Ceramics Texture Research at NIST*

The impetus for texture research in the Ceramics Division came from American Superconductor Corporation, Westborough, MA, (ASC) in 1994. During product development, ASC needed to do rapid, accurate crystallographic texture measurements on the high temperature superconducting wires (or “tapes”) they were manufacturing. Their goal was to correlate the observed texture with measured electrical and mechanical properties, which are known to be strongly influenced by texture. A major requirement was that the texture measurement technique could be performed using the conventional x-ray powder diffractometer available at ASC. A technique was developed at NIST for quantitative measurement of texture using scans performed on a conventional diffractometer; the scans can be obtained in about one hour. The data analysis method uses the technique of calculating the hkl omega scan from an untextured sample of the material being tested using a θ -2 θ scan of the sample over the hkl peak. The experimental omega scan from the textured sample is divided by the calculated omega scan to give the texture profile of the sample. The technique is best suited to the analysis of fiber texture. The software to perform the calculations of the analysis, TexturePlus, was developed at NIST. The technology was validated at NIST using SRM676 (untextured alumina) and successfully transferred to ASC in 1995.

It became clear that there were many potential users of this method in industry, particularly small businesses, and academia. As part of the thin film program, the technique was extended to analysis of diffraction data from thin films, where the thickness and linear x-ray absorption coefficient of the film were necessary input data. Validation of the thin film correction was achieved in 1998 with data obtained from electrodeposited films of copper, which are being introduced into chip interconnection technology as a replacement for aluminum. The software package TexturePlus was made available on the World Wide Web in 1999 on <http://www.ceramics.nist.gov/webbook/TexturePlus/texture.htm>. A workshop on Texture in Electronic Applications was held at NIST Gaithersburg in October 2000, attended by

about 40 researchers from industry, academia and national laboratories. At this meeting, the need for NIST to facilitate interlaboratory comparisons became apparent and will be the focus of future efforts.

Mark D. Vaudin, Ceramics Division

Crystallographic Support for the Free Radical Research Program

In 1956, the imaginative Herbert Broida, backed by the steady experimenter, Arnold Bass, proposed a basic research program to investigate atoms or very small molecules, with an unpaired electron in their outer shells, trapped in a solid matrix at low temperatures. The result of this proposal was the NBS Free Radical Program. Its concepts were supported not only by the NBS management, but also by the Department of the Army, as funding agency, as well as by several private-sector companies. Within the three-year duration of the Program, more than a hundred publications were produced.

Howard McMurdie, leading the NBS Crystallographic Section, responded by making equipment and personnel available. A liquid-helium cooled sample holder was built and fitted with beryllium windows to permit soft X-rays powder data to be recorded by a diffractometer. The detailed description of this apparatus by F.A. Mauer (in A.M. Bass and H.P. Broida, *Formation and Trapping of Free Radicals*, chapter 5, 117 - 167, Academic Press, New York and London (1960)) remains a good "beginners" introduction to low-temperature crystallography with sections on thermometry, calibration, vacuum technique, and gas handling features.

The X-ray diffraction studies and their crystal-chemical implications are described by H.S. Peiser (in A.M. Bass and H.P. Broida, *Formation and Trapping of Free Radicals*, chapter 9, 301 - 326, Academic Press, New York and London (1960)). At the outset he stressed the fundamental limitations: small concentrations of free radicals, the small diffracting power of hydrogen and the use of powder diffraction with limited texture-determining features. Nevertheless, the crystal structures of the matrices could thus be recorded and any influence of the free radicals was likely to be observable during deposition of the radicals and on warm-up of the conglomerate, when strongly exothermic reactions occurred. Pure argon and nitrogen deposited at 4.2 K showed considerable line broadening, indicating disordering which disappeared exothermically on annealing with formation of good crystals. Their structures were retained on re-cooling, enabling good cell-edge and coefficient-of-expansion measurements to be recorded. Evidence for a reported lack of a center of symmetry in cubic α -nitrogen was absent. The evidence supported the space group $Pa\bar{3}$ with $a/\text{\AA} = 5.644 \pm 0.005$.

H. Steffen Peiser

Crystallography of Construction Materials

Portland cement concrete over the past 100 years has become a ubiquitous construction material, with an annual consumption

at about 5 Pg (5.5 billion tons), second only to water in per-capita demand. The flexibility in application and speed of construction afforded by concrete has resulted in it largely supplanting other materials in construction of pavements, homes, and high-rise buildings. Improved understanding of the materials science of portland cement and concrete has come through increased knowledge of the chemistry and crystallography of the constituent materials; the clinker phases, cements, and cement hydration products.

NIST/NBS researchers have played an integral part in developing this understanding of portland cements. Work on the chemistry of portland cement clinker phases at NBS in the early 20th century (by P.H. Bates and A.A. Klein, Bureau of Standards Technol. Papers No. 78, 1917) demonstrated that $3\text{CaO}\cdot\text{SiO}_2$, or alite, was one of the primary constituents, as postulated by Henri LeChatelier. They also investigated the hydraulic properties of cement compounds in one of the first studies that addressed the relationships between cement performance and composition. Around 1925, the Portland Cement Association (PCA) sponsored a Fellowship at NBS that lasted until the mid-1960s. Researchers at NBS, including those who held the PCA Fellowship, were instrumental in forming the foundation of today's knowledge of portland cement chemistry.

Examination of the relationships between clinker phase constitution, individual phase solid solution, and reactivity of cements were a continuing theme of the work at NBS. At the same time, the chemistry and structure of hydration products were the subject of many studies. Work today at NIST is directed toward understanding the relationships between clinker composition and cement performance, and to better understand the newer blended cement systems. These studies build on the long history of cement science at NBS/NIST over the last century, combining them with recent computer modeling technology to deliver a virtual cement and concrete laboratory to the desktop that will aid in evaluating and optimizing cement-based materials.

Paul Stutzman, Building Materials Division

Positions Available

It is expected that the employers listed in this publication are equal opportunity employers who wish to receive applications from qualified persons regardless of age, national origin, race, religion, sex or physical handicaps. Please inform the Editor when the positions are filled, and of any positions that do not give opportunities to all applicants. Ads will appear in two successive newsletters unless the Editor is notified that the advertisement should be continued longer or discontinued earlier. For the most up-to-date listings check the ACA Home Page under the Positions Vacant heading.

<http://www.hwi.buffalo.edu/ACA/>

Inorganic Chemist – Crystallographer

A post-doctoral position is available for a person skilled in both inorganic synthesis and X-ray crystal structure determinations. A variety of organic-inorganic hybrids and crystal-engineered solids are to be synthesized, crystallized and their structures determined [see Sharma and Clearfield, *J. Am. Chem. Soc.* **122**, 1558 (2000); **122**, 4394 (2000)]. Our laboratory is well equipped with CCD Smart systems, automated diffractometers and two powder diffractometers. Four state of the art instruments will be added as a result of a new NSF Grant. Opportunities to learn structure solutions from X-ray powder data will be available. Send resume and a letter of recommendation to: Prof. Abraham Clearfield, Department of Chemistry, Texas A&M University, P.O. Box 30012, College Station, Texas 77842-3012, Clearfield@mail.chem.tamu.edu

Biological Macromolecular Crystallographer

The Biomolecular Structure Teaching and Research program of the University of Saskatchewan is seeking a highly qualified X-ray crystallographer to fill a tenure-track assistant professor position in the Department of Chemistry, with associate membership in the Department of Biochemistry. Candidates will possess a Ph.D. and relevant research experience, and should have a strong commitment to teaching and to developing a vigorous and creative research program. Further information about this position and the departments can be found on the Internet at addresses www.usask.ca/chemistry/ and www.usask.ca/medicine/biochemistry/.

Meetings Calendar

JUNE 2001

8-20 ACA Summer Course in Crystallography, Athens, GA. Georgia Center for Continuing Education, University of Georgia, <http://BCL15.bmb.uga.edu/aca2k.html>

JULY 2001

21-26 ACA '01 Los Angeles, CA. Local Chairs: Katherine Kantardjieff (CSU-Fullerton, kkantardjieff@exchange.fullerton.edu) and Dan Anderson (UCLA, dha@mbi.ucla.edu). Program Chair: Duncan McRee (Syrrex, dmcree@syrrex.com); website: www.hwi.buffalo.edu/ACA/ACA-Annual/LosAngeles/

JULY 2001

30 - Aug 5 13th International Conference on Crystal Growth, Kyoto, Japan: <http://iccg.gakushuin.ac.jp>

AUGUST 2001

12-16 13th American Conference on Crystal Growth and Epitaxy (ACCGE-13), Burlington, VT; there will be a joint session with the ACA: X-ray Diffraction for Crystal Perfection and Growth; website: www.crystalgrowth.org/conferences/accg13

SEPTEMBER 2001

23 - 28 Second International Workshop on Physical Characterization of Pharmaceutical Solids (IWPCPS-2), Lancaster, PA. Co-supported by ASSC, Glaxo Wellcome and the Cambridge Crystallographic Data Centre. Details: Angeline Zakrzewski (angeline@ASSCI.com), ASSC; Fax: (610) 594-2082; website: www.assci.com

MAY 2002

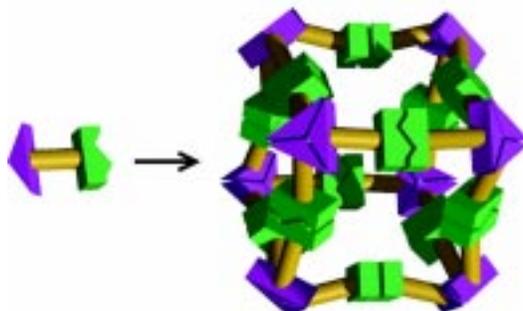
25-30 ACA '02 San Antonio, TX. Local Chairs: Ray Davis (UT Austin) and Marv Hackert (UT Austin, m.hackert@mail.utexas.edu). Program Chairs: Wally Cordes (Arkansas, wcordes@comp.uark.edu) and Travis Gallagher (travis.gallagher@nist.gov).

JULY 2002

31 -Aug 5 XIIth International Symposium on Supramolecular Chemistry (ISSC XII). Jerusalem, Israel. Contact: Israel Goldberg, Tel-Aviv Univ; e-mail: goldberg@post.tau.ac.il; <http://chemsg7.tau.ac.il/~issc/>

AUGUST 2002

6-15 19th IUCr General Assembly and Intl. Congress of Crystallography. Jerusalem, Israel. Secretariat : XIX IUCr P.O. Box 50006, Tel Aviv 61500, Israel; Fax: 972 3 5175674 or 972 3 5140077; e-mail: iucr@kenes.com



Beamtime Available to Independent Investigators

The Industrial Macromolecular Crystallography Association Collaborative Access Team (IMCA-CAT) announces the initiation of its Independent Investigator Program on its undulator insertion-device beamline, 17-ID, beginning October 1, 2000. The beamline is currently suitable for monochromatic data collections in a wavelength range of 0.82 to 2.4Å, and semi-automated data collections at multiple wavelengths for MAD experiments. If you are an independent investigator seeking beam time for an individual macromolecular crystal structure project, or represent a collection of investigators undertaking a larger program of macromolecular structure determinations, such as multiple investigators from a single institution, or a structural genomics initiative, you may submit an application via the APS at http://www.aps.anl.gov/xfd/communicator/useroffice/II_proposal.html. For further information, please contact Dr. Andrew J. Howard, CAT Director, at howard@iit.edu or 630-252-0534.

Oxford Cryosystems Comes to North America

Oxford Cryosystems Ltd is pleased to announce the opening of a new Sales and Service Center in North America. Oxford Cryosystems Inc has been set up to provide the highest quality sales and technical support for the Cryostream Cooler, now in use in hundreds of laboratories across the region.

The new facility will be run by expert sales and technical staff who will provide the latest support to all Cryostream Cooler customers as well as all other OxfordCryosystems products. Oxford Cryosystems Inc can be contacted at: Oxford Cryosystems Inc, 220 Wood Rd., Braintree, MA 02184-2403, Toll free: 1-866-OXCRY08, Fax: (781) 843-5945, info@OxfordCryosystems.com, <http://www.OxfordCryosystems.com>

ACA "no chad" Electronic Balloting

In 1999 the ACA membership authorized a modification to the by-laws to allow electronic balloting in the ACA election. At the ACA meeting in July 2000 Connie Chidester created an *ad hoc* Web Advisory Committee to implement electronic balloting and address other web issues. The new committee consists of Jeffrey Deschamps (chair, Naval Research Lab), James Fait (Argonne National Lab), and John Westbrook (Rutgers). The committee considered security, authentication, and privacy issues before recommending a strategy to implement electronic balloting. The final result represented a compromise between ease of implementation and bulletproof security. By combining encryption (via SSL) with unique user names and passwords we could ensure that short of a hacker attack on the server itself only ACA members could submit a ballot.

The turn out for the most recent election was about 25% greater than it was in 1999. Of the 597 ballots received 219 (almost 37%) were electronic. While some minor problems were encountered with electronic balloting (i.e. confusion on where the member number should appear) the process went smoothly and electronic ballots streamlined the counting process. If you didn't use the electronic ballot please consider it next year. Any comments or suggestions on the electronic ballot or other web issues can be sent to deschamps@nrl.navy.mil.

Jeff Deschamps

Reduced Rate Subscriptions for JSB

The Journal of Structural Biology (www.academicpress.com/jsb) would like to offer members of the ACA a reduced rate personal subscription of \$125 for 12 issues. The regular personal rate is \$343 (N. America) / \$394 (Rest of World).

Statement required by 39 U. S. C. 3685 showing the Ownership, Management and Circulation of ACA Newsletter, published four times per year for October 1, 2000. Publication No. 1958-9945. Annual subscription price is \$1.75

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