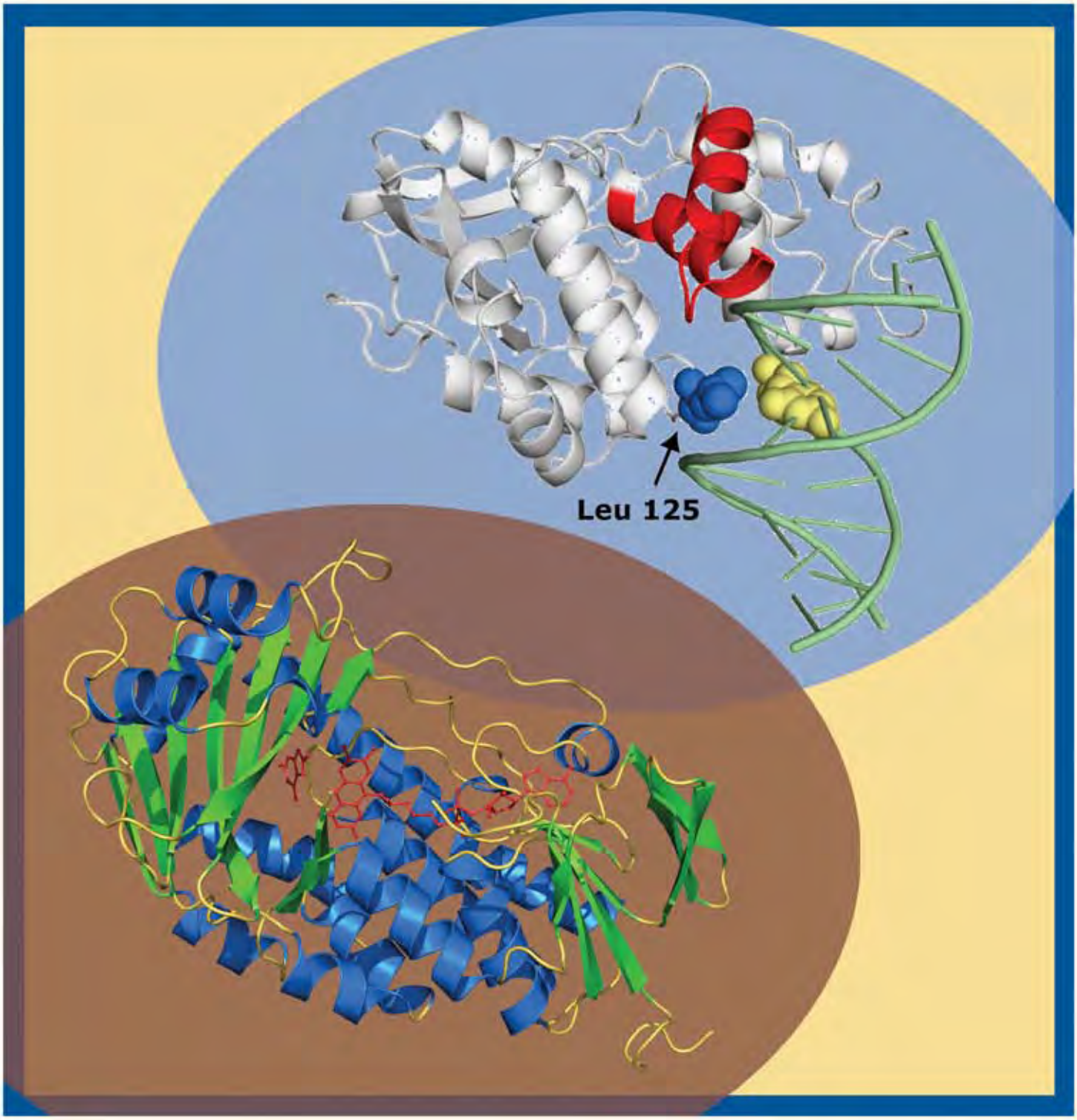
ACA RefleXions

American Crystallographic Association

Number 3
Fall, 2011



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Fall, 2011 *ACA HOME PAGE:* **www.AmerCrystalAssn.org**

Cover: Mechanistic Structural Enzymology in New Orleans see On the Cover, page 9.

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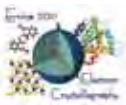

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Deadlines for contributions are: February 1st (Spring), May 1st (Summer), August 1st (Fall) and November 1st (Winter)

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As this issue of *RefleXions* goes to press, the IUCr has recently met in Madrid where plans are continued to progress for the **2013 International Year of Crystallography** (IYC), which will mark the centennial of the birth of modern crystallography. ACA Vice President George Phillips and CEO Bill Duax were among the attendees representing the ACA in Madrid.

In late May and early June it was a great pleasure for me to have the opportunity to greet many of you personally at our annual ACA meeting in New Orleans. The meeting was a great success, and 640 attendees enjoyed the outstanding scientific program assembled under the direction of Program Chair Chris Cahill. Local co-chairs Cheryl Klein Stevens and Ed Stevens made sure that everything ran smoothly. Many of us took advantage of Cheryl and Ed's list of recommendations to enjoy the fabulous New Orleans cuisine with friends and colleagues.

Planning is now underway for next summer's ACA meeting, which will be held in Boston July 28 - August 1, 2012. Don't forget that Boston will inaugurate our new, four-day format with scientific sessions running from Sunday through Wednesday. Our Boston Co-Program Chairs Bruce Foxman and Bruce Noll are putting together an outstanding program.

The ACA Council announced a number of important actions in New Orleans. These included the selection of the inaugural class of ACA Fellows. For citations and photos of these 16 distinguished crystallographers, see pages 6-7. Council accepted the Finance Committee recommendations to establish a new History Fund to maintain an electronic history of the ACA and its members over the years and to create a new Development Officer position for fundraising. Additional Council Highlights may be found on page 5.

The ACA is always striving to improve communications with you, our members. This year a team headed by Jamaine Davis, our representative on Council from the YSSIG (Young Scientists Scientific Interest Group) has created a LinkedIn website. If you'd like to try out LinkedIn, just follow the link on the ACA home page. ***Editor's note: plus -- the ACA is now on FACEBOOK!***

Finally, I'm pleased to announce that Kristina Vitale has joined ACA's staff. Kristina will be assisting Marcia Colquhoun and Crystal Towns in the headquarters office. We are greatly indebted to Marcia and Crystal, and now Kristina, for their dedicated work on all of our behalf. Please join me in welcoming Kristina to the ACA team.

Tom Koetzle

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RefleXions is pleased to announce that **Frank Fronczek,** Chemistry Dept, LSU, **ffroncz@lsu.edu** is our new **Puzzle Corner Editor**, replacing **Sidney Abrahams**, who decided to retire in order to devote more time to his continuing research projects at Southern Oregon U.

Historian: Virginia Pett

pett@wooster.edu

Art in Crystallography Editor: Edgar Meyer

model_em@yahoo.com
http://molecular-sculpture.com

The *RefleXions* editors are hoping someone will volunteer for the position of ***Opinions Column Editor.***

In the past our *Opinions* columns have featured two subjects: **Intelligent Design / the Evolution debates** and **Global Warming**. We can supply sources for both of these, and the column could consist of updates from these sources - or - the *Opinions* editor could choose another subject and put together something different. Please contact Connie Rajnak (**conniechidester@earthlink.net** or Judy Flippen-Anderson (**acareflexions@gmail.com**).

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L to r: Marvin Hackert, Marcia Colquhoun, Bill Duax, S.N. Rao, Tom Koetzle, Carrie Wilmot, George Phillips, Judy Kelley, David Rose

Photo courtsey of Marvin Hackert.

2011 ACA Council Meeting, May 27th

Jamaine Davis now represents the Young Scientists Scientific Interest Group (YSSIG) - a new *ex officio* position on the ACA Council.

President Tom Koetzle announced the 2012 ACA awards. The M. J. Buerger Award winner is **John H.C. Spence** (Arizona State University), the Charles E. Supper Instrumentation Award winner is **Ron Hamlin** (Area Detector Systems Corp) and the Warren Diffraction Physics award winner is **Paul Fenter** (Argonne National Labs).

Council has selected the the University of Notre Dame and Northwestern University to host the ACA Summer Course in Small Molecule Crystallography for 2012-2015. The courses will be organized by Allen Oliver (Notre Dame) and Amy Sarjeant (Northwestern). Council has established a new ACA fund to ensure the long term availability of the summer school courses.

Bernie Santarsiero has resigned as ACA Treasurer and **CFO S. N. Rao** will take over his duties for the remainder of his elected term. Council accepted a Finance Committee recommendation to create a new **History Fund** to maintain an electronic history of the ACA and its members and to create a new **Development Officer** position for fund raising.

Secretary Carrie Wilmot said that she had recorded the report of **Executive Officer Bill Duax** and **Director of Administrative Services Marcia Colquhoun** that there were 1,477 paid-up ACA members in 2011 thus far.

IUCr Representative Marvin Hackert announced that the IUCr was making 2013 the ***International Year of Crystallography*** to mark the centennial of the birth of modern crystallography.

YSSIG Representative Jamaine Davis stated that there was great enthusiasm among YSSIG members to get involved if given the opportunity. Since the primary interest of YSSIG members is their careers, a ***LinkedIn*** web-site was created. Under the new 4 day annual meeting format, the Mentor-Mentee dinner will most likely be replaced by setting up one-on-one meetings between mentors and mentees.

Annual Meetings: The 2012 ACA Annual Meeting in Boston will be the first to adopt the new 4 day format. A meeting in Hawaii in 2013 is possible. Serious negotioations are underway.

ACA Fellows: The Fellows Program recognizes a high level of excellence in scientific research, teaching, and professional duties as well as service, leadership, and personal engagement in the ACA and the broader world of crystallography and science. The first group of 16 ACA Fellows are: **Helen Berman, Philip Coppens, Johann Deisenhofer, Bill Duax, Judy Flippen-Anderson, Jenny Glusker, Herb Hauptman, Wayne Hendrickson, Carroll Johnson, Isabella Karle, Jerome Karle, S.N. Rao, Connie Rajnak, Michael Rossmann, George Sheldrick, and B.C. Wang.** For citations and photos, see the next two pages.

ACA Summer Courses: no macromolecular crystallography summer course was proposed for 2011 (the course has not been held for 2 years). Discussions at the individual SIG meetings during the week ndicated that the need for such a course exists and potential sites and organizers were identified. The Continuing Education Committee and the ACA Buffalo office will work with potential organizers to put together a proposal so that a macromolecular crystallography summer course can be held in 2012.

Latin American Division: Council would welcome the formation of a Latin American Division within the ACA analogous to the Canadian Division. This division could then elect a voting member to the ACA Council.

Carrie Wilmot

Ask not for whom the photocopier jams. It jams for thee.

Cartoon courtesy of Nick D. Kim. See www.lab-initio.com/index.html

The Inaugural Class of ACA Fellows with brief selections from their accomplishments, awards, and ACA service follows.

Helen M. Berman, Board of Governors Professor of Chemistry at Rutgers University, for her pioneering work in nucleic acid crystallography and for her leadership of the RCSB Protein Data Bank. Helen received the ACA Buerger Award in 2006. She served as ACA President in 1988.

Philip Coppens, SUNY Distinguished Professor, University at Buffalo, for his far-ranging accomplishments including pioneering x-ray charge-density analyses, and photocrystallography studies in which he and his group have spearheaded the use of time-resolved techniques for studies of molecular excited states. Philip received the IUCr Ewald Prize in 2005 and the ACA Buerger Award in 1996. He served as ACA President in 1978 and as IUCr President from 1993 - 1996.

Johann Deisenhofer, Virginia and Edward Linthicum Distinguished Chair in Biomolecular Science, UT Southwestern Medical Center. Hans is being honored for his groundbreaking advances in studies of light-driven biological processes, including the hallmark structure determination of the photosynthetic reaction center, for which he shared the Nobel Prize in Chemistry in 1988 with Robert Huber and Hartmut Michel.

William L. Duax, H. A. Hauptman Distinguished Scientist, Hauptman-Woodward Medical Research Institute, co-editor of the *IUCr Newsletter* and ACA CEO, for his work in biological crystallography including studies of ionopores and of steroid structures, and for his many years of service to the ACA. Bill served as ACA President in 1986 and as IUCr President from 2002–2005.

Judith L. Flippen-Anderson, RCSB Protein Data Bank, and co-editor of *ACA RefleXions* and the *IUCr Newsletter*, for contributions in small-molecule crystallography during her career at the Laboratory for the Structure of Matter, U. S. Naval Research Laboratory, and for her many years of service to the ACA. Judy has co-edited the *ACA Newsletter/RefleXions* since 1993. She served as ACA President in 1991.

Jenny P. Glusker, Professor Emeritus, The Institute for Cancer Research, Fox Chase Cancer Center, for her distinguished career as an inspiring teacher, mentor and author, and as a research scientist. Jenny's research accomplishments include studies of carcinogen-nucleic acid interactions, studies of metal ions in proteins, and her groundbreaking structure determination of vitamin B12. Jenny received the ACA Fankuchen Award in 1995, which she shared with Kenneth Trueblood. She founded and edited the *ACA Newsletter* for many years (until 1990). Jenny served as ACA President in 1979.

Herbert A. Hauptman, President of Hauptman-Woodward Medical Research Institute (retired), for his pioneering development of direct methods of phasing in crystal structure determination for which he and Jerome Karle shared the Nobel Prize in Chemistry in 1985. They also shared the ACA Patterson Award in 1984.

Wayne A. Hendrickson, University Professor, Columbia, for his far-reaching accomplishments in biological crystallography, including the development of macromolecular structure refinement protocols, and of MAD phasing methods that have revolutionized structure determinations with synchrotron radiation. Wayne was the first recipient of the ACA Patterson Award in 1981.

Carroll K. Johnson, Oak Ridge National Laboratory (retired), for his many accomplishments in mathematical and physical crystallography including studies of thermal motion, disorder, and topologies, and for the development of computer graphics techniques including the ORTEP package. Carroll served as ACA President in 1977.

Isabella L. Karle, X-ray Crystallographer, Laboratory for the Structure of Matter, U. S. Naval Research Laboratory (retired), for her distinguished research on biologically interesting molecules, including natural products and peptides, and for her seminal work with direct methods of structure solution. Isabella served as ACA President in 1976.

Jerome Karle, Chief Scientist, Laboratory for the Structure of Matter, U. S. Naval Research Laboratory (retired), for his pioneering development of direct methods of phasing in crystal structure determination for which he and Herbert Hauptman shared the Nobel Prize in Chemistry in 1985. They also shared the ACA Patterson Award in 1984. Jerry served as ACA President in 1972 and as IUCr President from 1981-1984.

Connie Rajnak, Co-Editor, *ACA RefleXions*, for her research contributions in structure-activity relationships and drug-receptor modeling as a crystallographer at Upjohn and Pharmacia, and for her many years of service to the ACA. She edited the *ACA Newsletter* from 1991-1992 and has co-edited the *ACA Newsletter/RefleXions* since 2002. Connie served as ACA President in 2000.

S.N. Rao, Graduate College Dean Emeritus, University of Central Oklahoma, and ACA CFO, for his distinguished career in university administration and teaching and in biological structure research, and for his many years of faithful service to the ACA where for more than 25 years, through good times and bad, Rao has worked to safeguard the ACA's finances.

Michael G. Rossmann, Hanley Professor of Biological Sciences, Purdue University, for his many outstanding contributions to macromolecular crystallography culminating in his legendary pioneering work on the structure of viruses. Michael received the ACA Fankuchen Award in 1986 and the IUCr Ewald Prize in 1996.

Editor's note: Peter Müller took most of these photos.

George M. Sheldrick, Professor of Structural Chemistry, Georg-August-Universität Göttingen, for his distinguished career in teaching and education, and in research where he has made fundamental advances in methodologies of structure solution and refinement for small and large molecules, as well as advances in instrumentation and in crystallographic software including the SHELXTL package. In Madrid this year George received the 2011 IUCr Ewald Prize, which he shared with Eleanor Dodson and Carmelo Giacovazzo. He received the ACA Patterson Award in 1993.

Bi-Cheng Wang, Professor and Ramsey-Georgia Research Alliance Eminent Scholar in Structural Biology, University of Georgia, for his distinguished research in macromolecular crystallography focusing on structure-function studies, and for his groundbreaking work on phasing methodologies and on advancing applications of synchrotron radiation. B.C. received the ACA Patterson Award in 2008.

Two New ACA Funds

ACA History Fund: We hope you have been enjoying the Living History articles that have been compiled by Virginia Pett, our ACA Historian, and published in *RefleXions*. In many cases the full autobiographical article was shortened for publication, but the full document with references has been archived in the AIP History Center Niels Bohr Library & Archives (NBL&A). Unfortunately, the documents are not available online from NBL&A. There are links but a request must be sent to the Center to obtain a copy of the full article and the associated information such as references, curriculum vitae, etc. The advantage of this process is that the articles have been safely archived and are available to scholars while we continue toward our ultimate goal of making everything available online through the ACA History Portal. In addition to the Living Histories, the Portal will provide access to other historical documents such as obituaries, ACA award lectures, selected focus articles and other items of historical interest.

To do this properly requires designing a new page for the ACA website to serve as the History Portal as well as developing the software needed to make the site fully searchable. It is no surprise that this will cost money so a new ACA fund, the ACA History Fund, has been created to cover the cost of supporting the initial creation of the site and it's continued maintenance (including the cost of storing the files). Thanks to the hard work of our generous ACA volunteers *RefleXions* has been slightly in the black and Council has accepted our suggestion to use some of this 'profit' as seed money for the History Fund.

Judy Flippen-Anderson

ACA Summer School Fund: Courses teaching the fundamentals of x-ray crystallography have disappeared at an accelerating rate, but young crystallographers need this information more than ever. To help fill this void the ACA has been offering, and underwriting, summer courses in both small-molecule and macromolecular crystallography. A new ACA fund has been established to help ensure that we will be able to continue and expand these courses for the long term. Both of these new funds will appear along with the other established ACA award funds on your dues renewal form. Please give strong consideration to supporting both our future (through the funds supporting young crystallographers) and our scientific legacy through the History Fund. You will also be able to donate to all ACA funds anytime the mood strikes by using the new '***Donate Now***' button on the ACA website (**www.amercrystalassn.org**)

Judy Flippen-Anderson

A panorama of the Exhibits Show at the ACA Annual Meeting in New Orleans. The NO Meeting section starts on page 19.

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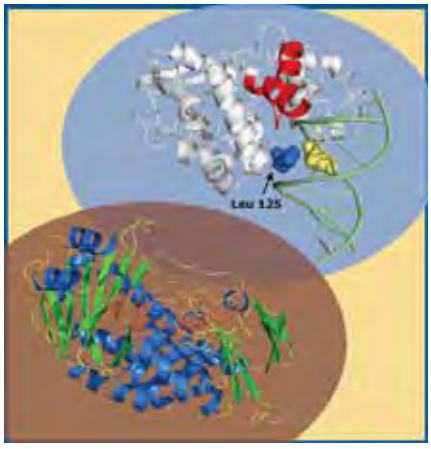
Images from Structural Enzymology II - Mechanistic at the ACA meeting in New Orleans



Both images were from talks presented in Session 1.03, see pages 27-28.

Upper right: from **Brian Bowman**, Chem. & Chem. Biology Dept., Harvard. Brian and co-authors reported the crystal structures of the DNA glycosylase AlkA in complex with undamaged DNA. (*Structure of Escherichia coli AlkA in complex with undamaged DNA* by Brian R. Bowman, Seongmin Lee, Shuyu Wang, & Gregory L. Verdine, *J Biol Chem*., 2010, **285** (46): 35783-91). The structures revealed a recognition mode in which the DNA is nearly straight, with no amino acid side chains inserted into the duplex, and the target base pair is fully intrahelical. A comparison of these structures with that of AlkA recognizing an extrahelical lesion revealed conformational changes in both the DNA and protein as the glycosylase transitions from the interrogation of undamaged DNA to catalysis of nucleobase excision. Modeling studies indicated that the DNA repair glycosylase AlkA uses its probe residue Leu125 to actively interrogate the minor groove of the DNA while searching for the presence of damaged nucleobases.

Lower left: from **Katherine Hicks**, Chem & Chem. Biol. Dept., Cornell. Urate oxidases have been well-characterized in the literature.These enzymes catalyze reactions using radical mechanisms, which do not require cofactors. In contrast, the urate oxidase HpxO from the ubiquitous pathogen *Klebsiella pneumonia* hydroxylates urate using FAD and NAD(P)H as co-factors and is a member of the Class A flavin-monooxygenase family. The image of the HpxO monomer is depicted in cartoon representation with the FAD cofactor and urate substrate as sticks in red. Katherine Hicks is a postdoctoral associate in Steven Ealick's lab at Cornell. Katherine presented the HpxO structure and also biochemical work in collaboration with Tadhg Begley (Texas A&M) in New Orleans. Taken together, the structural and biochemical characterization of HpxO support the existence of a novel mechanistic paradigm in purine catabolism. The biochemical analysis has been published (*Biochemistry*, 2009, **48** (14) 3033-3055); the structural work will be published soon.

New ACS Fellows, Jim Ibers & Stephen Lippard

The ACS honored 213 members from academe, industry, and government by induction into their 2011 class of ACS Fellows. ACA members **James A. Ibers**, Charles E. and Emma H. Morrison Professor of Chemistry at Northwestern University, at left, and, on the right, **Stephen J. Lippard**, Arthur Amos Noyes Professor of Chemistry at MIT are in this distinquished group.

Jim's research group studies transition metals and rare-earth compounds. Specifically, the Ibers laboratory focuses on metal-chalcogenide structures, which differ significantly from the more commonly studied metal-oxides. The Lippard laboratory primarily studies the synthesis, reactions, and physical and structural properties of metal complexes as they attempt to model the active sites of metalloproteins in order to design anti-cancer drugs. ACS Fellows are recognized for their outstanding achievements in and contributions to science, the profession, and ACS. Ibers and Lippard were honored, along with the other Fellows, at the ACS 2011 Fall National Meeting and Exposition in Denver, CO, in August, 2011.

MSA Research Grant to Sochalski-Kolbus

The 2011 Mineralogical Society of America's **Grant for Research in Crystallography** was awarded to ACA member **Lindsay Sochalski-Kolbus** of Virginia Polytechnic Institute and State University for her research ***High-temperature structural study of Li-feldspar.*** Funded in part by the Edward H. Kraus Crystallographic Research Fund and contributions from MSA members and friends, the grant recognizes students and young researchers in the areas of mineralogy, crystal chemistry, petrology, mineral physics, biomineralization, and geochemistry whose research includes crystal structure as an explicit and integral element.

L to r: Paul Stanley, Ante Qu, Lucy Chen, Brian Zhang, Andrew Das Sarma, Eric Speiglan, Warren Turner.

US Physics Team Brings Home 5 Medals

Almost 400 high school level physicists from 84 different countries traveled to Thailand in July to participate in the 42nd International Physics Olympiad. The US team scored fifth overall, placing behind Taiwan, China, Singapore and Korea, and tied Romania for eleventh place on the medal count. Of the 20 US Team members, 5 were selected to travel to Thailand and all 5 won medals. Two seniors, **Brian Zhang**, from Henry M. Gunn High School in Palo Alto, CA and **Ante Qu**, from West Windsor-Plainsboro High School South in Princeton Junction, NJ, came home with gold medals. The silver medalists were **Lucy Chen**, a senior at Ames High School in Ames, IA, **Andrew Das Sarma**, a senior at Montgomery Blair High School in Silver Springs, MD, and **Eric Speiglan**, a junior from Naperville North High School in Naperville, IL. The team was coached by Paul Stanley, Warren Turner, Andrew Lin, Jia Jia Dong, Quizi LI, David Fallest, and Marianna Mao. Started in 1996, the US Physics Olympiad Program is supported by the American Association of Physics Teachers, American Institute for Physics, and its member societies, including the ACA.

The 69th Annual Pittsburgh Diffraction Conference

Mark your calendars: The 69th Annual Pittsburgh Diffraction Conference and the 5th Annual Ohio Valley Crystallography Symposium will hold a joint meeting on November 2-4, 2011. The meeting will be held at Case Western Reserve University in Cleveland, Ohio and will include sessions in macromolecules, neutron diffraction, detectors, and small molecules. Two additional sessions will be dedicated to the research presentations of junior investigators. **Registration and abstract deadline is September 30, 2011.** Please visit **www.pdsconference.org** for more information.

CCP4 wins 2011 Rita and John Cornforth Award

The Royal Society of Chemistry has named the Collaborative Computational Project 4 (CCP4) winners of the **2011 Rita and John Cornforth Award.** This award is given to reward scientists working in collaborative research teams in either chemistry or the life sciences. CCP4 was rewarded for providing resources that benefit macromolecular structural chemistry worldwide and for exemplar team-ethos over many years. Established in1979, the CCP4 provides and supports an integrated suite of programs for macromolecular x-ray crystallography which is freely available to academics, not-for-profit organizations, and commercial researchers. CCP4 combines voluntary contributions of software from the community with that developed by their core team of dedicated developers. It also hosts a bulletin board that is probably the most complete repository of macromolecular crystallographic theories and practices in the world. The **Rita and John Cornforth Award** is open to everyone, but all applicants must be nominated as teams, not individuals. **Nominations for the 2012 award opened on Sept. 1, and close on Jan. 15, 2012.**

Use CellCheckCSD before you collect data

The Cambridge Crystallographic Data Centre (CCDC) and Agilent Technologies Inc. have developed a new service in their crystallography data collection and processing package CrysAlisPro. Crystallographers can now use CellCheckCSD to check their pre-experiment unit cell against the Cambridge Structural Database (CSD) before collecting a full diffraction dataset. The CSD is the world's largest repository of small molecule crystal structures, and searching that database will save valuable diffractometer time by identifying situations where starting materials or a reaction by-product have been crystallized by accident. This added functionality is downloadable free of charge from the CCDC website.

CIF deposition to the CSD now easier

The CCDC, which compiles and distributes the CSD, together with OlexSys Ltd have announced a new function of Olex2 that allows users to directly upload CIFs for direct deposition to the CSD. This addition to the popular small-molecule structure solution and refinement program simplifies structure deposition and facilitates sharing of unpublished data and work. The new CIF upload mechanism is incorporated in the latest release of Olex2, which can be downloaded frm the OlexSys website.

CCDC now accepts structure factors

The Cambridge Crystallographic Data Centre (CCDC) is pleased to announce that structure factors are now being accepted with CIFs for deposition to the CSD. CIFs and structure factors can now be deposited with the CCDC via our web-based deposition form at:

www.ccdc.cam.ac.uk/services/structure_deposit

Boris Batterman (1930 - 2010) Boris (Bob) Batterman, Cornell's Walter S. Carpenter Jr. Professor of Applied and Engineering Physics Emeritus, died Dec. 14 at his home in San Francisco. He was 80 years old.

Batterman joined the Cornell faulty in 1965 as a professor in both the Department of Materials Science and Engineering and the School of Applied and Engineering Physics. He served as chair of the School of Applied and Engineering Physics from 1974 to 1978, after which he became director of the newly established Cornell High Energy Synchrotron Source, CHESS, a national laboratory for synchrotron radiation research.

In 1983 Batterman received the Humboldt Award from the Federal Republic of Germany, and in 1985 he was named the Walter S. Carpenter Jr. Professor of Applied and Engineering Physics at Cornell. In 2001 Batterman retired from Cornell and spent much of his time in San Francisco, where he continued to be active in science with positions both at Stanford and at UC-Berkeley.

Born Aug. 25, 1930, in Brooklyn, N.Y., Batterman attended Brooklyn Tech and Cooper Union in New York before earning his undergraduate degree in 1952 and his PhD in physics in 1956, both at MIT.

Batterman's interests were varied; he climbed the Matterhorn in the Swiss Alps in 1960, an accomplishment of which he was most proud. He raced sailboats, collected and worked on old automobiles, and was an amateur astronomer. He is survived by his longtime partner, children, grandchildren, nieces and nephews.

From the Cornell University's ChronicleOnline, Jan 10, 2011

From the ChronicleOnline, Vol. 29, Number 3, Sept. 4, 1997. Boris Batterman, left, who retired recently as director of the Cornell High Energy Synchrotron Source (CHESS) after 19 years in that post, is congratulated by Gopal Shenoy of the Argonne National Lab, at an appreciation reception in Batterman's honor outside of Wilson Lab on Aug. 26. Shenoy was one of several noted participants at a symposium in Clark Hall earlier in the day, also in recognition of Batterman, the Walter S. Carpenter Jr. Professor of Engineering and professor of applied and engineering physics.

Photo by Robert Barker/University Photography.

Editor's note: a more extensive appreciation of Hiro will appear in the winter issue of RefleXions.

It is with great sadness that we announce that Dr. Hirotsugu (Hiro) Tsuruta, a senior scientist and biophysicist in the Structural Molecular Biology program at Stanford Synchrotron Radiation Lightsource (SSRL) at SLAC, passed away on August 25, 2011, in Japan, after a courageous fight with cancer. Funeral services took place in Sasebo, Japan, on August 27. Hiro worked at SSRL for more than 20 years, and with great dedication and scientific insight provided leadership for the build-up of the SSRL biological small angle x-ray scattering research program, and a bioSAXS beam line facility that today serves a large user community. He also served the international community in many ways. His contributions were numerous, and he leaves an international legacy in this growing area of science, for which he pursued new developments until the end.

Britt Hedman and Keith Hodgson

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For further information on the DTC visit www.oxcryo.com

Our new Puzzle Corner Editor, Frank Fronczek *ffroncz@lsu.edu*

Movie puzzler: We asked which Jimmy Stewart movie mentions crystallography, who his co-star was, and under what circumstances crystallography was mentioned. In the 1951 movie ***No Highway in the Sky***, Stewart plays aeronautical engineer, **Mr. Honey**, opposite Marlene Dietrich, **Miss Teasdale**. In one scene, Honey says of his daughter Elspeth, ***She's only twelve, but she's got a very good grasp of crystallography.*** He also bemoans the fact that schools don't teach more about crystallography, so she must have learned it from him.

Answers to summer issue puzzles (letters from Doug Dorset & Carol Huber):

Looking at the latest ACA Reflexions, I am certainly cognizant of the 'mystery people' in the top photo (bottom right) of page 11. (This publication is still produced in Buffalo, right?) I will identify them:

On the right is Walter Pangborn, the current vice president of HWI - he's still in Buffalo, I presume? On the left is Chris Gilmore, member of the Executive Committee of the IUCr, retired chair of the chemistry department, University of Glasgow. He was in Buffalo at MFB on sabbatical in 1980. In the center is yours truly who had just received the Patterson Award from the ACA. (I'm a bit thinner now but still alive.) As I recall I was once a department head at HWI but I spent so much time in dark rooms that I might be mistaken... So much for 'immortality' from prizes and work! (Actually I find it hilarious. It's great to be retired.)

Greetings from the ash-heap of history, *Doug Dorset*

Thanks for the article on women in the ACA/IUCr. I seem to be in the right age group to get all the name/picture connections on p. 5 this time - from the top:

Connie Elinor	Sine Judy K
Jenny Isabella	Penny Helen
Judy F-A , Fran , me	Elizabeth Dorothy

It was very nice that Eleanor Dodson got (a share of) the Ewald Prize. I've known her since the early 60s in Dorothy's lab. *Carol Huber*

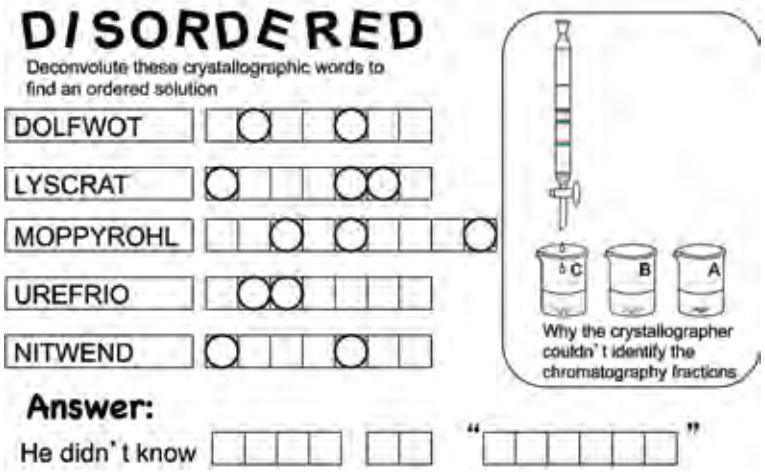
Obviously, the author of the posters at the bottom of page 11 (summer Puzzle Corner) was Wally Cordes. - Connie & Judy

New Puzzles - Fall, 2011:

What was "The Grand Ah-whoom"? How does it relate to disappearing polymorphs?

And also: - - - - ->

Errata: the editors regret the error in the summer 2011 issue of *RefleXions* (which **Jim Ibers** was quick to point out) --in the p 3 article on **Local Structure and New Energy Materials**: *traditional crystallography works pretty well down to 10 nm* should be ***(100 Å)*** not ***1 Å,*** as was printed.



Contributors to This Issue

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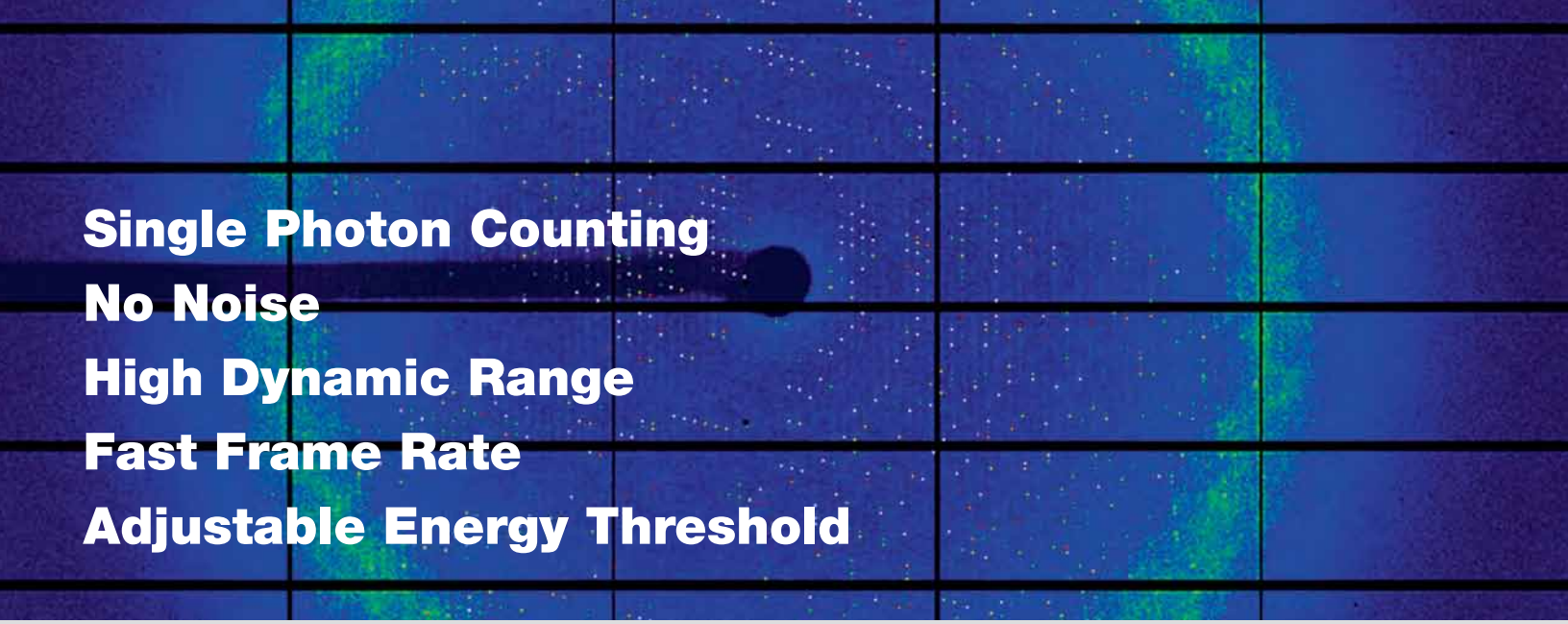

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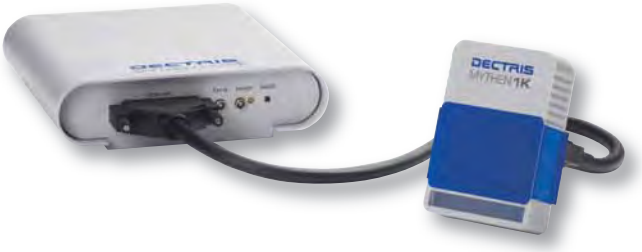

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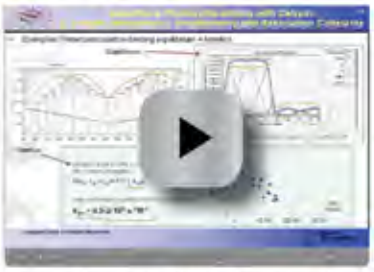

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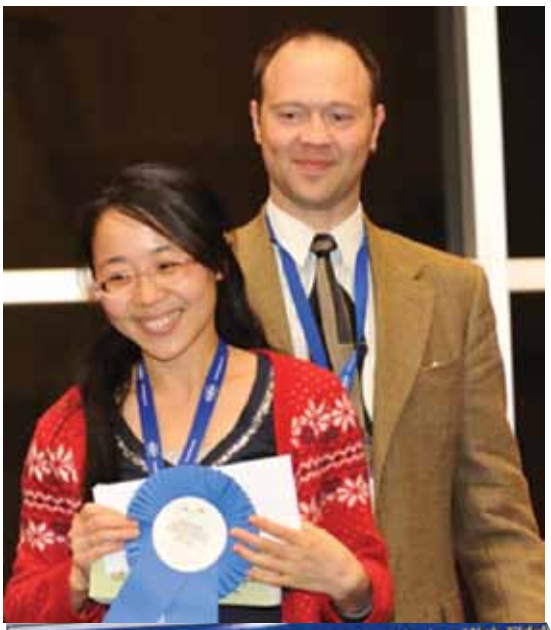
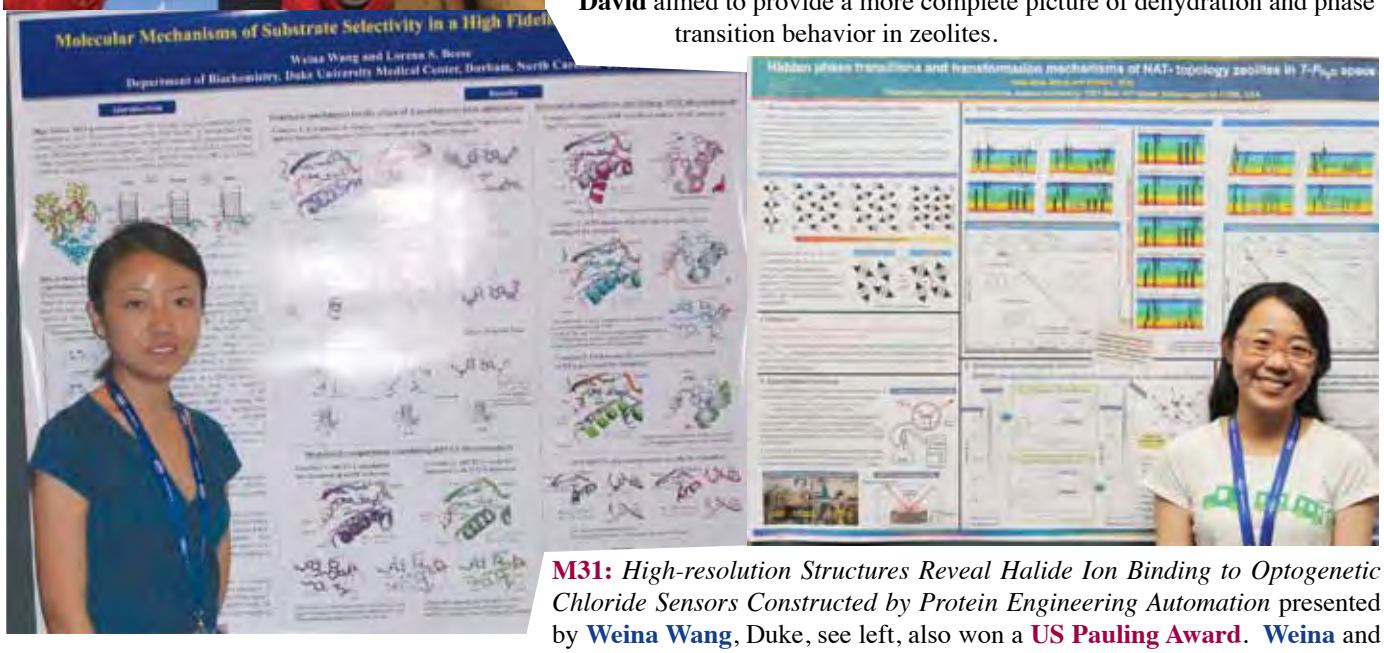


2011 Pauling Prizes

At left, **Hsiu-Wen Wang**, Indiana U, who won a **2011 Pauling Prize** for **M13**, *Hidden Phase Transitions and Transformation Mechanisms of Nat-Topology Zeolites in T-P(H_2O) Space*. Standing behind **Hsiu-Wen** is **Ilia Guzei**, the **2011 Poster Chair**.

The **Pauling Prize Selection Committee** this year did triple duty because they selected not only the **US** and **Canadian Pauling Award** winners, but also the **IUCr Poster Prize** winner (see the page-after-next). **James Kaduk, Joseph Ng, Heather Pinkett** and **Susan Buchanan** "volunteered" for this privilege.

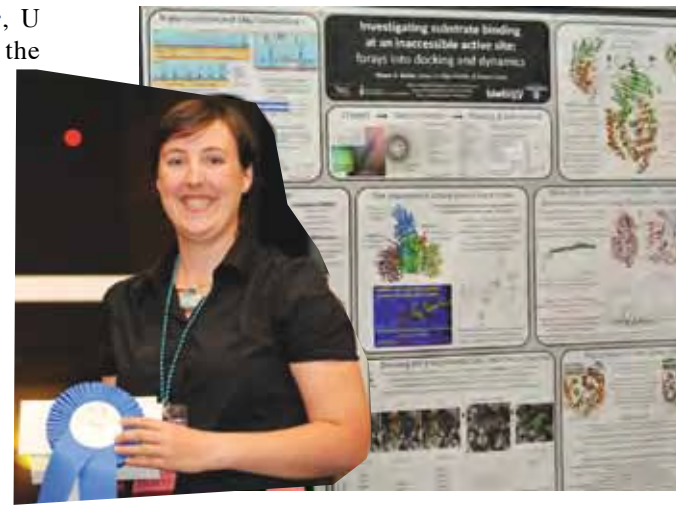
Hsiu-Wen and her co-author **David Bish** found that in natrolite, the anhydrous α1-metanatrolite phase- $Na_{16}Al_{16}Si_{24}O_{80}$, F112 occurred at elevated P(H_2O), whereas a previously hidden anhydrous phase, α2-metanatrolite-$Na_{16}Al_{16}Si_{24}O_{80}$, Fdd2, was found at low P(H_2O). In other words, a low-P(H_2O) atmosphere allowed access to a phase space that was not accessible at higher P(H_2O), where dehydration of hydrous natrolite was suppressed. Bearing in mind that during zeolite dehydration, the environmental parameters temperature and P(H_2O) are coupled and do not operate independently of one another, and that most previous studies ignored this effect, **Hsiu-Wen** and **David** aimed to provide a more complete picture of dehydration and phase transition behavior in zeolites.



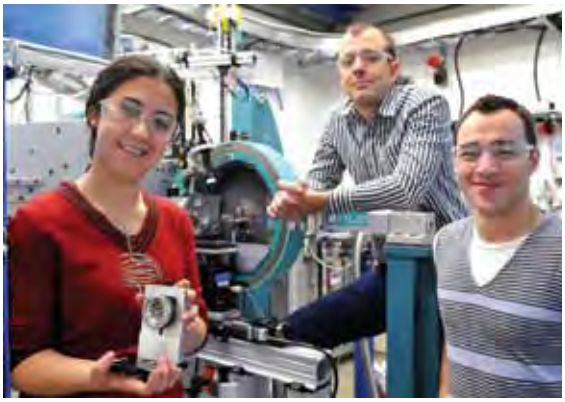
M31: *High-resolution Structures Reveal Halide Ion Binding to Optogenetic Chloride Sensors Constructed by Protein Engineering Automation* presented by **Weina Wang**, Duke, see left, also won a **US Pauling Award**. **Weina** and her co-authors **Joshua Grimley, Lorena Beese** and **Homme Hellinga**, also at Duke, set out to construct an affinity-matched OS (optogenetic sensor). In order to avoid cloning the candidates, they used a cell-free protein engineering method. They constructed, produced and assayed several hundred YFP variants of the 7 residues lining a previously identified halide-binding site. Using x-ray crystallography they studied YFP and the two variants that seemed the most promising. They hope to improve understanding of halide recognition and thereby assist future OS engineering efforts. **Brandon Goblirsch** (right) also won a **US Pauling Award** for **M09:** *Structural Features of the Unusual Thiolase OleA that Facilitates Long-Chain Hydrocarbon Biosynthesis*. **Brandon** and his co-authors **Janice Frias, Larry Wackett**, and **Carrie Wilmot** at U MN, are interested in biosynthetic pathways that generate long-chain/high-energy hydrocarbons as potential biofuels. OleA, a dedicated operon and a member of the thiolase superfamily, acts in the first stage of olefin production, and with this in mind they determined the crystal structures of OleA in its unbound and inhibited forms. Comparing the two structures suggests that rearrangements of active site residues are essential.

Canadian Pauling Prize Winner

Megan Barker, U Toronto, won the **Canadian Pauling** for: *Structure and Mechanism of Processing Alpha-Glucosidase I*. **Megan** and co-author **David Rose**, U Waterloo, solved the x-ray structure, to 2.04Å resolution, of yeast α-glucosidase I, (Cwht1p), and plan to study it in order to characterize it's mechanism of action; the processing of α-glucosidase I is the first step in N-glycoprotein processing.



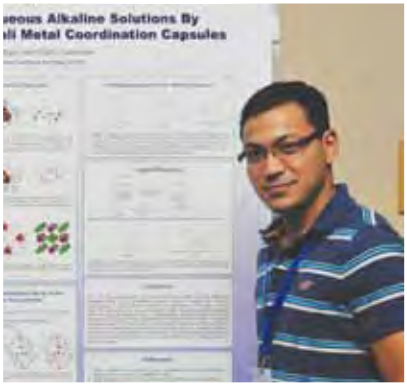
Karena Chapman, Argonne National Laboratory, won the **2011 Oxford Cryosystems Poster Prize** for **M45:** *11-1D-B, a Dedicated Instrument for X-ray Pair Distribution Function Measurements*. **Oxford Cryosystems** gives this cash prize to the best poster describing work in low temperature crystallography. Karena's poster reported her work with **Kevin Beyer** and **Peter Chupas** to optimize the instrument at APS. They improved data quality, reduced data collection times, and facilitated a whole range of sample environments for *in-situ* studies under non-ambient conditions. Now studies varying temperature, pressure and chemical environment are routine, and users collect hundreds or even thousands of individual PDFs per day. The **Oxford Cryosystems Selection Committee** members were: **James Fettinger, Dale Swenson,** and **Shao-Liang Zheng.**

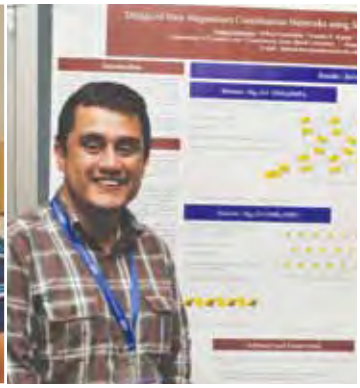


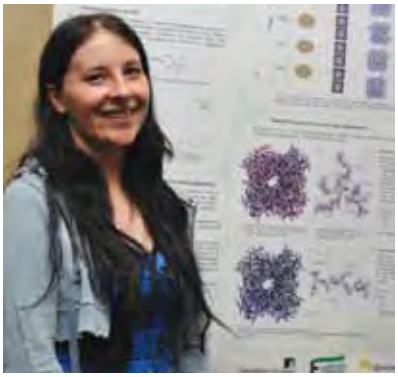
At left, Karena Chapman holds a diamond anvil cell next to collaborating scientists Peter Chupas and Gregory Halder. They were able to change the structure of a metal organic framework at pressures low enough for large scale industrial applications.

The **CrystEngComm Poster Prizes** went to **Debasis Banerjee**, Stony Brook U, for **S68:** *Design of New Magnesium Networks Using Solvents as Structure Directing Agents* and to **Arbin Rajbanshi**, ORNL, for **S18:** *Alkali metal-coordination cages for selective sulfate binding and separation*.

Debasis, below far right, and co-authors **Jeffrey Finkelstein** and **John Parise**, were able to demonstrate that varying the solvents of crystallization, despite subtle differences in those solvents, influenced the structural motifs in four magnesium based coordination networks using 4,4'-sulfonyldibenzoic acid as the organic linker. The utility of these materials, specifically as gas-adsorption agents, was also discussed.







Arbin and his colleagues **Bruce Moyer** and **Radu Custelcean** decided to use crystalline capsules to design hosts with sufficient rigidity to insure size and shape discrimination of the anionic guests. These capsules combine functionalized internal cavities with the rigid environment of a crystal lattice. Currently their experiments are aimed at exploring the potential utility of crystalline capsules in the treatment of nuclear waste. **Chris Incarvito, Claudia Rawn**, and **Vic Young** selected the winning poster. The **Royal Society of Chemistry**'s *CrystEngComm* is a peer-reviewed online-only journal that publishes original research and review articles

Chris Incarvito

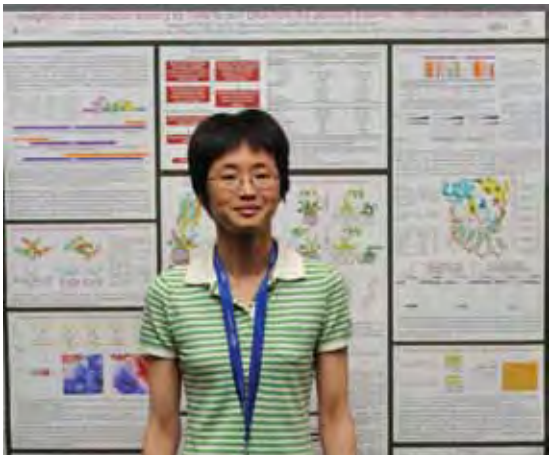
RCSB Poster Prize:

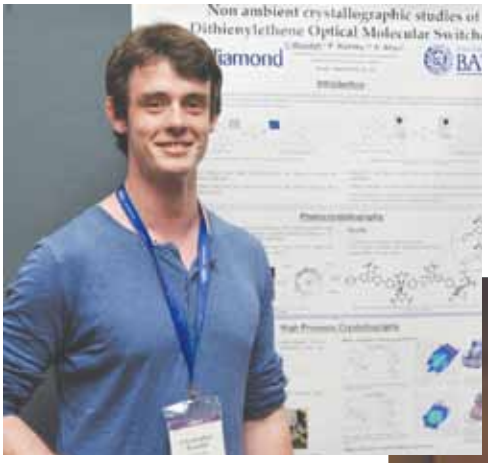
Briony Yorke, a graduate student at U Leeds, was awarded the **RCSB Protein Data Bank Poster Prize** for **M12**: *New Approaches to Time-Resolved Structural Studies of Macromolecules*. Together with mentors **Arwen Pearson, Michael Webb** and **Robin Owen**, **Briony** is developing a time-resolved diffraction technique to probe irreversible reactions at atomic resolution in real time. Taking advantage of a high-throughput synchrotron, hundreds of micro-crystals are used to collect time-resolved data, thus avoiding the limitations normally imposed by radiation damage to a single crystal. Results presented on aspartate decarboxylase showed the successful light-dependent initiation of the reaction in microcrystals, data collection and structure refinement of a covalent intermediate in the pathway. **Katrina Forest, John Rose**, and **Thomas Edward** selected Briony's poster.

Katrina Forest

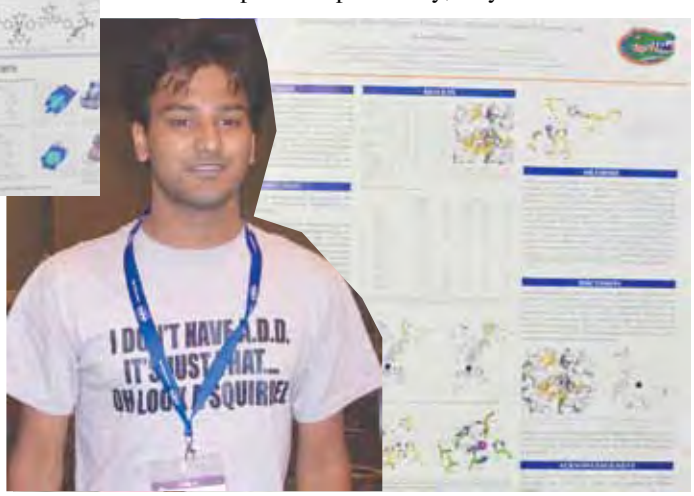
2011 IUCr Poster Prize

The **Selection Committee James Kaduk, Joseph Ng, Heather Pinkett** and **Susan Buchanan** who also selected the **Pauling Prize** winners, chose **Joyce Wong**, U Alberta, for **M27:** *Insights into Cooperative Binding of TraM to oriT DNA from the Crystal Structure of the pED208 Plasmid TraM-sbmA Complex*. Joyce and her co-authors **Jun Lu, Ross Edwards, Laura Frost** and **Mark Glover** reported the crystal structure of TraM of the F-like plasmid pED208 bound to its highest affinity binding site, sbmA, as well as the crystal structure of the apo-N-terminal domain. The structure shows that two TraM tetramers bind to sbmA in a cooperative fashion, remarkably without any protein-protein contacts, and propose that this enables TraM, while bound to DNA, to form many contact points with TraD during conjugation.



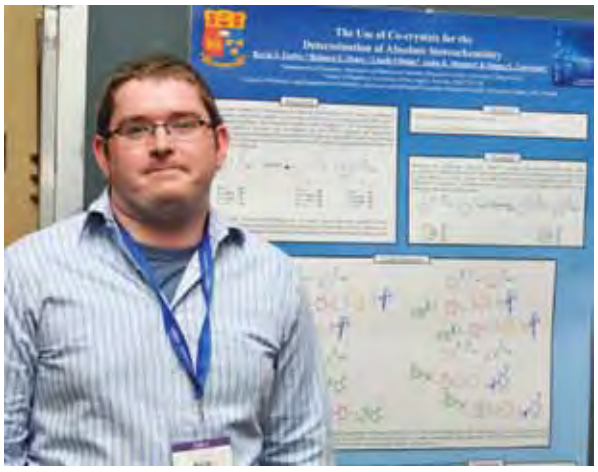


Christopher Woodall, U Bath, UK was awarded a ***Journal of Chemical Crystallography Honorable Mention*** for **S31:** *Non Ambient Crystallographic Studies of Dithienylethene Optical Molecular Switches*. **Chris** and co-authors **Paul Raithby**, and **David Allan** determined the structures of a series of new dithienylethene based systems and observed their solid state behavior. Dithienylene compounds are known to undergo electrocyclic ring closure and opening reactions when irradiated and are stable to thermal reversion and fatigue, making them ideal for applications involving molecular memories and switches. In **S31**, the authors reported several reversible single-crystal-to-single-crystal transformations that demonstrate unprecedented levels of conversion.



The ***Journal of Chemical Crystallography Poster Prize*** went to **Mayank Aggarwal**, below, U Florida, for **S14:** *Carbonic Anhydrase: Inhibitors and Activators*. **Mayank** and his co-authors **Fabbio Pacchiano, Balendu Avvaru, Arthur Robbins, Claudiu Supuran** and **Robert McKenna** studied carbonic anhydrases (CA), --metalloenzymes that catalyze the reversible reaction of CO_2 hydration to bicarbonate and a proton. Specifically, they studied the CA mutant H64A CA II, because it was known to be "rescued" by small molecules. They determined the structures of several chemical rescue compounds, which mimic the imidazole ring fuction of His64 in the proton shuttling mechnaism of the enzyme.

The ***JCC* Selection Committee** comprised **Khalil Abboud, Marilyn Olmstead**, and **Tatiana Timofeeva.**



The **American Chemical Society Poster Prize Selection Committee: Alicia Beatty, Nigam Rath, Felix Vajdos, Paul Wood,** and **Jim Britten**, chose **S25:** *The Use of Co-crystals for the Determination of Absolute Stereochemistry* by **Kevin Eccles,** University College, Cork, Ireland. **Kevin** and co-authors **Rebecca Deasy, László Fábián, Anita Maguire,** and **Simon Lawrence** noticed that Co-crystals, (structurally homogeneous crystalline materials that contain two or more neutral components), have the potential to modify the physical properties of the individual components without altering the chemical nature of the molecules. The combination of co-crystallization with x-ray diffraction and chiral HPLC proved effective when they set out to determine the absolute stereochemistry of a series of chiral 3-arylbutanoic acids. (These are oily and viscous and usually not well-behaved.) Co-crystallization offers advantages over salt formation because co-crystals dissociate in solution, so that identical HPLC conditions can be used for both the materials of interest and their co-crystals.

2011 ACA Meeting - New Orleans, LA, May 28th - June 2nd

The day long *Transactions* symposium on **Time Resolved Photochemistry and Electron Density** that was organized to honor **Philip Coppens** for his many contributions to crystallography and to celebrate his 80th birthday was a highlight of the meeting. Other highlights were the presentation of the **Patterson Award** to **Keith Moffat** and his award lecture; the **2010 Fankuchen Award** lecture presented by **David Watkin**, who was unable to come to Chicago last year; the presentation of the **Etter Early Career Award** to **Yurij Mozharizskyj**; the session on **Evolution of Powder Diffraction Software** in memory of **Lachlan Cranswick**; and the **Plenary Lecture** by **Nadrian Seeman** on ***DNA: Not Merely the Secret of Life***. Program Chair **Chris Cahill**, as well as the SIG Chairs did a marvelous job of organizing a stellar array of speakers and sessions. Local Chairs **Ed Stevens** and **Cheryl Klein-Stevens** had a relatively easier job because the French Quarter of the city of New Orleans was a delightful venue - great restaurants within easy walking distance!!

The winter issue of *RefleXions* will carry reports on the workshops and from the **Travel Award** winners as well as many more photos (the Mentor-Mentee Dinner; more photos by Peter Müller).

Ilia Guzei, Poster Chair for the meeting.

Below, ACA Past President Judy Kelly, with Bob Sweet.

Ron Hamlin, ADSC, at right, will receive the 2012 Charles E. Supper Instrumentation Award in Boston. John Spence, ASU, will receive the 2012 Buerger Award.

The Awards Banquet overview above is by our own staff photographer, Peter Müller, shown ON THE JOB at lower right. Almost all photos in this issue, including all the space-filling "art" photos were taken by Peter; exceptions are noted in the legends. An exception is the one above from Marvin Hackert: Marcia Colquhoun, Marvin, and Crystal Towns at the Awards Banquet.

Philip Coppens' Plenary Lecture: From the Sun to Femtosecond Lasers and Back: Photo-Induced Dynamic Processes in Solids

Philip Coppens, a Distinguished Professor of Chemistry at SUNY, Buffalo, is a former ACA President and a past president of the IUCr. Among his many awards, he received the 1994 ACA Buerger Award, the 1996 Gregori Aminoff Prize of the Royal Swedish Academy of Sciences, the 2005 Nishikawa Prize of the Crystallography Society of Japan, and the 2005 Ewald Prize of the IUCr. Philip is also a member of the inaugural (2011) class of ACA Fellows. He was introduced by Jason Benedict, a research professor in his laboratory.

The ability to follow the 3D structural dynamics of photochemical processes continues to revolutionize our understanding of light-induced structural changes in crystalline solids. To illustrate the accessibility of photocrystallography to chemical researchers, Philip presented results on the kinetics of single crystal to single crystal isomerizations that can be done "at home" with a conventional diffractometer and suitable light source. Resolving the structural changes which occur on very short time scales does require extremely bright synchrotron or X-ray Free Electron Laser (XFEL) sources. In his early experiments, monochromatic techniques were used to examine light induced structural changes on the microsecond time scale. His work has now transitioned into polychromatic Laue techniques which probe structural changes in as little as 70 picoseconds after excitation with much greater precision. The observed structural changes are generally not accurately modeled by theory. In the case of a binuclear rhodium complex, $Rh_2(PNP)_4BPh_4$, gas phase calculations predict an Rh-Rh contraction of 0.37 Å instead of the observed 0.14 Å. Taking the crystal environment into account using quantum mechanical and molecular mechanical (QM/MM) methods, the predicted contraction falls to 0.16 Å in agreement with the value from experiment.

Always looking to the future, Philip spoke of a few systems in which the structural changes are predicted to occur on a femtosecond time scale. These experiments will require the use of the Linac Coherent Light Source (LCLS) in Stanford, -- and Philip seems ready to make the trip.

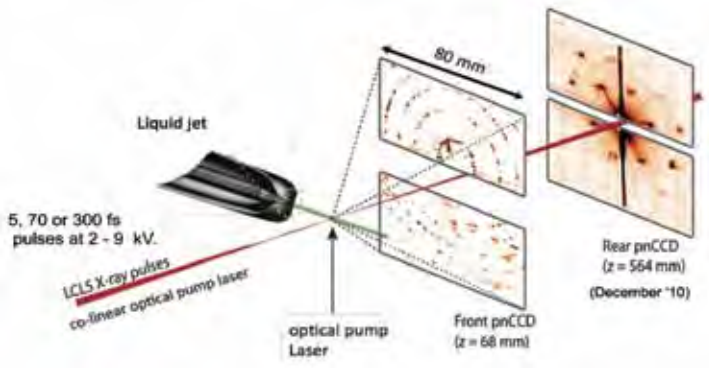
Jason Benedict

TR.01: Time-resolved and Charge Density - in honor of Philip Coppens

The 2011 *Transactions* symposium was dedicated to Philip Coppens in honor of his outstanding contributions to the field of crystallography and on the occasion of his 80th birthday. A remarkable assortment of friends, colleagues, collaborators, former students and postdocs presented a mixture of personal anecdotes and scientific research that Philip made possible.

Marc Messerschmidt, a former postdoc and now a lead scientist at the Linac Coherent Light Source (LCLS), discussed a few of the technical aspects of the experimental challenges which must be considered when moving from a synchrotron to an XFEL source, which is so bright that samples are annihilated with a single pulse. By using higher harmonics of the fundamental, the intensity of the source can be reduced to a point where samples are able to withstand thousands of x-ray pulses without significant damage. Because shorter wavelengths are used, this technique has the additional advantage that higher resolution data can be collected.

If one chooses to collect data using the fundamental, refreshing the sample at the operating frequency of the LCLS becomes critical. **John Spence*** at ASU, working with Bruce Doak and Uwe Weierstall, reported details of a remarkable liquid jet injector which fires (at up to 1 MHz) a constant stream of droplets one micron in diameter each containing a single protein crystal into the XFEL vacuum chamber. (See figure below.) The



Set-up used by Chapman et al. for femtosecond nanocrystallography. H. N. Chapman et al: Femtosecond x-ray protein nanocrystallography. Nature 470, 73-77 (2011); M. M. Seibert et al: Single minivirus particles intercepted and imaged with an x-ray laser. Nature 470, 78-81 (2011).

data consists of millions of diffraction patterns from Photosystem I nanocrystals prepared in the laboratory of Petra Fromme, each from a different nanocrystal with a random orientation. The small size of the crystals introduces shape-transform effects which can

cont'd on next page

** Editor's note: John Spence will receive the ACA's 2012 M. J. Buerger Award in Boston next year.*

L to r: Yu-Sheng Chen, Jason Benedict, Eric Collet, Ed Stevens, Lauren Hatcher, Claude Lecomte, Finn Larsen, Phillip Coppens, Alan Pinkerton, Sebastian Pillet, Radoslaw Kaminski, Yu Wang, Piero Macchi, Peter Lee, Wilfred Fullager, Krzysztof Wosniak, Lin Chen, Birger Dittrich, Parthapratim Munshi. Photo courtesy of Marvin Hackert.

complicate indexing; however, if treated properly, the inter-Bragg intensities can be used to reconstruct the size and shape of the nanocrystal and provide information for new phasing methods.

In addition to sharing her recent results on the structural dynamics of copper diimine complexes that can potentially serve as sensitizers in photovoltaic cells, **Lin Chen**, Northwestern U and ANL, also presented results of femtosecond x-ray absorption spectroscopy at LCLS illustrating the ultrafast reorganization of some iron spin crossover complexes.

Using light to drive the spin crossover transition in a variety of iron complexes was a popular topic throughout the symposium. **Eric Collet**, U Rennes, combined time-resolved x-ray diffraction with transient absorption spectroscopy on single crystals to demonstrate multi-step switching. The first step involves molecular transformation on a picosecond time scale due to laser excitation. This step is followed by lattice changes which arise from propagating sound waves on a nanosecond to microsecond time scale. The final step is diffusive heating which occurs on a microsecond to millisecond time scale and can lead to an enhancement of high spin complexes. **Sebastien Pillet**, CRM2, U Henri Poincaré, presented mechanistic and kinetic insights about light induced phase transitions by comparing measured reaction rates to numerical simulations which take into account the dimensionality of the nucleation and growth of the photoproduct within the lattice. **Yu Wang**, National Taiwan U, one of Philip's earliest postdocs, presented results of an iron spin crossover compound which undergoes a phase transition to a commensurate modulated structure which can be induced either thermally or by application of light at very low temperatures.

Using small crystals which require synchrotron radiation and a new LED illumination device, **Lauren Hatcher**, U Bath, recipient of the Synchrotron SIG's **Etter Young Lecturer** award, presented the first fully-reversible single crystal nitro-nitrito linkage isomer which could be induced either thermally or photochemically.

The rest of the talks featured charge density. **Claude Lecomte**, Nancy U, reported results of joint charge and spin density refinements on a Mn-Cu coordination polymer. Claude plans to measure the inelastic Compton scattering in a copper dimer complex soon, and thus measure the complete density matrix. After showing how molecular polarizabilites can be extracted from charge density data, **Piero Macchi**, U Bern, talked about the way to measure and/or calculate important properties such as solvent accessible voids, area of active surfaces and the influence of the host on the electronic properties in metal organic frameworks. Piero also detailed the cause of and solution to low energy x-ray contamination found in multilayer optics for the increasingly popular Mo microsources.

Another one of Philip's former postdocs, **Tibor Koritsanszky**, Middle Tennessee State U, described how to generate improved "bound atom" radial distribution functions which enable more precise interpretations of x-ray data from molecular crystals. **Ed Stevens**, U New Orleans, one of Philip's earliest PhD students, compared experimental charge densities to theoretical density functional calculations to analyze a variety of weak interactions in several small organic molecules. **Radoslaw Kaminski**, a PhD student at U Warsaw who has spent many months working in Philip's lab, presented structural features of new aryl boron complexes which included a nuanced analysis of aryl-aryl interactions and their comparison to the structure of benzene.

The 'proper' treatment of protons in charge density refinements inspired a majority of the remaining presentations and sparked lively discussions. After expressing his dissatisfaction that so many recent charge density studies used isotropic hydrogen thermal parameters, **Krzysztof Wozniak**, U Warsaw, compared and contrasted results of several methods for estimating anisotropic displacement parameters (ADPs) and their effects on properties such as weak C-H...O bond lengths and integrated charges. **Birger Dittrich**, U Goettingen, presented a method for generating theoretical ADPs by using QM/MM and ONIOM. (These are hybrid energy methods that combine different levels of theory into one calculation.) The result was considerably better than isotropic refinement. Because the calculated ADPs are not refined, Birger suggested this technique will allow multipole refinement of normal resolution data.

Stressing the importance of neutron data in charge density refinements, **Parthapratim Munshi**, ORNL, updated the audience on the capabilities of IMAGINE, a single crystal neutron diffractometer at the High Flux Isotope Reactor. The brilliance of this source will dramatically reduce the size of the crystals required and will increase the speed of data collection.

Alan Pinkerton, U Toledo, presented compelling evidence to the contrary, with several structures in which the proton positions and ADPs were derived entirely from x-ray data and were virtually indistinguishable from structures based on neutron data.

Finn Larsen devoted his entire lecture to his time with Philip and to Philip's impact on Aarhus University. Finn's recollections featured images from school newspapers he collected in Buffalo showing the war related riots of early 1970. Many more images followed including those of early devices, scientific achievements, and special photographs of both he and Philip at various meetings throughout their careers.

Thank you Philip for your exceptional contributions to the field of crystallography and Happy Birthday!

Jason Benedict

Posters About Crystallization

M54 by **DE Mortenson**, KA Satyshur, SH Gellman, and KT Forest described the pseudoracemic crystallization of a small protein containing a pentafluorophenylalanine in its hydrophobic core. **David Mortenson** is a graduate student who works jointly in the laboratories of Katrina Forest and Sam Gellman at U Wisconsin. David synthesized the peptides, crystallized them, collected diffraction data, employed direct methods or molecular replacement for phasing, and learned the art of macromolecular structure refinement.

Quasi-racemic structure of the villin headpiece subdomain (VHP) obtained by cocrystallization of L(F17->F$_5$Phe)VHP (left) and D-VHP (right). Although they differ by the presence of a fluorinated core residue, the two peptides are related by a pseudo-inversion. Chiu, T.K., Kubelka, J., Herbst-Irmer, R., Eaton, W.A., Hofrichter, J., Davies, D.R. PNAS 2005, 102, 7517. (Original crystal structure of non-racemic VHP)

While crystallization of pseudo-racemic protein mixtures in centrosymmetric space groups has been published (early 1990s) and used successfully since to determine (small) protein structures, few systematic investigations regarding the induction of racemic crystallization have been reported and the technique is not (yet?) widespread. The authors found that pseudo-racemic mixtures of small proteins (~35-mers) lead more readily to crystals than mixtures with only the L- or D-amino acid peptides, and in some cases, no crystals at all were obtained with the L-amino acid peptide alone. As suspected, centrosymmetric space groups result in some cases. The novelty here is the chemical incorporation of fluorinated phenylalanine as a stabilizing amino acid. In principle, any chemically achievable non-natural amino acid can be incorporated into a chemically synthesized peptide, and the method may be a generally applicable path towards the crystallization of often recalcitrant small peptides.

Bernhard Rupp

Posters on Macromolecular Structure

David Jeruzalmi, Harvard, presented **T-14**: *The UvrA-UvrB DNA Damage Sensor: Structure and Mechanism*. Nucleotide excision repair is a complex multistep pathway used by the cell for the repair of DNA duplex destabilizing lesions in the genome. In bacteria, the UvrA-UvrB complex acts as the damage sensor, identifying lesion-deformed DNA within the large excess of normal DNA present in the genome.

The crystal structure of UvrA•UvrB damage sensor is shown superimposed with the molecular envelope calculated from solution small-angle x-ray scattering (SAXS), which is depicted as gray mesh. The two protomers of UvrA are shown in different shades of gray, with the signature domain II in cyan and blue. UvrB molecules are shown in different shades of orange. The UvrB binding domain of UvrA are in different shades of green. For clarity, the boundaries of one molecule each of UvrA and UvrB are outlined.

The authors use a combination of SAXS and high resolution crystal data to determine the disposition and number of UvrB molecules present in the damage sensor complex. Also, the authors showed the structure of a novel UvrA conformer, which when compared to the UvrA-UvrB damage sensor structure reveals that conformational switching between an "open" damaged DNA binding state and a "closed" undamaged DNA binding state may be an aspect of the genome-scanning phase of damage sensing. This conformational switching appears to be dependent on ATP hydrolysis as the nucleotide state of UvrA affects the binding properties of the damage sensor complex to DNA. The structures suggest a mechanism by which, upon DNA damage identification, UvrA dislodges from the damage sensor complex, thus loading UvrB onto the lesion-deformed duplex in order to facilitate repair of the damaged DNA.

Rakhi Rajan, Northwestern: Mondragon lab, presented **T-07**: *Identification of the DNA Repair Active Site of Topoisomerase V by Structural and Functional Studies*. Topoisomerases are essential cellular enzymes that regulate the topology of DNA. Topoisomerase V is novel because it exhibits both topoisomerase and lyase DNA repair activity. The authors use a combination of biochemistry and structural biology to identify the residues on topoisomerase V that are responsible for its lyase DNA repair activity. A deletion construct consisting of the N-terminal 600 residues of topoisomerase V was shown to be the minimal fragment necessary for lyase activity. Mutation of three critical lysine residues to alanine within this fragment resulted in a 90% reduction of lyase activity as compared to that of the wild type protein. Finally, the crystal structure of a slightly longer fragment of topoisomerase V revealed the architecture of the DNA repair active site at atomic resolution and provides the molecular basis for further experiments aimed at understanding the structure/function relationships of this important enzyme.

Brian Bowman

SP.03: Keith Moffat receives the Patterson Award

Keith Moffat, Louis Block Professor in Biochemistry & Molecular Biology, is a founding member of the U Chicago's Institute for Biophysical Dynamics. He also serves on the U Chicago's Science Council, and serves on the Scientific Advisory Committees of the Advanced Light Source in Berkeley, the Linac Coherent Light Source at Stanford and the Energy Recovery Linac Project at Cornell.

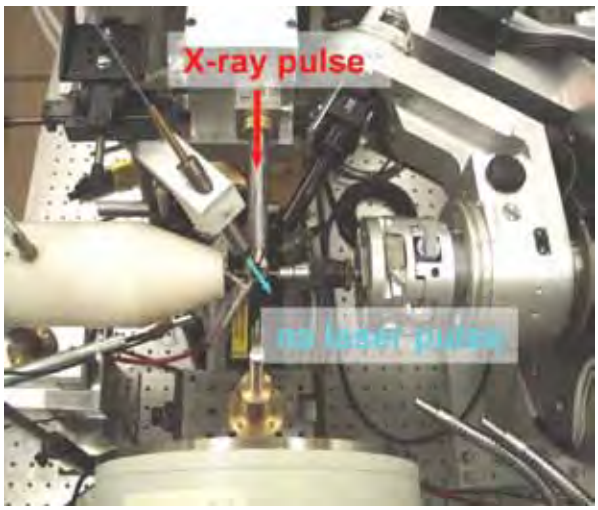
Keith graduated with first-class honors in physics from U Edinburgh. He then moved to King's College, Cambridge to do his PhD in biophysics with Max Perutz at the MRC's Laboratory of Molecular Biology.

Keith accepted the **2011 Patterson Award** from ACA President Tom Koetzle (see above). The first part of Keith's talk *Time-resolved Macromolecular Crystallography: From Hours to Femtoseconds* was historical. Max Perutz, who died in 2002, was an early mentor. In 1969, when he was a postdoc with Quentin Gibson at Cornell, excellent time resolution was not possible since x-ray exposures were typically measured in hours. It was clear by the time Keith was a beginning professor at Cornell in 1973 that two things would be needed: a synchrotron and a high power pulsed laser. Planning for what ultimately became the CHESS synchrotron facility began in 1976. MacCHESS began informally in 1981 and became fully operational with its first NIH funding in 1983. With Keith's demonsration of the possibilities of Laue diffraction, *the time resolution dropped to the tens of seconds*. Keith directed MacCHESS 1983-1990. In 1987, while on sabbatical, Keith solved, together with Durward Cruickshank and John Helliwell, the energy overlap problem that had previously hindered the ability to extract accurate and complete sets of structure amplitudes from Laue images. MacCHESS staff also developed and used image plate x-ray detectors; precursors of today's accurate CCD detectors.

Before leaving CHESS, Keith and his students borrowed a prototype of the APS undulator (for x-ray pulses) and in combination with the first ultrafast shutter train that could isolate single x-ray pulses at CHESS or the ESRF, went on to solve macromolecular structures. Now the time resolution was *120 picoseconds, for a 10^4 gain in time*. The algorithms for extracting structure factors were developed by a number of people including Marian Szebenyi, Brenda Smith Temple, and Zhong Ren.

In 1990 Keith moved to U Chicago where he was made Director of BioCARS. He served as Deputy Provost for Research there 2002-2010.

Since 2004, Keith and his colleagues have published many papers on time-resolved crystallography. They studied photoactive yellow protein (PYP) because it is a blue-light photoreceptor exhibiting a photocycle in which several intermediates span *time- scales from nanoseconds to seconds*. At last the action of a molecule could be tracked in time. Keith showed a movie displaying the photocycle of PYP[1]. He went on to describe pump-probe experiments with XFELS on "large" crystals of PYP that diffracted at better than 1 Å (see image below).



Pulse-probe instrument at BioCARS

In order to solve these structures they would need to characterize the spectrum within the "monochromatic" source. In other work on *not* light sensitive problems he reported using a new "optogenetics"[2] technique to design and engineer molecules and cellular processes to become light sensitive; specifically, they designed a histidine kinase to render it sensitive to blue light.

1. Ren, Z., B. Perman, V. Srajer, Z. Ren, T. -Y Teng, C. Pradervand, D. Bourgeois, F. Schotte, T. Ursby, M. Wulff, R. Kort, K. Moffat K "Molecular movie at 1.8 Å resolution displays the photocycle of photoactive yellow protein, a eubacterial blue-light receptor, from nanoseconds to seconds," Biochemistry (Wash.) 40 (46) 13788-13801 (2001).

2. In 2010 optogenetics was deemed the Method of the Year by Nature Methods. See www.stanford.edu/group/dlab/optogenetics/index.html.

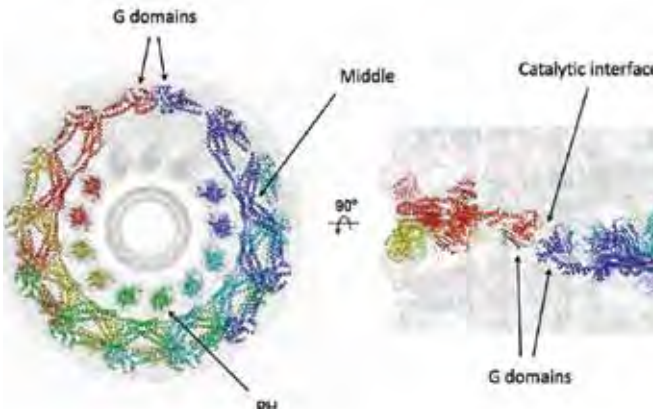
Connie Rajnak

Jenny Glusker

In back, l to r: Joe Ng, Brian Bowman, John-Paul Bacik, Fred Dyda, Dayne West. In front: Zachary Wood, Erik Montemayor,Katherine Hicks, Zoe Fisher.

1.03: Structural Enzymology II- Mechanistic

Fred Dyda, NIDDK, gave an excellent talk describing the mechanism of dynamin. Dynamin catalyzes the scission event of clathrin-coated vesicles that separates them from the cell membrane using GTP hydrolysis. Previous structural work using non-hydrolyzable GTP analogs were unable to resolve the mechanism of membrane fission. Dyda showed that a transition-state analog, GDP-AlF4 induced dynamin's GTPase domains to self-assemble into dimers. This dimerization is required for dynamin's assembly stimulated GTP-ase activity. In addition, analysis of a new, 11Å resolution cryo-EM map of full length dynamin assembled on lipid tubes allowed docking of all fragments of dynamin, revealing how the characteristic helical arrays form. These helical arrays are needed to promote membrane fission. Furthermore, comparing the GDP-AlF4 dynamin complexes to GMP-PCP-stabilized complexes allowed Dyda and co-workers to identify transition state-dependent conformational changes that are key for the scission event to occur. These conformational changes of the dynamin complex suggests a mechanism by which GTP hydrolysis could induce a comformational tightening of the dimer, and drive membrane fission.



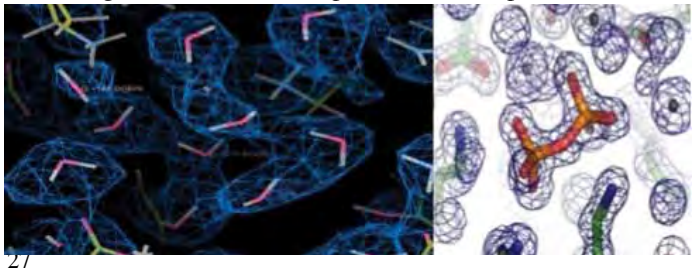
From Fred Dyda: Cryo-EM maps with the crystal structure of the G-domains of Dynamin docked into experimental density, showing that at a minimum, one complete turn of the helical complex must form for scission.

From Joe Ng: Neutron density map showing 'winged' waters in the hydrogen bonding network of IPPase.

Katherine Hicks*, Cornell, presented her work on a novel urate oxidase, HpxO from *Klebsiella pneumoniae*, that is involved in purine catabolism. All urate oxidases characterized to date are cofactor-independent and utilize a radical-mediated mechanism to hydroxylate urate and form the unstable product, 5-hydroxyisourate. Through a combination of biochemical and structural studies, Hicks and collaborators demonstrated that *K. pneumoniae* HpxO is a flavin-dependent monooxygenase that utilizes the cofactors FAD and NAD(P)H to oxidize urate. Structural characterization of the wild-type enzyme complexed with urate showed that HpxO has a typical fold for a Class A flavin-dependent monooxygenase. The FAD cofactor, which is known to undergo movement during the reaction cycle, adopts the IN conformation in the crystal structure. This conformation allows for FAD oxygenation by molecular oxygen and leads to the formation of a flavin-hydroperoxide intermediate, which then hydroxylates the urate substrate. Structural characterization identified an active site arginine residue that is involved in hydrogen bonding interactions with the substrate. Kinetic characterization revealed that mutation of this residue leads to the decoupling of NAD(P)H oxidation from urate hydroxylation. The structure of this mutant has been determined and provides insight into how HpxO catalyzes a reaction normally carried out through radical chemistry.

Dayne West, U Florida, talked about the importance of a 'water wire' in the active site of carbonic anhydrase II (CAII). CAII uses a Zn ion to stabilize a nucleophilic hydroxide. This hydroxide attacks CO_2 to produce bicarbonate. The rate-limiting step of CAII appears to be the transfer of protons from a Zn-bound water to bulk solvent. By changing the polarity of the active site, West showed that the water structure of the active site could be altered with significant consequences to the catalytic rates. One interesting mutation (Y7F) reduced the number of ordered water molecules in the active site, and actually increased the reaction rate 10-fold.

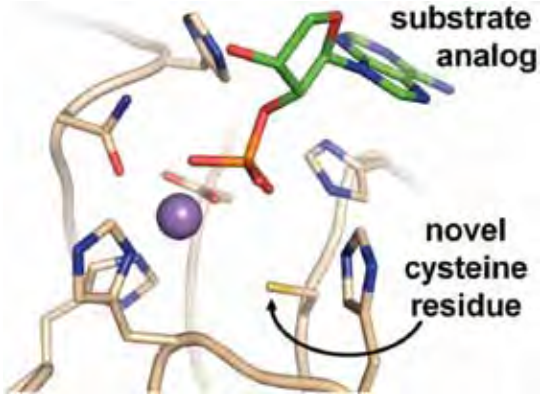
Joe Ng, U Alabama-Huntsville, described a structure-based mechanism of an archaeal inorganic pyrophophatase (IPPase) from *Thermococcus thioreducins*. The action of this enzyme is important in shifting the overall equilibrium in favor of synthesis during a number of ATP-dependent cellular processes such as in the



polymerization of nucleic acids, production of coenzymes and proteins and sulfate assimilation pathways. The reported work on the IPPase has shed new light on what was an accepted mechanism. Combining x-ray and neutron diffraction studies, Joe's research team observed that a conserved water network in the catalytic site appears to be important for transferring protons to the bulk solvent and nucleophilic activation. Here, a conserved water network shown in the neutron structure appears to form a relay system for transferring a proton from the nucleophilic water to bulk solvent (see figure on preceding page). The substrate bound in the catalytic site is shown in the high resolution x-ray structure.

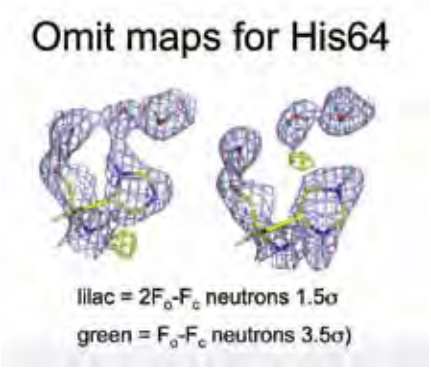
Brian Bowman*, Harvard, provided us with a unique view of DNA repair. While most DNA repair complexes have focused on the interaction between the enzyme and the lesion, Bowman considered how DNA repair enzymes interrogate undamaged DNA. To do this, Bowman designed a disulfide crosslink between the DNA glycosylase AlkA and an oligonucleotide to trap a stable complex. The crystal structure reveals that the enzyme uses a leucine residue as a probe to interrogate the minor groove of the DNA in search of lesions. Additionally, a comparison of the present structure with that of AlkA-bound damaged DNA revealed conformational changes in both the DNA and protein as the glycosylase transitions from the interrogation of undamaged DNA to catalysis of lesion base excision.

Eric Montemayor, UT Health Sciences Center at San Antonio, presented several crystal structures of the intron debranching enzyme Dbr1. These structures were the first to be determined of an enzyme capable of hydrolyzing the unique 2',5'-phosphodiester bond within spliced introns. Montemayor demonstrated that Dbr1 contains a metallophosphoesterase domain that is linked to an accessory domain lacking detectible similarity to any other protein. An active site cysteine was also observed in a position normally occupied by aspartate in other metallophosphoesterases. *In vivo* functional data revealed that this cysteine is required for enzyme activity. Inductively coupled mass spectrometry (ICP-MS) data, in combination with a substrate analog bound structure, were used to propose an enzyme mechanism for Dbr1.



From Eric Montemayor: Dbr1 active site with novel Cys labeled.

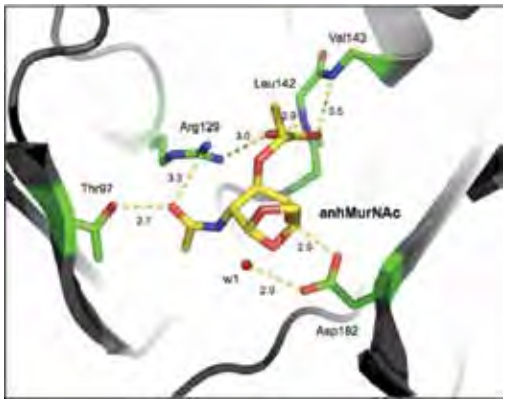
Zoe Fisher, LANL, gave an exciting talk on her recent work on human carbonic anhydrase II (HCA II). HCA II converts carbon dioxide to bicarbonate using a zinc-bound hydroxide as a nucleophile. The rate limiting step is the transfer of a proton from the Zn-bound water molecule to form the catalytic hydroxide. Previous proposed mechanisms implicated the active site histidine 64 as a proton shuttle in the mechanism, accepting the excess proton from the hydrogen bonded water network that spans the 8 Å distance across the active site. Using x-ray and neutron diffraction studies, Fisher sheds new light on the role of histidine and the contribution of the water networks and hydrogen bonds. For the first time a complete hydrogen bonded water network is observed, ready to transfer the proton to His64; see figure.



From Zoe Fisher: Neutron density showing the His 64 in two conformations, suggesting that rotation about Chi 2 may mediate proton transfer to bulk solvent.

Previous work suggested that the histidine changes rotameric states about the Chi 1 torsion angle to transfer the proton to bulk solvent. Fisher's work also shows that the protonated histidine can flip, offering an alternative pathway to transferring the proton by rotating about Chi 2.

John-Paul Bacik, U Manitoba, presented his work on anhydro-N-acetylmuramic acid kinase (AnmK). John-Paul proposed a mechanism by which AnmK cleaves the 1,6-anhydro bond of anhMurNAc and phosphorylates the released O6 atom based on crystallographic and functional studies of AnmK. The mechanism is analagous to inverting glycoside hydrolases, in which a conserved aspartic acid residue (Asp182) deprotonates a water molecule to initiate hydrolysis of the 1,6 anhydro bond by attacking the anomeric carbon of the sugar. John-Paul suggests that the freed O6 atom attacks the gamma phosphate of ATP, resulting in transfer of the phosphate to the sugar, presumably through an in-line associative mechanism of phosphoryl transfer.



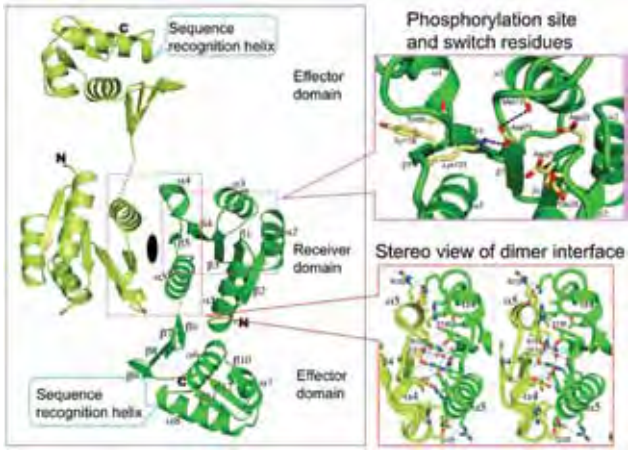
From John-Paul Bacik: AnmK active site in complex with its natural sugar substrate, anhMurNAc. Bacik, J. P., Whitworth, G. E., Stubbs, K. A., Yadav, A. K., Martin, D. R., Bailey-Elkin, B. A., Vocadlo, D. J. and Mark, B. L. (2011) J Biol Chem 286, 12283-12291.

Zachary Wood

***** Editor's note: Images from Katherine Hicks and Brian Bowman are on the cover (see On the Cover, page 9)***

Macromolecular Structure & Function Posters

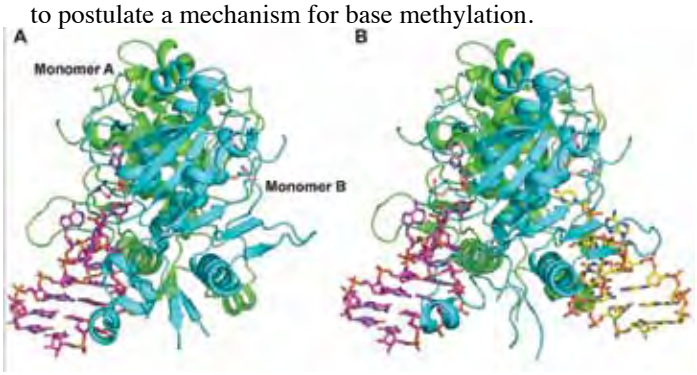
M32: *Crystal Structure of PhoP from Mycobacterium tuberculosis,* by **Shuishu Wang** and Smita Menon, Uniformed Service U, presented the first crystal structure obtained for this response regulator which controls the expression of over 110 *M. tuberculosis* genes and is an important virulence factor. Like other members of this protein family, PhoP is composed of a receiver domain and a DNA-binding effector domain and is activated by phosphorylation of a conserved aspartate, Asp71, in the effector domain. The two domains are connected by a flexible, protease-sensitive linker which is unstructured. This, coupled with the lack of interactions between domains, suggests that the domains are free to move independently.



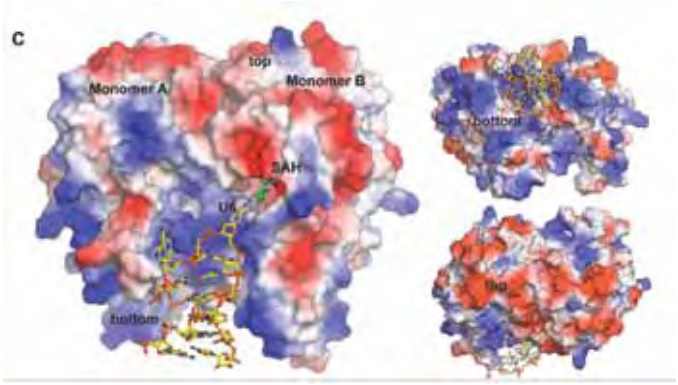
Although size exclusion chromatography showed that non-phosphorylated PhoP exists in a concentration-dependent equilibrium between monomeric and dimeric states, it crystallizes as a dimer mediated by interactions between receiver domains. Phosphorylation of Asp71 may alter interactions with conserved "switch residues" which link phosphorylation to structural changes. This conformational change is thought to alter the dimer interface, strengthening intermolecular interactions and stabilizing the dimer. This in turn enhances DNA-binding by bringing two copies of the effector domain together to bind to the direct DNA repeats for which PhoP family members are specific. Given its importance for *M. tuberculosis* virulence, PhoP is an attractive drug target. Insights gained from the 3D structure will help to design drugs against TB. ***For more detail see Menon & Wang, Biochemistry, DOI: 10.1021/bi2005575, (available online).***

Eric Yuki

T65: *Structural Characterization of the Role of Nep1, a Pseudouridine N1-Specific Methyltransferase, in Ribosome Biogenesis* by **Nicole LaRonde-LeBlanc**, S. Thomas, C. Keller, A. Syzk, & J. Cannon, U Maryland, describes an enzyme required for maturation of 18S rRNA, Nep1. **T65** presented the dimeric structure of Nep1 alone and in complex with one and two molecules of cognate RNA. Consistent with its role as a methyltransferase, a uridine base is flipped into the active site. Interactions between the substrate, protein residues and a co-crystallized S-adenosyl homocysteine allowed the authors to postulate a mechanism for base methylation.

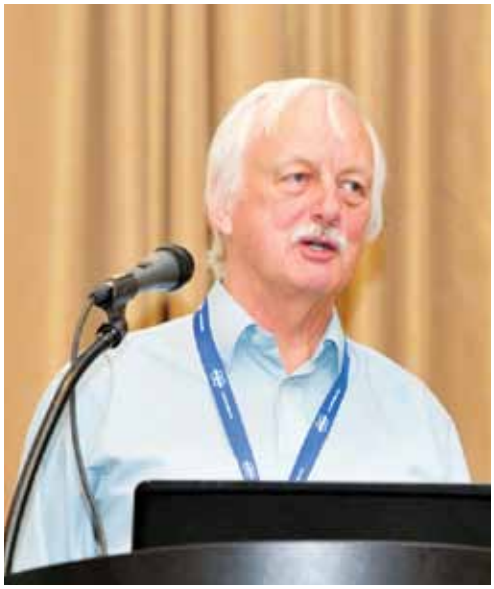


Nep1 is also thought to play a role in loading the ribosomal protein S19. The Nep1-RNA complex structure indicated that the protein stabilizes a stem-loop structure of the RNA. Apparently the Nep1 binding alters the rRNA secondary structure, an observation that has important implications for Nep1 RNA chaperone activity. The idea is that when Nep1 binds to 18S rRNA the structure is altered; this promotes binding to the ribosomal protein S19, - an RNA chaperone activity.



This work has been published: see Nucleic Acids Res. 39, 2445. © Thomas, Keller, Szyk, Cannon & LaRonde-LeBlanc, 2010. Published by Oxford University Press.

SP.04: Fankuchen Award to David Watkin



David Watkin of Oxford was the 2010 recipient of the **Fankuchen Award** which recognizes *contributions to crystallographic research by one who is known to be an effective teacher of crystallography*. He was unable to accept his award last year in Chicago, but we were pleased that he could be at the ACA meeting in New Orleans this year. He was cited "for his stewardship of the crystallographic software package CRYSTALS for x-ray structure refinement and analysis." Watkin is also the co-founder of the successful BCA Intensive Course in x-ray structure analysis and is well recognized for his love and teaching of crystallography.

His award lecture: ***Crystallography – Is the Gold Standard Getting Tarnished*** was well received. He began by recalling his early years in crystallography and ended with future challenges. He reviewed the development of the field and the impact of computing on it. He noted that today one could do more computing in one day on a PC than was possible in 5 years of computing on a VAX computer as recently as 1990. He also noted that in the early days before computers, crystallographers had to think more to solve structures, and cited as an example Kathleen Lonsdale, who was able to solve the structure of hexachlorobenzene in 1931 as a rigid body using just two parameters. He then posed the questions - - *has crystallography turned into "stamp collecting" as we turn out more and more structures?* and -- *should we be concerned if not every atom of every structure is refined as well as possible?*

David being congratulated after his lecture by Bruce Foxman.

He proposed that crystallographers should consider the purpose of doing an analysis before determining the structure, and if a quick refinement answers the question being asked, then investigators should not be forced to do exhaustive refinements. He said in conclusion that there are many challenges remaining for the upcoming generations of young crystallographers beyond exciting new structures - such as structure prediction, crystal growth, and how molecules share "information" with each other.

Marvin Hackert

SP.05: Plenary Lecture by Nadrian Seeman

Ned Seeman, the Margaret and Herman Sokol Professor of Chemistry at New York University, gave a plenary lecture entitled ***DNA: Not Merely the Secret of Life***. With a command of knowledge that can only come from the founder of the respective field, Ned delivered a comprehensive overview of DNA nanotechnology that had broad scientific appeal. This introspective talk highlighted the growth of the field that Ned created, all the while exploring the use of DNA and other nucleic acids as molecular scaffolds for other species in efforts to engineer nanoelectronic and nanomechanical devices. Ned shared the **2010 Kavli Prize for Nanoscience** with Don Eigler for the "development of unprecedented methods to control matter on the nanoscale".

Ned craftily took the liberty to draw comparisons between his beautiful molecular architectures and the seemingly endless source of motifs present in cultural heritage pieces across the global-historical art spectrum.

His successful research efforts are in no small part due to his ability to recognize, and in some cases predict, the potential function of these systems, and this has sustained his position at the front-line of molecular nano-research. In addition to designing nanotubes and polyhedra Ned has designed a series of "walking" machines based on reversible binding DNA motifs and even a nano-assembly line capable of producing engineered and functional molecules. This session was followed by the complementary session **Small Molecule Molecular Machines** (see page opposite).

Chris Incarvito

Editor's note: see Science, 3rd June, 2011, vol 332: DNA Nanotechnology Grows Up, a NewsFocus article by Robert F. Service reporting on the 8th Annual Conference on Foundations of Nanoscience held in April, 2011 at Snowbird, Utah. Service included some historical background on Ned Seeman and others in this field.

L to r: Miguel Garcia-Garibay, Carla Daly, Cortnie Vogelsberg, Jeremiah Gassensmith, Gokhan Barin, Chris Incarvito.
Photo by the NO student AV crew.

8.18: Small Molecule Molecular Machines

Jeremiah Gassensmith, Northwestern, began with an intriguing overview of supramolecular assemblies and their associated mechanical properties. Rotaxanes and catenanes are interlocked molecules (that is they have two or more non-covalently linked substructures that are "locked" by means of encumbering constraints- a covalent bond would have to be broken to free the substructure). These provide scaffolds on which nanodevices and even bio-imaging agents can be affixed to provide researchers with an extensive toolbox.

Cortnie Vogelsberg, UCLA, described a clever synthesis of a series of molecular rotors that exploit electron-rich carbon atoms and electron-deficient iodines. Variable temperature solid-state NMR experiments revealed discrete molecular rotors in the gigahertz region. Her group synthesized and crystallized 1,4-bis(iodoethynyl) bicyclo[2.2.2]-octane (BIBCO). BIBCO molecular rotors are some of the fastest engineered molecular machines observed to date.

Carla Daly, University College Cork, described her efforts to create a hydrogen bond molecular switch based on the oxidation state of sulfur. Her structure/function analysis focused on the use of hydrogen bonds in amides as the mobile switch component. Carla also described the synthesis of these materials and the advantage of using a modular approach to vary attributes within a single molecule.

Miguel Garcia-Garibay, UCLA, gave an overview of the challenges facing this emerging field and presented elegant examples of systems that benefit as much from their immobility as those which are engineered to perform a specific motion. Miguel artfully described the symbiotic relationship between chemical analytical tools and x-ray crystallography.

Gokahn Barin, Northwestern, concluded with a talk that incorporated aspects of the previous presentations. His work demonstrated that the introduction of Cu-templated pseudorotaxanates into a metal-organic framework (MOF) can result in carefully engineered molecular switches that preserve the structural integrity of the MOFs.

Chris Incarvito

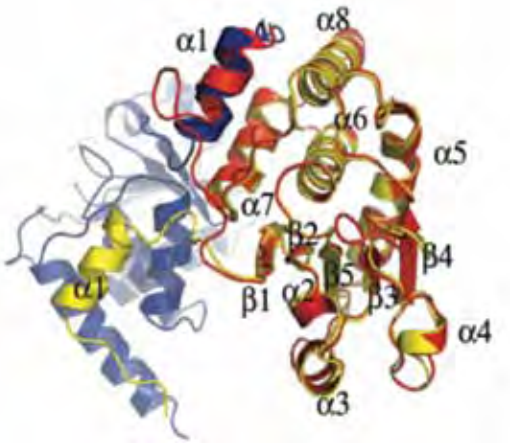
SP.01 Etter Early Career Award

The **2011 Margaret C. Etter Early Career Award** went to **Yurij Mozharivskyj**, an Associate Professor in the Department of Chemistry at McMaster U. Yurij's talk discussed his efforts to form guiding principles for the design of new magnetocaloric materials, which have potential for large-scale industrial applications, such as magnetic refrigeration.

From the many abstracts submitted, the YSSIG selected 8 people to present talks. They chose **Karen Ruane**, McGill U, to receive their **Etter Young Lecturer Award**. Karen described the identification of a new class of chaperones from *E. coli*, denoted Spy. The structure of Spy differs from all previously solved chaperone structures in that Spy functions as an ATP-independent chaperone which suppresses protein aggregation and aids in protein refolding.

ACA President Tom Koetzle (left) presenting the Etter Award to Yurij Mozharivskyj.

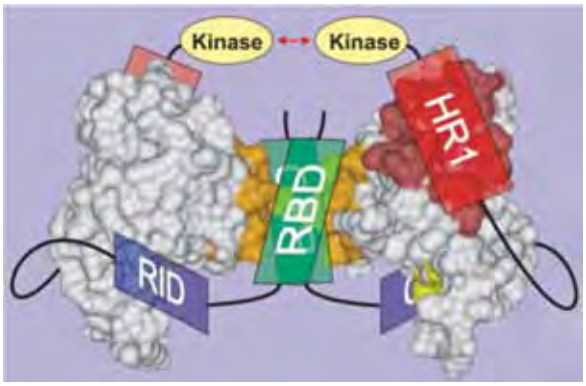
James Fairman, NIDDK, described the crystallization of the N-terminal transmembrane β-domain of intimin using three distinct methods. Initmin is an autotransporter from Enterohemorrhagic *E. coli* (EHEC). The β-domain is implicated in facilitating the transport of the virulent passenger domain of intimin through the outer membrane; understanding this transport mechanism will be useful in the design of vaccines against EHEC. These three structures will be used to compare the packing interactions and structural differences observed with crystals obtained by the three methods. What is important about this structure is that it is one of the first membrane protein structures to be solved by lipidic cubic phase (LCP) to such high resolution (1.85 Å).



George Lountos, NCI-Frederick, reported the x-ray crystal structure of a human dual specificity phosphatase-27 (DUSP27), which has a varied electrostatic potential surface around the substrate binding pocket, suggesting different substrate binding specificity between homologues. DUSP27 has a domain swapped dimer not found in other homologues (see image). Differing binding pockets could represent different physiological substrates; the 3D structure will aid in the design of potent inhibitors that may be useful anticancer therapies.

From George Lountos: X-ray structure of DUSP27. Lountos, G.T., Tropea, J.E., and Waugh, D.S. Structure of human dual-specificity phosphatase 27 at 2.38 Å resolution. Acta Cryst. D Biological Crystallography. 67:471-9 (2011)

Badri Dubey, U Duesseldorf, discussed structure-function relationships in the GTPase-effector using RhoA as a model. Badri focused on the interacting surfaces of Rho GTPases and the quantitative analysis of their structures in complex with regulators and effectors. One such effector is Rho-associated coiled-coil kinase (ROCK). The proposed model of Rho-mediated ROCK activation shows dimeric coiled-coil ROCK-RBD recognizing and interacting with the switch region of two GTP-bound RhoA molecules. The RID domains then approach loop 6 of RhoA, and the resulting structural rearrangement cancels the autoinhibitory function of the kinase. the HR1 domains bind to contact site 1 of the RhoA molecules and induce dimerization of the kinase domains of the two ROCK molecules, leading to autophosphorylation and activation.

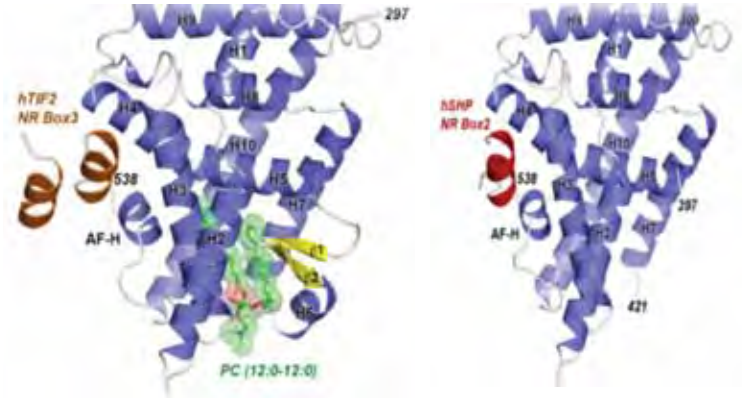


From Badri Dubey: RhoA mediated ROCK activation mechanism. This figure was originally published in J. Biol. Chem. (2004) 279, pp 53419-26, by L. Blumenstein & M.R. Ahmadian, Models of the cooperative mechanism for Rho effector recognition: Implications for RhoA-mediated effector activation. © the American Society for Biochem. & Molecular Biol.

Pawel Sledz, Cambridge, presented an extensive crystallographic characterization of the C-terminal polo-box domain (PBD) found in polo-like kinase 1 (Plk1) with known phosphopeptide ligands. This work is important because it affords insight into the possible molecular recognition motifs used by Plk1 for progression through mitosis, and helps to explain how a single phosphate-binding site can bind a variety of ligands.

Kinlin Chao, U Maryland, identified critical protein-protein interface residues specific for the interactions between the RON tyrosine kinase receptor and the β-chain of the macrophage stimulating protein (MSPβ), which regulates cell adhesion, invasion motility and apoptosis. The structure of the binding domain of RON in complex with MSPβ solved by Kinlin and colleagues, suggests that the mechanism for RON receptor activation may be different from the proposed model for other tyrosine kinase receptors.

Ron Diskin, Caltech, presented the crystal structure of a clade C HIV-1 gp120 bound to the CD4 host receptor. In addition to providing the first visualization of an autoreactive antibody Fab complex with two different antigens, this structure reveals important interactions that facilitate cross clade structural analysis.



From Paul Musille, on the left: ligand-bound structure: ribbon diagram of DLPC bound hLRH-LBD (α-helices, blue; β-strands, yellow) with the human TIF (hTIF) NR box 3 peptide (orange). The bound phospholipid is depicted as sticks (C, green; O, red; P, magenta; N, blue) surrounded by transparent spheres.

On the right: apo structure: ribbon diagram of apo hLRH-LBD (α-helices, blue; β-strands, yellow) with the human SHP (hSHP) NR box 1 peptide (red). Residues 398-420 lack traceable main chain density and have been omitted from the structure.

Paul Musille, Emory, gave a very interesting talk on the characterization of liver receptor homologue-1 (LRH-1). LRH-1 regulates genes involved in lipid homoestasis and steroidogenesis. Paul used molecular snapshots to visualize LRH-1 in its apo and liganded forms. It is hoped that this will aid the understanding of activation of nuclear receptors, and will help in the evaluation of other structural components of nuclear receptors that are involved in the functional switch from active to inactive.

Jamaine Davis

L to r: Adam Godzik, Matthew Zimmerman, John Johnson, Wladek Minor, Thomas Womack, Steve Bryant, Helen Berman, Howard Robinson. Photo by the NO student AV crew.

1.01: Use of Databases in Structural Biology

1.04: Membrane Protein Crystallization

L to r: Jian Xu, Mark Hunter, Michael Wiener, James Love, Hilary Stevenson. Photo by the NO student AV crew.

Across bottom of page, after the Awards Banquet, from left: Sue Byram, Angela Roa-Espinosa, Jenny Glusker, Andrey Yakovenko, Carroll Johnson, Tiffany Kinnibrugh.

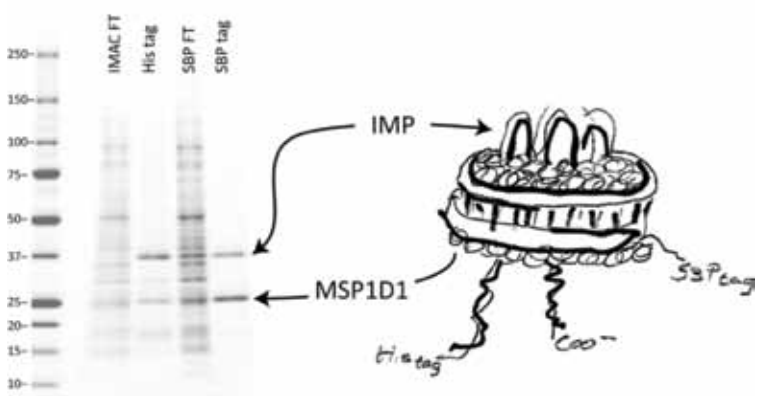
Bottom right: Ray Davis

1.02: L to R: Ward Smith, Margaret Gabanyi, Catherine Cormier, Michael Malkowski, Brian Fox, George Phillips, Robert Stroud. Photo by the NO student AV crew.

1.02 PSI: Tools for the Home Lab

One of the signature achievements of the Protein Structure Initiative has been the development of tools to improve the efficiency of macromolecular structure determination. These tools are available to all and this session highlighted just a few of them. **Michael Malkowski** began the session with a description of the crystallization service at Hauptman Woodward Institute and the Membrane Protein Structural Biology Consortium that is freely available to all. Mike described the development of crystallization screens for soluble and membrane proteins as well as the systematic evaluation of phase boundary data for chemically diverse crystallization conditions including eleven detergents commonly used to crystallize membrane proteins.

George Phillips, U Wisconsin, described his program ACMI for automatic interpretation of electron density maps at low resolution. The approach uses a probabilistic model to determine the protein backbone trace. The application of a filtering procedure is then used to produce a set of accurate all-atom protein structures for maps at around 3.5 Å resolution.



From Brian Fox: Purification of His-tagged integral membrane proteins (IMP) that are co-translated with streptavidin binding protein (SBP)-tagged membrane scaffold proteins. Plasmids and protocols available at www.uwmenbraneproteins.org/ and http://dnasu.asu.edu/DNASU/

Brian Fox, U Wisconsin, presented his experiences with cell-free expression systems to produce membrane proteins for structure studies. Brian has implemented automated, high throughput cell free expression for protein production, including membrane proteins such as components of the sphingolipid biosynthesis pathway. (See figure above.) Fox stressed that cell-free expression technology is also available for use in small labs that do not have the robotics used to automate the process, ensuring that the small scale reactions usually can be scaled up seamlessly.

The structural proteomics of eukaryotic membrane proteins was described by **Robert Stroud**, UCSF, head of the Membrane Protein Expression Center. Stroud described the approach and reported the results from expression systems for eukaryotic membrane proteins in yeast and mammalian cells. The Structural Biology Knowledgebase Web Portal was described by **Margaret Gabanyi**, Rutgers. The SBKB is a central location for access to advances in structural biology and structural genomics including linking protein sequences, three-dimensional structures and models to biological function. The SBKB contains information about protocols, materials and technologies related to macromolecular structure and function, including more tools for the home lab.

Catherine Cormier, AZ State U, presented an introduction to the PSI: Biology-Materials Repository. The Materials Repository is a resource for protein expression plasmids. The repository currently stores and distributes over 130,000 plasmids, including all protein expression plasmids from the PSI. Investigators are able to search for and order plasmids, and receive them under a simplified Institution-wide materials transfer agreement.

Alexandra Ainsztein and Ward Smith

NO student AV crew. In back, from left: Michael Stevens, Nicole Bryson, Kimisha Sawyers, Patrick Dupart. In front: Tuyet-Trinh Nguyen, Jamal Pratt and Alaina Guillory.

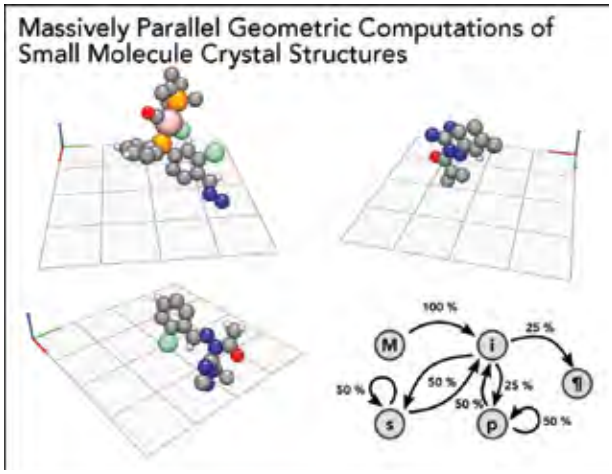
Bill Duax looking up at Chris Gilmore looking up.

L to r: Jason Mercer, Olga Smirnova, John MacDonald, Jennifer Swift, Peter Müller, Carroll Johnson, Herbert Bernstein.

2.01: General Interest I

Jennifer Swift, Georgetown U, described her use of atomic force microscopy and dynamic light scattering to study the growth and particle sizes of monosodium urate monohydrate crystals, the crystals associated with development of gout. At physiological pH, 7mM urate solutions gave crystals 2 μm in size. Images of crystal growth showed both monolayers and multilayers. The multilayers were shown to grow faster at all concentrations.

The General Interest SIG chose **Jason Mercer**, Memorial U of Newfoundland, to receive the **Etter Young Lecturer Award**. Jason discussed a model for structure prediction that initially arose out of an application he wrote to describe the motions of spaceships. His modified application allows high throughput structural calculations aggregated over one to thousands of processors. Geometric queries can be posed and evaluated against hundreds of thousands of small molecule crystal structures, for example, the automatic extraction of a 3-D grammar for distances between like fragments using Markov Chains. Jason also is working on an algorithm using Monte Carlo steps to define molecular volumes.



from Jason Mercer and Louise Dawe: Random small molecule fragments generated using a method similar to text generation with Markov Chains. Molecular grammar, spatial relationships and transition rates mined from the Cambridge Structural Database were run on hundreds of processors using custom made software to generate the fragments.

John MacDonald, Worcester Polytechnic Inst., reported his results on the phase separation of different enantiomers when crystallization takes place on a chiral surface. Up to 75% enantiomeric enhancement was obtained compared to crystallization on glass. The chiral surfaces were self-assembled monolayers of D- or L- cysteine and the compounds were D/L-3-phenylacetic acid and D/L-N-acetylleucine. Orientated crystal growth was also observed.

Olga Smirnova, Kyoto U, discussed an alternative to quantum theory. The use of a wave density tensor, the 1-D Bragg's Law, is to be used with sub-boson particles. The Bragg planes correspond to a spectrum with different wavelengths in the x-ray region.

Herb Bernstein, Dowling College, explained the definition of Lee-Richards molecular surfaces and how a rolling ball is used as a probe atom. The current method of near trees uses a random set of "balanced trees" to computationally simplify the calculation of molecular surfaces when the number of atoms is large.

Carroll Johnson, ORNL, summarized developments in crystal structure rigidity dynamics via thermal motion analysis. A new approach uses rotational (symplectic) space rather than translational space. This approach could be used to analyze transition dynamics in non-destructive phase changes.

Since David Rae was unable to attend the meeting, the remaining time was used for a question and answer period involving all the presenters.

Marilyn Olmstead

L to r: Joe Olechno, Garth Simpson, Juergen Graf, Melissa Menard, Mathias Meyer, Bradley Hintz, Allen Oliver, Charles Campana, Bill Duax. (Not pictured: Stefan Kolek.)
Photo by the NO student AV crew.

2.02: General Interest II

Charles Campana, Bruker-AXS, began by presenting some unconventional techniques for utilizing 2-D area detectors, in particular single crystal instruments being used for powder diffraction. Continuing the hardware theme, **Jurgen Graf**, Incoatec GmbH, discussed progress in developing new multi-layer optics for micro-source x-ray tubes. The increased flux observed would be a boon to any laboratory. **Bradley Hintze**, Duke, gave an enlightening talk on the problems facing validation of structures in the PDB. His research is aimed at improving the quality of archived data through the use of the Richardson Lab's ***MolProbity*** software suite.

"What is data quality?" was the question asked by **Matthias Meyer**, Agilent Technologies, as he reported data collection techniques on multiple wavelength diffractometers that should improve data quality. **Melissa Menard**, LSU, discussed her research into the nature of superconducting mimics and the methodology behind developing such new materials. Because of twinning and mixed occupancy sites, these new materials present many challenges to analysis.

Bill Duax, Hauptman-Woodward Inst., gave an energetic and enthusiastic talk on his Ribosome S19 findings and on the methodology for sequencing the S19 gene. This research, as well as the data analysis, was performed by a cohort of high school students.

The use of lasers to identify new compounds, especially in drug design, was reported by **Garth Simpson**, Purdue. This laser technique applies the knowledge that chiral compounds (including many organics and drugs) are often second harmonic generators; specific wavelengths of laser emissions can be examined to rapidly determine if a new target compound has been synthesized. **Joe Olechno**, Labcyte Inc., continued the hardware discussion with his talk about a new acoustic method for accurate sample preparation. In particular, accurate and rapid screening techniques have been developed with this new technology. Finally, **Stefan Kolek**, Douglas Instruments, discussed a topic of interest to all crystallographers - crystal growth. In particular he emphasized the use of micro-seeding to enhance production of crystals suitable for x-ray diffraction studies.

Allen Oliver

L to r: Nickolay Chirgadze, David Cooper, John Badger, Talabady Bhat, Felix Vajdos, Jia Sheng, Steven Sheriff.
Photo by the NO student AV crew.

3.01: Challenges and Opportunities in SBDD

The Structure Based Drug Discovery session began with a talk by **Joe Badger**, DeltaG Technologies and Zenobia Therapeutics. Joe emphasized that a practical approach for analyzing and describing the chemical diversity in the small fragment libraries used for crystallographic screening is needed. Joe designed and assembled an efficient fragment-based screening library, using the PDB and enumerating protein binding motifs. It appears possible to develop a fragment compound set that would allow a well-defined initial screen to be completed within a period of time as short as a single synchrotron shift.

Felix Vajdos, Pfizer, reported their discovery of novel fragment inhibitors of Acetyl CoA Carboxyltransferase using a novel NMR-based functional assay. He and

Session 3.01, cont'd his team have identified a number of bona fide ACC inhibitors, which inhibit the second half-reaction, catalyzed by the carboxyltransferase (CT) domain of the full-length protein. One of these fragments binds cooperatively with a potent CT domain inhibitor of the spiroketone class, suggesting opportunities for lead optimization through hybridization of the two ligands. The crystal structure of this fragment bound to a "humanized" yeast CT domain protein was determined, and the structure suggests opportunities for optimization via hybridization with the well-known ACC inhibitors tepraloxidim and pinoxiden.

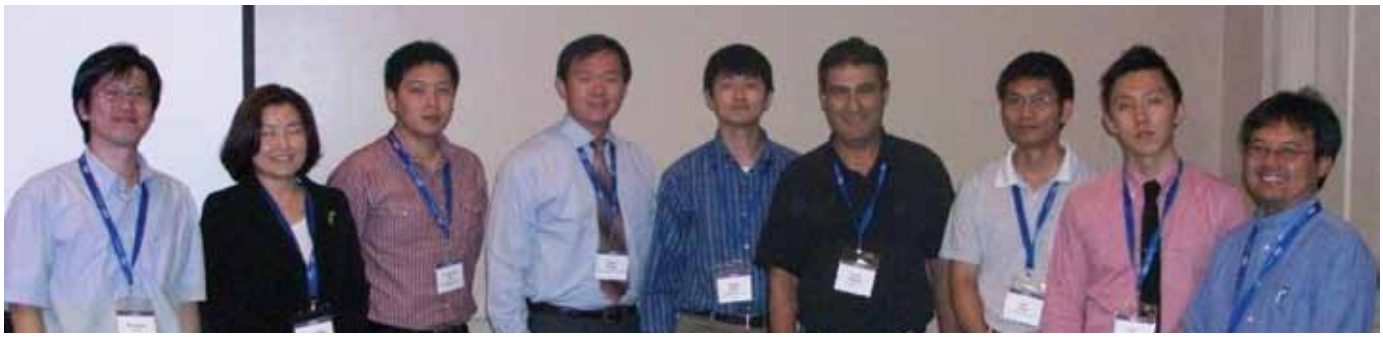
Talapady Bhat, NIST, discussed a rule-based, automated method for managing, querying, and integrating disparate structural information on fragments and also complete inhibitors. The method is called Chemical Block Layered Alignment of Substructure Technique (Chem-BLAST). Using this approach a user can gradually build the fragment and ligand of their choice starting from few 'roots' (highly re-usable fragments are called 'roots' which are defined using atomic connectivity rules and familiar concepts pertaining to fragments).

Steven Sheriff, Bristol-Myers Squibb, presented recent data on HCV RNA-dependent RNA polymerase (NS5B) which is essential for viral replication and makes an attractive target for therapeutic intervention. The structures of NS5B with inhibitors bound to the P495 and primer grip sites were determined. An example of a P495 site inhibitor bound to genotype 1b was also reported; the inhibitor displaces the Δ1 finger tip that normally binds to this site. Moreover, crystals of this complex have served as the basis for studies of primer grip binding compounds. Several examples of a piperazine carboxamide series were presented including one where the structures were determined with both genotype 1b and 1a enzymes. This series binds in the primer grip site, which is at the interface of the palm, fingers, and thumb domains and interacts with the mobile C-terminus of the polymerase. In fact, the benzylsulfonamide moiety of these molecules binds in a hydrophobic pocket occupied by Trp 550 and Phe 551 in the apo structure of the NS5B. Different parts of the C-terminal region interact with the ligand in the 1b and 1a complexes.

Jia Sheng, Georgia State U, spoke about a novel strategy for studying crystal structures of nucleic acid-small molecule complexes. The development of synthetic and enzymatic strategies for the systematic replacement of oxygen atoms with selenium at various positions was discussed. This Selenium Derivatized Nucleic Acids (SeNA) strategy may facilitate crystallization growth and in addition streamline the process of phase determination for DNAs and RNAs. The SeNA approach should provide a powerful tool for determination of DNA and RNA structures and their complexes.

David Cooper, U Virginia, gave a detailed analysis of the interaction between small molecule ligands and proteins, an interaction that plays a crucial role in the structure based drug discovery process. Due to the inconsistent quality of the structural information on the small molecules contained within macromolecular structures in the PDB, the identification, assignment, and validation of these ligands is still a challenging process, even at high resolution. David used HKL-3000 in combination with a sophisticated database to help address these issues.

Nickolay Chirgadze



L to r: Byeongdu Lee, Young-Shin Jun, Chia-Hung Chu, Cheng Wang, Zhang Jiang, David Vaknin, Lin Yang, Peter Gin, Masafumi Fukuto.

Photo by the NO student AV crew.

4.01: Surfaces and Interfaces

Cheng Wang, LBNL, introduced soft x-ray scattering, where x-ray energies close to the absorption edge of constituent atoms (e.g., near the carbon k-edge of 285 eV) are employed to increase the scattering contrast between low-scattering organic materials. Integrating this soft x-ray scattering with surface sensitive techniques such as reflectivity and grazing-incidence x-ray scattering (GISAXS) has proven highly effective in characterizing nano-structures within organic photovoltaic and block copolymer thin films.

David Vaknin, Ames Lab and Iowa State U, discussed recent results on ion-specific bindings to bio-mimetic Langmuir monolayers at the air-water interfaces. Using x-ray reflectivity, grazing-incidence x-ray diffraction, and grazing-incidence x-ray fluorescence spectroscopy, his team discovered that ions of the same valence can show very different binding mechanisms. For example, given identical ion concentrations in the aqueous sub-phase underneath the arachidic-acid monolayer, La^{3+} neutralizes the interface, while Fe^{3+} inverts the charge at the interface. These findings provide insights for improved understanding of ion-specific binding effects and electrostatics in aqueous media.

Lin Yang, BNL, reported a recent study on the self-assembly of tobacco mosaic viruses (TMVs) into ordered 2D superlattices on substrate-supported cationic DOPC/DOTAP lipid monolayers. Their *in-situ*, hard-x-ray GISAXS measurements (16-17 keV) at the buried substrate-solution interface demonstrate that the

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presence of Ca^{2+} promotes alignment and dense packing of the rod-like TMVs. The analysis of GISAXS data reveals that Ca^{2+} ions not only strengthen the screening of the Coulomb repulsion between TMVs but also give rise to counter-ion-mediated inter-TMV attractions. This study illustrated the potential of GISAXS analysis for extracting inter-particle interaction potentials.

Young-Shin Jun, Washington U., presented the study of nucleation and growth of iron oxide nanoparticles at an environmentally relevant mineral-water interface using *in-situ* GISAXS. A combination of SAXS and GISAXS allowed them to monitor in real time and distinguish between homogeneous and heterogeneous, surface-driven nucleation processes that occurred simultaneously under aqueous conditions. This work has implications for the formation and transport of nanoparticles and toxins in both environmental and biological systems.

Peter Gin, SUNY, reported the screening effect of highly compressible supercritical carbon dioxide on attractive polymer/substrate interactions.

Byeongdu Lee, ANL, showed the *in-situ* GISAXS characterization of the property changes of the reactive nano-particles and atomic clusters on flat catalytic support substrates under high pressure and high temperature.

Chia-Hung Chu, NSRRC in Taiwan, reported using x-ray Bragg-surface diffraction to measure the 3D strain-field as a function of detection penetration depth in the silicon substrate near the iron silicides film interface.

Zhang Jiang and Masafumi Fukuto

L to r: Richard Kirian, Greg Hura, Michal Hammel, Philip Anfinrud, Thomas Grant, Lee Makowski, Sichun Yang.
Photo by the NO student AV crew.

4.03: Information in SAXS/WAXS Data

This session reviewed not only what is possible now, but what will be possible in the SAXS/WAXS field in the near future.

Sichun Wang, Case Western, presented *Structural determinants of multidomain complexes: integrating SAXS and biophysical computations*, in which he noted that 86% of the "protein universe" is made up of multi-domain proteins. Sichun described his method for combining molecular dynamics (MD) and SAXS for characterization of protein ensembles. Coarse-grained (CG) MD is required for these very large complexes, and Sichun has developed a method for rapidly calculating SAXS patterns from CG models in order to test the results of CGMD. His work involved analysis of the linkage of ensemble and function in tyrosine kinases.

Michal Hammel, LBNL, discussed *Robust, High-throughput (HT) Solution Structural Analyses by Small Angle X-ray S cattering*. Using a combination of SAXS, computational methods and crystallography, Michal and his collaborators at the SIBYLS beam line at the ALS are pushing SAXS analysis of the size and shape of macromolecules to a new level of throughput. As an example of the approach he outlined their recent work on proteins involved in double-strand DNA break repair in humans. Combining SAXS data with information from other sources including crystallography, Michal generated an image of the complex at sufficient detail that individual amino acids involved in intermolecular interactions could be identified.

Phil Anfinrud, NIH, described his tour de force time resolved WAXS (TR-WAXS) studies of myoglobin and a dimeric hemoglobin, carried out in collaboration with Friedrich Schotte, Hyun Sun Cho, Narangaatar Dashdorj, William Royer, Robert Henning and Tim Graber. Acquiring nanosecond time resolution on the dissociation of CO from myoglobin, Phil was able to demonstrate transient changes in molecular volume as small as $22Å^3$. He outlined in detail the technological innovations both at BioCARS at the APS and at ESRF that were needed for the work to succeed. It seems inevitable that TR-WAXS will be a major highway in the future of x-ray solution scattering. As the community moves down this path, it will recognize this ground-breaking work as paving the way.

Richard Kirian, AZ State U, introduced us to "Snapshot SAXS for *ab initio* imaging from spatial correlations". ***Editor's note: see the figure in TR.01, page 23.*** Richard, working with Kevin Schmidt, Dilano Saldin and John Spence, is developing a method first introduced by Kam in 1977. In this method, x-ray data is collected from protein solutions with an exposure time so short that the proteins do not rotate in solution while being exposed. Scattering from a sample exposed under these conditions contains angular correlations modulated by inter-particle interference effects. These two contributions render the pattern non-circularly symmetric. Summing up large numbers of these patterns preserves the angular correlations while averaging out the interparticle effects. With an eye to use of the LCLS for this kind of work, Richard is involved in overcoming the substantial obstacles currently impeding the practical implementation of this method. The possibilities for deriving three-dimensional structural information about important molecular complexes is both intriguing and exciting.

Cont'd on next page

4.03: SAXS/WAXS, cont'd The SAXS SIG chose **Thomas Grant**, Hauptman-Woodward Inst., to receive their **2011 Etter Young Lecturer Award**. Thomas' lecture described work being carried out with Joseph Luft, Jennifer Wolfley, Hiro Tsuruta, Gaetano Montelione, and Edward Snell. His paper, *Small Angle X-ray Scattering as a Complementary Tool for High-throughput Structural Studies* was based on the premise that the amount of information that can be extracted from SAXS patterns is greatly enhanced when used in coordination with other biophysical experimental and computational approaches. In spite of the title, his presentation was more reminiscent of Dmitri Svergun's assertion that crystallography is a tool that complements SAXS (rather than the other way around). Tom started his talk with a memorable demonstration of the dangers of overinterpreting SAXS data. He showed the audience a reasonable looking molecular envelope calculated from "SAXS data" that turned out to be the daily closing values of the Dow Jones' Industrial average during the recession. Using proteins from the Northeast Structural Genomics Center, Thomas carried out HT analysis of more than 150 protein samples. Data was completely collected during a 3 day run, using 8 one-second exposures on static samples. 77% of the samples yielded useful data. Twenty-eight corresponded to proteins for which a high resolution structure was available. Comparisons of the calculated envelopes with crystal structures demonstrated that the oligomeric state inferred from crystallography was not always correct; and that mixtures of oligomeric states were occasionally observed. Thomas made a strong case for SAXS as a tool to be used in parallel with crystallography and NMR for analysis of macromolecular structures.

The final talk of the session was given by **Greg Hura**, LBNL, who described work carried out in association with Shelly Claridge, Marc Mendillo, Christopher Putnam, Richard Kolodner, A.Paul Alivisatos, and John Tainer. Greg outlined a remarkable set of experiments analyzing protein function in the repair of mismatched DNA base pairs. SAXS from DNA double strands labeled on each end with gold nanoparticles gave rise to patterns from which the separation of gold nanoparticles could be readily derived. In order to map out the behavior of the DNA and DNA repair enzymes under many conditions, a HT approach was required. The collective results establish the potential of SAXS with gold labels to explore critical DNA distortions common to the repair, replication, transcription, and packaging of DNA.

Lee Makowski

L to r: Audry Kovalevsky, Saul Lapidus, Ilia Guzei, Ian Wilson, Peter Davies, Allen Oliver, Alberto Albinati, Peter Müller, Patrick Carroll, Nathan Schley.

5.01: Cool Structures

The Cool Structures session was so popular that the talks spilled over into "More Cool Structures" on the last day. One of the first-time student attendees, **Nathan Schley**, Yale, talked about catalysts used in the conversion of water to di-hydrogen and di-oxygen; a clearly important field in today's climate. Nathan located and described short H-bond interactions that are believed to be causal in the degradation of water. **Peter Müller**, MIT, followed with a talk about a challenging and difficult problem structure. This was especially of interest for new crystallographers as he showed in detail how to handle such problems. **Patrick Carroll**, U PA, continued this instructional theme with his description of a difficult, twinned, high Z, borane compound.

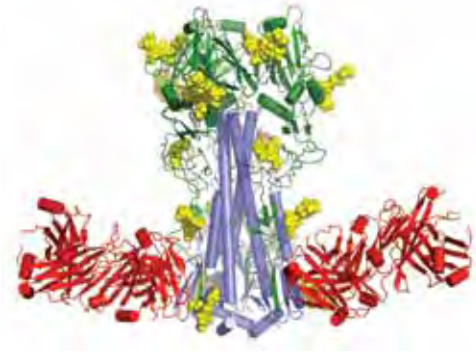
Alberto Albinati, U Milan, gave an overview of his continued research into Ru-H complexes, particularly multiply coordinated hydrides and the variation in their coordination geometries. **Yulia Sevryugina**, Int'l Inst. of Nano & Molecular Medicine, see right, discussed the challenges she faced with a series of iodo-boranes; again an informative talk about how one can approach these problems.

Ilia Guzei, U Wisconsin, reported the isolation and characterization of three polymorphs demonstrating the elegant technique he applied via heating and cooling of the polymorphs to anneal them into single crystals. The time-lapse movie he showed was one of the highlights of the session.

Saul Lapidus, Stoneybrook U, the second first-time student speaker in the session, delved into the complicated mathematics involved in analyzing powder diffraction data, even for relatively straightforward systems. The complimentary methods of neutron diffraction and protein x-ray diffraction were discussed by **Andrey Kovlevsky**, LANL. He described a program that combines data from both methods in order to place water hydrogens in protein structures. **Peter Davies**, Queen's U, gave an exciting talk on the effects imparted by anti-freeze proteins located on the surface of ice crystals and how they disrupt the ordering and aggregation of water molecules on the surface of the ice.

Bruce Noll, Bruker-AXS, led the spillover session with a talk discussing synchrotron related quirks. **Zachary Wood**, U Georgia, see left, followed with a talk on the packing defects found in the structure of glucose dehydrogenase. ***cont'd on next page***

Ian Wilson, Scripps, presented research targeting features of the influenza virus towards the development of a new vaccine to fight influenza. The zone of interest is the "stem" of the virus, a location that had not been previously targeted by researchers, see figure at left. This looks to be a promising approach to en endemic problem.



From Ian Wilson: an image generated by Damian Ekiert who did the structural work. Antibody 8020 binds to a conserved site in the hemagglutinin stem and broadly neutralizes group 2 influenza viruses. The hemagglutinin trimer is in green (HA1) and blue (HA2) with carbohydrates in yellow and the antibody Fab in red. This work appeared in Science online (Ekiert et al.) on July 7. Science DOI: 10.1126/science.1204839

Solid state studies involving new superconducting materials were reported by **Peter Zavalij**, U Maryland. In particular Peter discussed how to handle the disorder and twinning these high symmetry compounds often adopt. **Jesse Rowsell**, Oberlin College, gave an overview of clathrate complexes. His talk was focused on structures with Z' much greater than usual, including one structure with a record 56 unique molecules. **Marilyn Olmstead**, UC-Davis, presented the continuation of her studies into larger fullerene compounds, including a 92 carbon fullerene, one of the largest reported to date. Finally, **Marcus Bond**, SE Missouri State U, reported a unique "cross-hatched" layer structure that defies conventional rationalization and that seems to act as an egg-tray template for inversion related organic cation pairs.

Allen Oliver

L to r: Mary Parker, Bernhard Rupp, Eric Reinheimer, Marvin Hackert, Stephanie Cowin. Photo courtesy of Marvin Hackert.

6.02: Would You Publish This?

What do inorganic coordination compounds, rotaxanes, tetraazatetrapyridopentacene, and buckyballs have in common? --They give lousy crystal structures! Thus went the third annual ***Would You Publish This?*** session sponsored by the Service Crystallography SIG.

Allen Oliver, Notre Dame, started by describing a series of $[Cu(phen)_2^{2+}]n$ (n=2,3) complexes that routinely exhibit anion and solvent disorder and tend to crystallize with Z' > 1. A related $[Cu(bipy)_2^{2+}]n$ complex was even more perplexing as it formed a polymeric structure with oddly shaped displacement ellipsoids suggesting disorder. In all cases, metrical parameters and refinement statistics were sub-optimal. **Amy Sarjeant**, Northwestern, showed a series of oligorotaxane structures that should show discrete entities, but instead give the same structure regardless of host oligomer size -- a polymeric rotaxane. The synthetic procedure and mass spec data eliminate the possibility of a polymeric host. Occupancies of the host oligomer were fixed in order to model each "polymer" as the correct oligomer structure.

6.01: Undergraduate Research Symposium

The selection committee for this year's **Undergraduate Research Symposium** was pleased to give awards to **Mary Elizabeth Parker**, U Tennessee, for her research on *Single Crystal Growth, Crystallography and Magnetic Properties of Maus' Salt*, and to **Stephanie Cowin**, U Missouri-St. Louis, for *Steric Effects on the Synthesis of Metal Halide Layers and Clusters*. These were exceptional talks and during questions and answers afterwards both students demonstrated their ability to think critically and to communicate their thoughts. Each student received a $200 award that included membership in the Society for Physics Students, a subsidiary of the American Physical Society. Congratulations to Mary and Stephanie for outstanding undergraduate research in crystallographic science!

Eric Reinheimer

Muhammed Yousufuddin, U Texas-Arlington, described a dimeric tetraazatetrapyridopentacene structure that exhibits pseudomerohedral twinning and solvent disorder. The refinement statistics are terrible, but the structure of the "region of interest" is unambiguous.

Khalil Abboud, U Florida, showed a well behaved cationic Mn12 cluster with composition confirmed by mass spectroscopy and Mn formal charges assigned by BVSA. Pyridine/pyridinium ambiguity in cluster ligands combined with anion disorder made it impossible to determine the overall charge of the cluster, however. **Louise Dawe**, Memorial U of Newfoundland, described two C60 * corannulene structures. The host corannulenes are ordered, but the C60 and solvent are not. The crystal also had a minor non-merohedral twin. Inclusion of the weak twin data gave a less sensible refinement model compared to ignoring the twin component. Larry Falvello, U Zaragosa, presented very poor structures of two Co-clusters. The structures were not of sufficient quality to publish independently, but were of sufficient quality to publish as evidence that the material underwent reversible, temperature-dependent hydration/dehydration.

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Although the type of structures differed significantly and there was no general consensus among the audience about whether or not the individual structures should be published, there were common threads that all chemists and crystallographers should consider when publishing structures: (1) be reminded that crystallography is one of many analytical techniques and complimentary analytical techniques should always be used to verify thatthe structure is correct. (2) If twinning or Z' > 1 is observed, try a different temperature! The sample might have undergone a phase change. (3) It is OK to publish moderate and poor quality structures as long as (a) there is full disclosure of problems, uncertainties, and refinement tricks used, and (b) the authors do not over-interpret the results.

Carla Slebodnick

L to r: Bruce Foxman, Mario Bieringer, Matthew Suchomel, Craig Bridges, Antonio dos Santos, Gregory Halder, John Parise, Catherine Renouf, Christine Beavers. Photo by NO student AV crew.

8.01: In situ Diffraction Studies

Mario Bieringer, U. Manitoba, described how he used crystallography as a tool to follow chemical reactions. Transition metal oxides were heated to 1300° C and monitored for phase changes. In the case of $InVO_3$, a metastable form was discovered. The oxidation of $ScTiO_3$ bixbyite was also examined *in-situ*, yielding two new cubic phases, $ScTiO_{3.5}$, and $Sc_4Ti_3O_{12}$ with expulsion of TiO_2.

Antonio dos Santos, ORNL, illustrated the importance of transition metals in the earth's core with respect to the magnetosphere, and crust phenomena such as earthquakes. Cobalt oxide is an interesting example because it has a controversial magnetic structure. Using the SNAP diffractometer at SNS, the magnetic and crystallographic structures of CoO were determined at pressures up to 10GPa, and it was determined that the magnetic transition was coupled to the structural change.

Matthew Suchomel, Argonne, illustrated the developments related to *in-situ* diffraction at the APS. There are now two beamlines at the APS dedicated to powder diffraction, and both now have *in-situ* capability. At 11-BM, sample heating and cooling apparati are currently available. A diamond anvil cell is currently being commissioned and reaction and gas flow cells are also available for use.

John Parise, Stony Brook U, reported his research on fluids, melts and liquids, using pair-distribution functions (PDFs). As a refresher, he reminded us that all liquids are fluids, but not all fluids are liquid. His experimental design to study the melts was ingenious; a ball of melt, heated by a laser, was levitated on a steam of gas, so that there were no container-melt interfaces. This set-up was used to investigate the structure of $CaSiO_3$ while cooling from 2000° to 500°C.

Bruce Foxman, Brandeis U, gently chided the crystallographic community for being too lax concerning crystal-to-crystal transitions. The accurate comparison of before and after with respect to crystallographic and diffractometer coordinates is lacking in many reports, and Bruce would like that to change. He demonstrated the ease with which one could determine the axial relationships between mother and daughter products, using his TOPOTAXY program.

Catherine Renouf, U St. Andrews, explained how metal-organic frameworks (MOFs) could be used in drug delivery. The framework M-CPO-27 was desolvated and loaded with NO gas *in-situ* at beamline 11.3.1 at the ALS. This MOF-NO complex has great potential for NO delivery in medical applications; determining the gas-loaded structure will aid in the development of other useful compounds.

Gregory Halder, Argonne, spoke about his studies of spin crossover transitions in MOFs at the 1-BM beamline at APS. Using variable temperature and gas flow cells, one MOF was determined to undergo a desolvation through a phase change; the spin-crossover transitions were observed as well. Another spin-crossover transition was triggered at higher than ambient pressures using a DAC, (digital-to-analog converter). At the end of Greg's talk, the room was still full; such a tribute to a wonderful group of speakers.

Christine Beavers & Craig Bridges

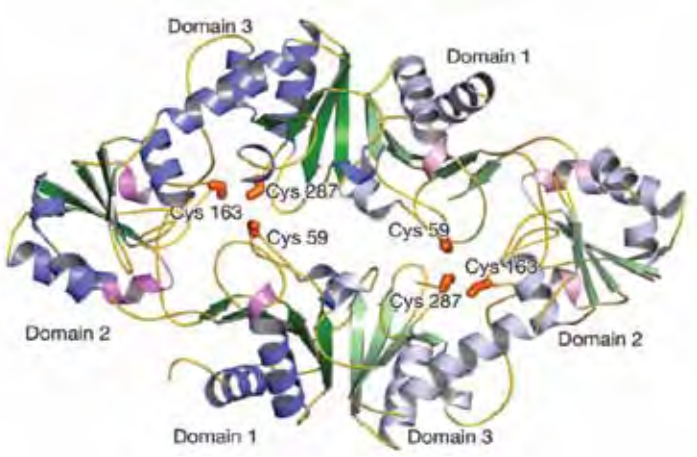
Marcia Colquhoun and Crystal Towns

8.02: Structural Enzymology I, Spectroscopy & Complementary Methods

L to r: Narayanasami Sukumar, Allen Orville, Andrew Torelli, Karen Allen, Ritimukta Sarangi, Michael Murphy, Erik Yuki. Photo by the NO student AV crew.

Karen Allen, Boston U, described new work on stachydrine demethylase from *Sinorhizobium meliloti* 1021. This multicomponent enzyme system catalyzes the NAD(P)H and O_2 dependent demethylation of N,N-dimethylproline (stachydrine) to form N-methyl proline and formaldehyde. The crystal structure of the oxygenase component (Stc2) in the unliganded holo enzyme state was reported. The structure reveals the prototypical two-domain structure shared by other Rieske non-heme iron oxygenases first described by naphthalene 1,2-dioxygenase. Surprisingly, the electron density for the aerobic enzyme substrate complex is most consistent with proline bound to the mononuclear iron site. They used beamline X26-C of the NSLS to characterize the Rieske iron-sulfur cluster by UV-visible spectroscopy throughout x-ray data collection in conjunction with resonance Raman spectra collected before and after diffraction data. Thus, they were able to show that oxidized crystals are reduced by solvated electrons generated by the x-ray beam at 100°K. Moreover, the data shows that the kinetics of the reduction process differed dramatically for the substrate complex compared to that of the unliganded enzyme. A reaction mechanism was proposed for the in-crystallo chemistry promoted by x-ray photons.

Andrew Torelli, Cornell, presented a very complex system with clarity, scientific rigor and broad context. His focus was on diphthamide, a hypermodified histidine residue that is absolutely conserved in translation elongation factor 2 (EF2) from eukaryotes and archaebacteria. Its biosynthesis occurs in several steps, but the first step is the most complex reaction and involves the transfer of a 3-amino-3-carboxypropyl (ACP) group from a molecule of S-adenosylmethionine (SAM) to the target histidine in EF2. Andy and colleagues studied the enzyme, PhDph2, from *Pyrococcus horikoshii* and they reported a structure of PhDph2 co-crystallized with products of the SAM-cleavage reaction. The [4Fe-4S] cluster in PhDph2 is used to generate a novel ACP radical from a co-substrate SAM molecule. This reactive species then forms a C-C bond with the weakly-nucleophilic C2 atom of the target histidine, thereby completing the first step of diphthamide biosynthesis. All other proteins that use a [4Fe-4S] cluster to generate a SAM-derived radical are members of the radical SAM superfamily; however these enzymes strictly form 5'-deoxy-5'-adenosyl radicals. The results provided insight into how orientation of the SAM molecule relative to the [4Fe-4S] cluster dictates which bond is cleaved to form radical species.



From Andrew Torelli: Ribbon diagram of the PhDph2 homodimer with one monomer in dark colors and the other in light colors. Each monomer is also colored according to secondary structure, with dark and light green for β-strands, dark and light blue for α-helices and violet and pink for 3_{10} helices. The three conserved cysteine residues for each monomer are shown in the stick representation, in orange. Y. Zhang et al, (2010) ©Nature 465, 891–896.

Ritimukta Sarangi, SSRL, highlighted results of combined single-crystal x-ray absorption spectroscopy (XAS) and diffraction obtained at SSRL beamline 9-3. Results from two recent studies were reported. In the first study, single crystal XAS studies on the Ni-containing active site of methyl coenzyme M reductase was combined with solution XAS and EXAFS data. The data were used to determine the redox state of a putative Ni(III)-methyl intermediate and coupled to structure determination. In the second study, the electronic structure of oxyhemoglobin was explored using both solution and single crystal Fe K-pre-edge and near-edge XAS studies to differentiate between two putative electronic structure descriptions. These data provide important electronic structure information on unstable, trapped intermediate and transient species. Results of this nature can be used to guide the structure determination process, especially to develop the best strategy for diffraction data collection of unstable intermediates.

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Michael Murphy, U British Columbia, gave a talk entitled, *Insights into the Mechanism of Heme Degradation by the IsdG-Like Family of Enzymes*. This family of enzymes catalyzes the reductive degradation of hemin, releasing iron for cellular processes. The group focused on IsdG and IsdI, components of a heme-iron utilization and acquisition system from the pathogen *Staphylococcus aureus*. Crystal structures of IsdG and IsdI reconstituted with hemins and the inhibitor Co-protoporphyrin IX were reported. These 1.5 – 1.9 Å resolution structures reveal that the porphyrin ring is highly ruffled such that it has the greatest distortion from planarity of any protein bound heme group. Preliminary data from single-crystal spectroscopy at NSLS beamline X26-C suggested that the crystals are rapidly reduced in the x-ray beam during data collection. In the IsdI-heme complex that was photoreduced in the x-ray beam they showed electron density for a sixth iron ligand and modeled it as a dioxygen species. The heme ruffling places the β– and δ–meso carbons in close proximity to the bound oxygen species consistent with cleavage of the ring at these carbons.

Erik Yuki, U MN, discussed very recently published results on MauG, a c-type di-heme enzyme responsible for the post-translational formation of the catalytic tryptophan tryptophylquinone (TTQ) cofactor in methylamine dehydrogenase (MADH). An overall aim of the group is to characterize the catalytic intermediate in this reaction, which has the spectroscopic signature of an unprecedented bis-Fe(IV) species. One of these is described as Fe(IV)=O while the other is Fe(IV) coordinated by Tyr and His amino acids. The intermediate is produced by reaction of di-ferric MauG with either hydrogen peroxide or oxygen plus reducing equivalents. CO and NO were used as oxygen surrogates in structures of the diferrous, Fe(II)-CO and Fe(II)-NO forms of MauG in complex with its preMADH substrate. The structures suggest likely roles in oxygen binding and activation for certain amino acid residues in the distal pocket of the high-spin heme.

Narayanasami Sukumar, Cornell, spoke about the role of amicyanin, an electron transfer protein from *Paracoccus denitrificans*, in the electron transfer between the methylamine dehydrogenase and cytochrome c-551i. The copper site of amicyanin consists of three equatorial ligands (two histidines and a cysteine) and a weak axial ligand (methionine). Accordingly, it is a member of the "blue" or "type-I" copper proteins and exhibits intense electronic absorption bands in the visible spectrum (λmax = 600nm and 450nm). Narayanasami outlined several studies of isoforms with alterations to the methionine 98 residue that serves as the axial Cu ligand. In the M98Q amicyanin, the O atom of Gln provided a distorted axial copper ligand that correlated with an increase in reorganization energy for the ET from MADH. In the M98A isoform a solvent molecule occupies the axial ligand site and yields similar redox and ET properties to that of native amicyanin. He concluded with recent crystallographic and biochemical results on the M98L and M98K isoforms.

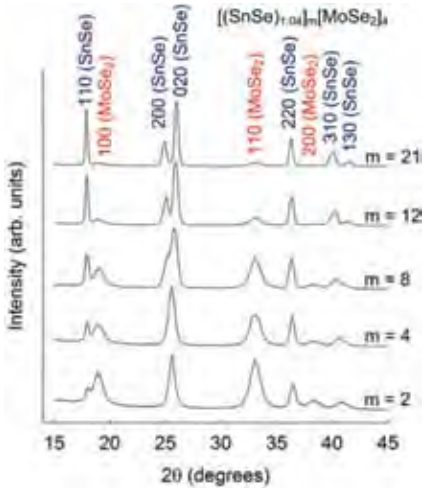
Allen Orville

L to r: Daniel Fredrickson, Matt Beekman, Robin Macaluso, Michael Lufaso, Bradford Fuller, Daniel Bugaris, Tiffany Kinnibrugh, Svilen Bobev, Peter Khalifah. Photo by the NO student AV crew.

8.03: Crystallography and the Search for New Materials

This symposium was sponsored by Agilent Technologies, Bruker, and Rigaku in addition to the ACA. Introductory remarks by **Svilen Bobev**, U Delaware, briefly recapped the importance of different crystallographic techniques used to characterize newly synthesized solids.

Next, **Robin Macaluso**, U Northern Colorado, talked about the crystal growth of new Ln-Pd-Ga and Ln-Pt-Ga intermetallic compounds (Ln = lanthanide metal) at the border between the Hume-Rothery and the Zintl phases. Robin showed how symmetry of the structure changes when experimental conditions are subtly varied: an effect concomitant with partial order of the transition metal and gallium atoms.



From Matt Beekman: In-plane synchrotron x-ray diffraction showing (hk0) reflections from the SnSe and $MoSe_2$ components of the intergrowth structures $[(SnSe)_{1.04}]_m(MoSe_2)_4$ for different values of 'm'.

Matt Beekman, U Oregon, introduced a new class of layered materials based on intergrowth (MX)a(TX2)b structures. These materials do not fit the established rules for being called crystals, yet they are not amorphous solids. Matt showed results from x-ray diffraction and x-ray reflectivity experiments, confirming strong Bragg peaks from thin films of $[(PbSe)_{0.99}]_1(WSe_2)_1$ and related chalcogenides, which he dubbed "ferecrystals", i.e., almost crystals.

Daniel Bugaris, USC, described the synthesis and the structural characterization by single-crystal x-ray diffraction of a series of hydroxides with the general formula $A_2B(OH)_6$. They were prepared by a "hydroflux" method which resembles traditional hydrothermal and flux-growth techniques. Surprisingly, the hydroxides prepared by Bugaris were found to be moisture sensitive, despite the fact that the crystals were formed in aqueous media.

Daniel Fredrickson, U WI, reported that Hückell or DFT theory can complement the crystallographic characterization of intricate intermetallic structures. Dan went on to discuss the interpenetration of polar and non-polar domains in $NaCd_2$, the competition of steric and electrostatic effects in CoSn-type structures and the importance of "chemical pressure" for the realization of $SrAg_5$ and Ca_2Ag_7 structures.

Michael Lufaso, U North FL, presented the preparation and the structure elucidation of solid solutions of the type $Bi_2(Fe_{1-x}Mn_x)_4O_9$, which could be useful gas-sensing materials. He discussed different (often conflicting) results from the various synthetic routes used previously by other groups, and showed results from exhaustive characterization of the Mn-Fe solubility limits by single-crystal and powder x-ray diffraction, and neutron powder diffraction.

Tiffany Kinnibrugh, Texas A&M, described the synthesis of new aluminum monophenylene diphosphonates. Her structures were solved and refined from high-resolution synchrotron data collected at beam-line 11-BM at ANL. Tiffany also presented evidence for the reversible hydration/dehydration of the structure, collected by means of DSC/TGA and subsequent x-ray diffraction experiments.

Bradford Fulfer, LSU, described the synthesis of new intermetallic aluminides, in which an interplay of $ThMn_{12}$ and $CaCr_2Al_{10}$ type structures exist. He discussed the structural disorder due to Mn-Al mixing and other intricacies of the structures, which were resolved by single-crystal x-ray diffraction.

Peter Khalifah, Stony Brook U and BNL, focused on materials for photocatalysis and battery applications. Peter detailed results from conventional crystallography, TEM tomography, solid-state NMR, and PDF analysis based on neutron powder diffraction data in thin films of the type $(GaN)_{1-x}(ZnO)_x$ and $LaTiO_2N$. Centimeter-sized single crystals of the Li-battery material $LiFePO_4$ were grown and the structure was solved based on data gathered on the TOPAZ instrument at SNS-ORNL.

Svilen Bobev

L to r: Mike Hoyland, Jerry Jasinski, Arnie Rheingold, Judy Flippen-Anderson, Bruce Noll, Larry Falvello, Ilia Guzei, Robin Rogers, Richard Cooper. Photo courtesy of Ilia Guzei.

8.04. Scholarly and Pragmatic Aspects of Crystallographic Publication Practices

This session was sponsored by the Small Molecule, Service, and General Interest SIGs, ACA, Agilent Technologies, Bruker, and the IUCr. **Mike Hoyland**, who is actively involved in data digitization at the IUCr, described the historical background of the CIF format -- the conceptual successor to the "standard crystallographic file structure" pioneered in 1984 -- which has evolved into an open access publication method in *Acta Cryst. Section E*. The use of checkCIF is necessary due to the sheer number of manuscript submissions to the IUCr journals; however, the alerts should be viewed as useful checkpoints rather than as a brick-wall-like barrier to publication. Whereas there are limitations to the validation algorithms, the latter offer tremendous help to authors, reviewers, and co-editors (think PLATON, checkCIF, publCIF). Future improvements to the existing structural tests and the implementation of new ones are expected on a continuous basis, and user feedback is encouraged.

Jerry Jasinski, Keene State College, who has published 33 papers to date this year, described the rigorous *Acta Cryst. C/E* manuscript preparation workflow followed by his undergraduate students. An important aspect of the process is to teach the students to avoid padding papers with "fluff" in order to expand their contents. If all authors submitting manuscripts to *Acta Cryst. C* and *E* followed the *Notes for Authors* as closely as is required of Jerry's students, the coeditors would have more time to spend on the structural science and chemical ramifications of the structures.

Richard Cooper, Oxford U, cast a strong vote in favor of the invaluable checkCIF. Richard, curator of the crystallographic software package CRYSTALS, emphasized that CRYSTALS is a teaching tool and that it is imperative to train and educate crystallographers to evaluate the results objectively and to interpret structural peculiarities correctly. With a few reasonable assumptions one may expect over 95% of the structures with more than 1 level A/B alert can still be correct. In the end, the data must support the structure, and checkCIF alerts should be understood and corrected or explained, rather than viewed as obstacles on the way to publishing a structure. As did other speakers, Richard gave a frank appraisal of some of the pros and cons of CIF definitions and checkCIF tests, including the relative frequencies of valid and "false positive" alerts. On occasion checkCIF definitions may appear oversimplified or overcomplicated, in which cases alert interpretation is not straightforward. An interesting question "Do different structures require different levels of validation?" certainly provided food for thought.

Bruce Noll, Bruker, confidently demonstrated the facile use of Bruker APEX2 software for preparation of crystallographic data for publication. Among other features, APEX2 can populate most CIF data fields with correct experimental data, run a quick and convenient data validation, generate a checkCIF report, and create an HTML-based structural report with an interactive JMOL molecular graphics window. In the case of well-behaved structures the CIF file created by APEX2 is suitable for deposition to the CSD and is essentially ready for submission to *Acta Cryst. E*.

It was a rare pleasure to hear **Judy Flippen-Anderson**, *ACA RefleXions*, who delivered an evolutionary tale *From Independence to Service and Science to Technology* based on her experiences as a crystallographer, database specialist, and co-editor of *Acta Cryst. E*. In the (g)olden pre-PC days it was not unusual to see two papers per structure - one crystallographic and one chemical. Later one chemistry paper combined both aspects, and now the structural details are frequently relegated to the supporting information. *Acta Cryst. E* papers have become glorified database entries and she felt that the quality of the papers in this journal has recently been slipping. Judy suggested ways to improve the quality of papers might include requiring one author to have an entry in the *IUCr World Directory of Crystallographers* and restoring a short paper format with a mandatory *Comment* section, while maintaining the open access charge structure. Judy also showed interesting structural database deposition statistics - the slope of the curve showing the total number of structures deposited to the PDB each year tracks the trend observed for the CSD with an approximate 20 year offset.

Robin Rogers, U Alabama. discussed the influence of culture on publication practices. For example, whereas copying masterpieces is a common undertaking in the world of art when students learn to draw, plagiarism is unacceptable in science. Unabashed copying of someone else's results is inexcusable; however, a different interpretation of the results may be welcomed. In chemical crystallography there is a distinction between papers that report science and papers that report technique. If the technique is important its quality should approach the best attainable; if chemistry is the focal point then imperfections in the technique are forgivable. One should remain open to a diversity of ways of interpreting results in the appropriate context.

Arnie Rheingold, UC San Diego, who last attended an ACA meeting in 1993, spoke about finding standards for chemically important structures. When crystallography plays a supporting role there are caveats to interpretation of the results -- one must be objective and chemically knowledgeable. For example, symmetry may average six Cu-O distances in an octahedral Cu complex residing on a 3bar axis and "eliminate" the mandatory Jahn-Teller distortion. Clearly, in this case one must interpret the disorder rather than report a novel and unique Cu complex without a Jahn-Teller effect. The most frequent source of complaints for a crystallography editor is the presence of thermally mobile and poorly modeled solvent molecules. If a solvent can be modeled and refined, then this is the avenue to follow; but after reasonable attempts fail to accomplish this task, PLATON/SQUEEZE is a respectable alternative. Importantly, the use of SQUEEZE, as well as the use of any constraints and restraints, should be properly documented. Finally, the co-authorship guidelines in the *Acta Cryst*. journals should follow the ACS standards -- all people who have contributed to the results should be listed as authors.

An open discussion, in which everyone present was invited to voice an opinion on the broad topic of publication practices followed. It was reiterated that authors should always give proper credit to all cited work even if the information is freely available from electronic sources. Preferably individual structures from the CSD should be cited instead of a blanket CSD reference. **Peter Stephens** pointed out that structure validation gives a significant number of spurious warnings for powder diffraction analyses, including A-level alerts that are not applicable to powder studies such as the absence of anisotropic U(ij) values. **Mike Hoyland** commented that more work is needed on structure validation for powder data. Another question addressed the alert generated by molecular fragments lying outside the basic unit cell. This stimulated a discussion about the optimum choice of asymmetric unit. In response to a comment by **Radu Custelcean**, **Ian Bruno** raised the question of whether it would be useful if each entry in the Cambridge Database had its own DOI. **Judy Flippen-Anderson** commented that entries in the Protein Data Bank already have their own individual DOI.

Ilia Guzei and Larry Falvello

L to R: Bill Weis, Qun Liu, Graeme Winter, Dominika Borek, Alex Soares, Rita Giordano. ***Photo by the NO student AV crew.***

8.06: Microcrystals and Back to Merging Datasets

Zbyszek Otwinowski, UT Southwestern Medical Center, discussed the various types of non-isomorphism that appear in multi-crystal and micro-beam experiments, methods for their identification, and their influence on diffraction data processing and phasing procedures. He proposed that even in cases where the crystal is microscopically non-uniform, it may be better to use the traditional approach, in which the beam illuminates the whole volume of the crystal. This approach optimizes the signal-to-noise ratio and averages non-isomorphism within the crystal caused by cryo-cooling. This approach can result in better estimates of phasing differences and permits the use of a lower dose per oscillation to partially mitigate the decay and non-isomorphism induced by x-ray radiation.

Graeme Winter, Diamond Light Source, described how his software, Xia2, which is implemented at DLS beamlines, handles multi-crystal experiments. The polyhedra virus crystals were used to exemplify handling the reduction of multiple data sets. An algorithm classifies crystals into isomorphous groups based on the correlation coefficient between the diffraction intensities of different crystals. In the case of weakly diffracting crystals, noise affects the calculations of the correlation coefficients for single reflections which causes problems for clustering. In such cases, selection criteria based on R-merge values calculated between groups of diffraction images from different crystals are more effective. Additional improvement is achieved when R-merge values for each diffraction image are included in calculations because this allows modeling contributions from decay during diffraction. Experiments have shown that despite all the progress in this area there is still room for improvement because the procedure is still very sensitive to changes in intensities of reflections, which presumably arise from radiation-induced effects.

Qun Liu, BNL and NY Structural Biology Center, reported that some weak anomalous signals from multiple crystals are measured more accurately than those from a single crystal, supporting structural analysis when the single crystal route fails. Qun presented protein structures in which phasing from a weak anomalous signal was only achievable by merging data sets from multiple crystals. The case a 500 amino acid membrane protein transporter was particularly interesting; it was phased from sulfur (S-SAD) by merging data from seven different crystals.

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Bill Weis, Stanford, explained how complete data sets for the β2-adrenergic receptor were acquired from multiple crystals and the challenges in that data analysis. Using micro-beams at the ESRF and at the GM/CAT beamline at the APS proved necessary to minimize background scattering that, due to very small sized crystals, would dominate the measurement noise. also, the crystals grew in the lipid cubic phase and often multiple crystals were retrieved and cryo-cooled together. The micro-beam was critical here because one could selectively illuminate a single crystal. The combination of a highly focused micro-beam with the small size of the crystals led to rapid decay of the diffraction data such that only a few images collected from a single, micro-beam-illuminated spot on the crystal. Merging the data, from many narrow slices of reciprocal space, turned out to be very challenging. Bill suggested a list of improvements to the existing software - in particular, stable procedures for partial refinement of intensities integrated from multiple crystals are needed.

Rita Giordano, ESRF, Grenoble, talked about non-isomorphic frozen crystals. She discussed a clustering method based on the correlation coefficient between scaled intensities. The analysis identified factors ranging from the timing of cryo-protection to the age of the crystals that contributed to non-isomorphism between crystals, and showed that merging diffraction data sets within the same cluster leads to better estimation of the anomalous signal and consequently improved phasing. The cluster method can be used to select isomorphous crystals' sub-groups for merging analysis.

Finally, **Alexei Soares**, BNL, showed how sound waves can be used to eject crystals from the crystallization drop and to position them on the grids used for diffraction data collection. A system produced by Labcyte and used in an ECHO liquid handler was adapted for that purpose. Since it can handle any density of liquids used for crystallization, the method is applicable to a broad range of conditions. The controlled speed of ejection and the reproducibility of the process are superior to manual crystal harvesting.

Dominika Borek and Alex Soares

8.07: Materials for a Sustainable Future

Tim Anderson, U. Florida, began by presenting the research of his group on thin film voltaics and specifically how they are studying the phase equilibria of various systems making up binaries, and ternaries of quaternaries of interest. Tim also highlighted the use of High Temperature X-ray Diffraction (HTXRD) to study the kinetics of Cu(In,Ga)Se_2 (CIGS). **Melanie Kirkham**, ORNL, presented her current work on systems that demonstrate thermoelectric behavior, using HTXRD to study phase transitions. X-ray powder diffraction data were used to solve several previously unreported polymorphs.

Aaron Celestian, Western Kentucky U, reported the work his group has done using time resolved *in-situ* x-ray and neutron diffraction to study ion exchange in microporous silicates. The group aims to selectively remove targeted radioactive ions in waste solutions, a goal that relates directly to issues faced by the nuclear energy industry. **John Roudebush**, UC Davis, presented work on thermoelectrics; **Wendy Queen**, NIST, discussed hydrogen storage; and **Xiaoping Wang**, ORNL, reported research being done at the Spallation Neutron Source on battery materials.

Eliot Specht, ORNL, discussed structural defects in materials and their relation to superconducting properties. X-ray diffraction techniques were described which characterize the stacking faults and nanoprecipitates which have been created in $YBa_2Cu_3O_7$ high-temperature superconductors in order to increase critical current density. These defects have increased the critical current of superconducting films greatly. They are identified by characteristic peak broadening and peak shifts in x-ray diffraction patterns.

Karena Chapman, ANL, discussed how the Pair Distribution Function (PDF) could be used to provide insights into the local and intermediate ranges of Li containing compounds. **Craig Bridges**, ORNL, presented on using Small Angle Neutron Scattering (SANS) to study the insertion of Li into carbon during charging.

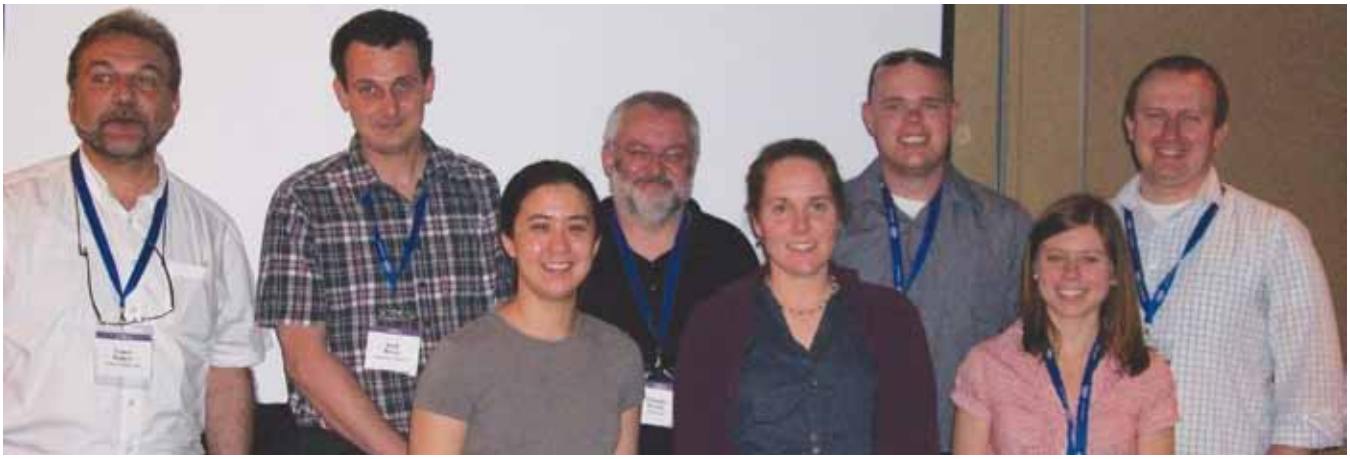
Kevin Rhodes, U Tennessee, highlighted a collaborative effort between UT and ORNL using a novel electrochemical cell in conjunction with x-ray powder diffraction for studying the structural changes that occur in $Li(Mn_{1.5}Ni_{0.5})O_4$ during charging and discharging. Kevin was the Powder Diffraction SIG's choice to receive a **Margaret C. Etter Student Lecturer Award**, (see right).

At left, Kevin Rhodes accepting the Etter Award from Session Chair Claudia Rawn.

Photo by NO student AV crew.

Hanno zur Loye, U South Carolina, reported using both x-ray and neutron powder diffraction to better understand the structure of $Sr_2Fe_{1.5}Mo_{0.5}O_{6-\delta}$, a Solid Oxide Fuel Cell (SOFC) anode material. **Valeri Petkov**, Central Michigan U, and **Bryan Chakoumakos,** ORNL, discussed using PDF and analysis of the Atomic Displacement Parameters (ADPs), respectively, to study the disorder of Li containing compounds. **Jason Hodges**, ORNL, described recent improvements to the SNS high resolution powder diffractometer, POWGEN, and its capabilities for studying energy related materials. There were also posters on transparent conductive oxides, nuclear materials, and hydrogen storage materials.

Claudia Rawn & Ashfia Huq



L to r: Valeri Petkov, Emil Bozin, Karena Chapman, Thomas Weber, Katherine Page, Benjamin Greve, Phoebe Allan, Olaf Borkiewicz.
Photo by the NO student AV crew.

Phoebe Allan was chosen by the Materials SIG to receive their Etter Young Lecturer Award. Jim Kaduk is shown with Phoebe and the award certificate above.
Photo by the NO student AV crew.

8.08 The Devil is in the Details: Local Structure and Diffuse Scattering

Phoebe Allan, U. St. Andrews, was selected by the Materials SIG to receive an **Etter Young Lecturer Award**. Phoebe discussed pair distribution function (PDF) studies of metal-organic framework materials with potential medical applications in the controlled release of therapeutic gases such as NO or CO. The PDF method was used to understand structure transitions which proceed *via* an amorphous intermediate and to probe the absorption sites of the NO molecules within the porous framework.

Benjamin Greve, Georgia Tech, presented a study of anomalous thermal expansion materials with the ReO_3 structure and told how this thermal expansion behavior changed with pressure. **Karena Chapman**, Argonne, used time-resolved PDF experiments to probe the nucleation and growth of supported silver nanoparticles with potential catalytic applications. Quantitative kinetics information was recovered allowing a detailed reaction mechanism to be delineated.

Emil Bozin, BNL, presented a study that uncovered an unusual temperature dependence of the local structure in the thermoelectric material lead telluride. Based on the insights from variable temperature PDF studies, a new type of phase transition was proposed in which fluctuating structural dipoles appear at high temperature from a low temperature state that has no dipoles.

Olaf Borkiewicz, Argonne, used PDF studies to probe novel cathode materials being developed for next-generation batteries. The PDF allowed phases and phase fractions to be determined throughout the electrochemical reactions despite simultaneous reduction in particle size to the nanoscale. **Thomas Weber**, ETH Zurich, presented a quantitative analysis of single crystal diffuse scattering using a three dimensional PDF analysis. This provided insight into the local structure of a metallic quasicrystal. **Clare White**, Lujan Neutron Scattering Center, LANL, presented a series of experiments probing the structure and formation of geopolymers. These have potential applications as environmentally-benign alternatives to concrete. Finally, **Valeri Petkov**, U Central Michigan, used high-energy anomalous PDF experiments to gain element-specific insights into the structure of bimetallic nanoparticles.

Karena Chapman

L to r: Peter Müller, Dean Johnston, David Watkin, Bruce Foxman, Joe Tanski, Eddie Snell, Bill Furey, Nigam Rath, Amy Sarjeant.

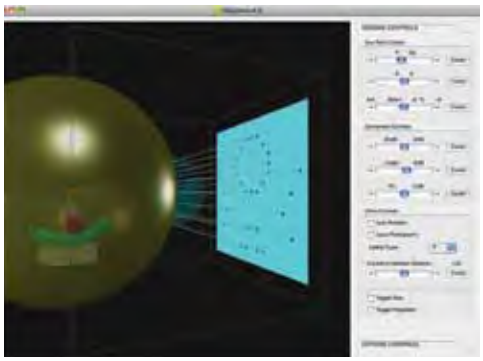
8.10: Crystallographic Teaching Techniques

David Watkin led the session with a retrospective look on data collection practices and how these relate to the not-quite-so-modern idea of MoO, (Multiplicity of Observations). By taking us on a tour of diffraction practices from Weissenberg and Precession techniques to current electronic area detector technologies, David underscored the need for high data redundancy in order to overcome errors inherent in data collection. This is a point that has far-reaching implications for analytical education in general.

Dean Johnston, Otterbein U, described how he incorporates crystallography into the already crowded curricula of undergraduate students. As he explained, crystallography can underscore and augment many topics already covered in undergraduate classes. For example by incorporating 3D molecular structure analysis with studies of VSEPR (valence shell electron pair repulsion) and chemical bonding, these concepts come alive to students. The wealth of tools available to crystallographers should not be overlooked when teaching undergraduates. Dean introduced two web modules developed at Otterbein, crystal packing and crystallographic point groups (**http://crystals.otterbein.edu**)

Joe Tanski, Vassar, discussed how he incorporates crystallography into his undergraduate course work. By looking for samples that are easily crystallizable and have some practical applications, Joe is able to tie structure analysis to the real world, and that holds the interest of his students. Joe suggests looking for samples in your own stock room, or looking for topically interesting compounds in *C&E News* articles. After learning to solve, refine and analyze crystal structures, Joe teaches his students to write papers on their results - which can often be submitted to appropriate journals.

Bill Furey, U Pittsburgh, highlighted the differences and similarities of teaching macromolecular crystallography. Drawing on his many years experience at the Cold Spring Harbor crystallography course, Bill showed us the many ways in which difficult concepts can be communicated to students. By using interactive software, students are able to explore the interactions of crystal properties with hardware geometries and radiation source characteristics. See right, the Ewald Sphere his program displays*.



Editor's note: the beautiful Ewald Sphere Bill used in his program came from George Phillips.

Switching gears to graduate-level education, **Peter Müller** described his syllabus for the MIT x-ray crystallography courses he instructs. Peter's courses are structured to educate students on the theory and practical aspects of crystallography. Homework problem assignments, for example symmetry, Bragg's Law, structure factors and Patterson techniques (to name a few) are worked in front of the class before they are turned in. This interesting approach allows students to draw from each other's experiences in order to obtain the maximum allotted points.

Eddie Snell regaled us with stories abort his Crystal Cookery project at the HWI. In order to assess crystallization effects of everyday chemicals, students are encouraged to collect common supermarket items that can be used to grow crystals of target proteins. Eddie works through the scientific method with his students as they attempt to identify new crystallization "silver bullets" from such items as hairspray, ketchup, and the ever-popular "Sesame Street Fizzy Tub Colors." Results of this massively parallel crystallization experiment were presented by his students at the end of the project and were submitted recently to the special education edition of *J. Applied Crystallography* (**v43**, 2010).

Getting back to small molecule crystallography, **Nigam Rath** outlined several experiments he uses to teach x-ray diffraction to undergraduate students at U Missouri-St. Louis. These experiments are designed to work in concert with general or organic chemistry synthetic labs. Nigam uses readily available and easily synthesized materials to show problems of structural analysis that can only be solved by crystallography. For example, while running the condensation reaction of citral and malonic acid, students produce an unexpected tricylcic lactone not easily characterized by mass spectrometry or NMR. Thus, students experience first-hand the utility of crystallography as an analytical technique.

Finally, **Bruce Foxman** told of his experiences at Brandeis. In his study of crystallographic topotaxy, Bruce was stymied by the problem of displaying, in a coherent way, the disparate crystal structures that result from a topotactic reaction. Bruce's students took up the challenge and together they developed a protocol for aligning structures. Bruce encourages anyone interested to send him mother and daughter phase data which he will then run through his topotaxy calculator.

Amy Sarjeant

8.09: Structural Enzymology 3 - Biology

L to r: Nicole Koropatkin, Matt Redinbo, Tasio Pyburn, Martin Boulanger, Michael Murphy, Etter Young Lecturer designated by the BioMac Sig - Rebekah Nash, Nicholas Noinaj. Photo by NO student AV crew.

8.11: Diffraction Studies of Industrial Materials

The session began with a perspective and discussion led by **Peter Lee**, DOE, on industrial uses and accesses to major user facilities supported by the DOE, a subject that concerns both the facility operators and users. Peter pointed out recent changes the DOE has implemented to make these facilities more user-friendly to industry; he also answered many questions from the audience.

Jonathan Almer, ANL, described synchrotron techniques and APS instruments which are important for industrial applications, including high-energy x-ray microtomography, high-energy diffraction microscopy (HEDM), and combined SAXS/WAXS. Jon stressed the advantages of high-energy x-rays for non-destructive, *in-situ* measurements of the phase, internal strain, and texture of bulk materials. He discussed the 3D grain structure mapping of Ni-based superalloys by HEDM using *in-situ* XRD, and the structure evolution of $LiCoO_2$ which is a cathode material for Li-ion batteries, as well as many issues related to industrial access to the APS.

Angus Wilkinson, Georgia Tech, reported an *in-situ* study of oil well cement at the APS using high-energy x-ray diffraction. The goal was to better understand the chemical kinetics of the cement under real world conditions. Taking advantage of the high penetrating power provided by high energy x-rays, large (10 mm) samples were measured inside titanium tubes, controlling pressure and temperature. Diffraction and shear wave reflection were measured simultaneously to monitor the modulus during the cement setting process. Validating models of the reaction kinetics versus temperature and pressure can help optimize cement setting conditions in the field (the study was funded by Halliburton).

Jim Kaduk, IIT, reported solving the structure of a cement related phase $Sr_{2.4}Ca_{0.6}Al_2O_6$ prepared at 1300°C. The crystal structure, based on high-resolution synchrotron data, could be best described by a monoclinic supercell, 32x the cubic cell which had been previously observed in this system. The structure distortion from cubic was found to be complex and, in addition to Rietveld refinement, symmetry mode analysis and DFT optimization were employed to obtain the final atomic coordinates.

Zhong Zhong, BNL, presented *in-situ* strain and phase mapping measurements using high-energy white-beam x-rays at the X17B1, the NSLS's superconducting wiggler beamline. The first part of his talk dealt with examples of strain-mapping in coatings, shot- and laser-peened surfaces, friction stir welds, and crack propagation around crack-tips in engineering alloys - which revealed an interesting "overload" phenomenon. The second part described applications of this technique to energy storage systems including both Li-ion battery and the sodium metal halide battery being commercialized by GE. In this case, the diffraction data were taken from real battery cells, charging and discharging under operating conditions.

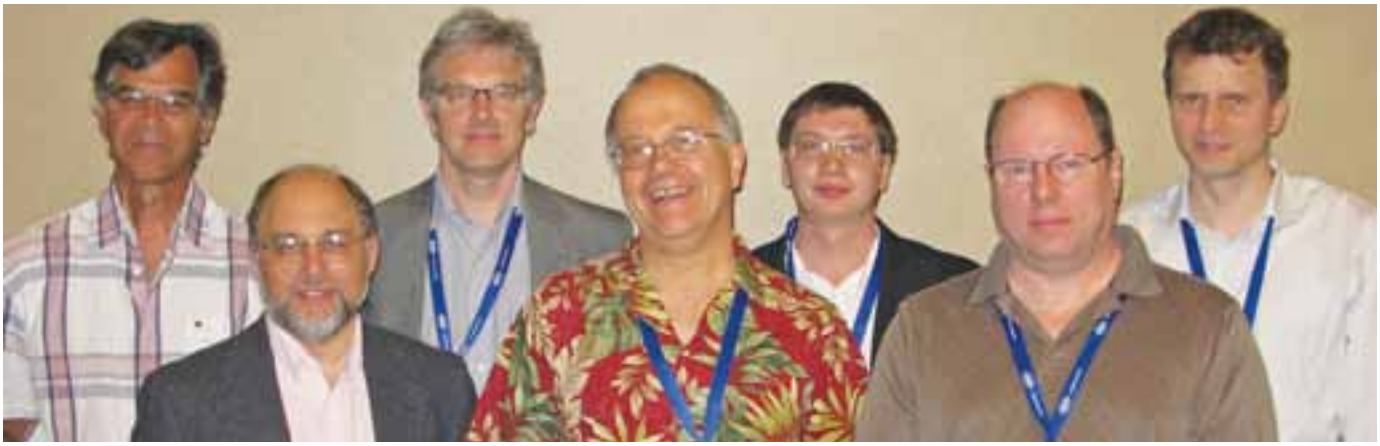
Jean Jordan-Sweet talked about the microelectronic material studies by IBM at the NSLS, from academically-oriented collaborations with university partners to problem-solving for IBM's manufacturing lines. She first described the wide range of microelectronics materials and the challenges that face the industry, and then focused on using synchrotron techniques, *in-situ* rapid annealing and pole-figure mapping, to study three generations of metal silicides used in manufacturing microelectronics. Detailed pole-figures can be obtained as a function of temperature; novel types of texture, for example axiotaxy, were identified.

Xun-Li Wang, ORNL, introduced their new materials engineering difffractometer, VULCAN, at the Spallation Neutron Source. VULCAN features a unique load frame with tension, compression and torsion which is capable of multi-axial loading. Applications range from residual stress mapping in engineering components, to *in-situ* study of deformation behavior, to time-resolved measurements of transformation kinetics. Xun-Li reported that VULCAN has been used to address practical problems in industry, e.g. failure analysis of superconducting cables used in the ITER project, and studies of the phase-transformation texture in Ti alloys. Currently the plan is to add an induction heater that will provide high temperature capability.

Yan Gao and Bryan Chakoumakos

8.12: Macromolecular Assemblies

L to r: Chris Hill, Brenda Schulman, Song Tan, Owen Pornillos, Christine Huang, Jason Stagno, Silvia Bilokapic. Kunchithapadam Swaminathan had to leave early and so is not pictured. Photo by the NO student AV crew.



L to r: Jim Cline, Brian Toby, Simon Billinge, Peter Stephens, Andrey Yakovenko, Jim Britten, Pavol Juhas. Photo courtesy of Marvin Hackert.

8.13: Evolution of Powder Diffraction Software: In Honor of Lachlan Cranswick

In addition to the scheduled speakers, this session included reminiscences of **Lachlan Cranswick** from the audience as well as email contributions from **Haohong Chen, John Evans, Jeff Nicolich, Sudhindra Rayaprol,** and **Nicola Scarlett**. Many people described the importance that Lachlan's work on CCP14 and distribution of software via CDs had on their own scientific careers. In session 8.16 see page ??, Ron Peterson described the mineral he has discovered and named **Cranswickite**.

Brian Toby, APS, Argonne, described recent enhancements to EXPGUI and CMPR software, two widely used programs for powder diffraction analysis. **Jim Britten**'s talk on the MAX3D software at McMaster U (available free of charge to academic users – Lachlan would surely approve) was a partial departure from the session's nominal topic of powder diffraction software. MAX3D permits visualization of images from 2D x-ray detectors directly in reciprocal space, so that puzzling streaks and blobs on the CCD images can be readily interpreted as stacking faults, pole figures, etc. **Ian Swainson**, Canadian Neutron Beam Centre, Chalk River, gave a moving review of Lachlan's life, especially in the period when he lived in Canada. **Simon Billinge**, Columbia, presented a number of recent results on the nanoscale structure of modern functional materials.

Andrey Yakovenko, Texas A&M, reported new developments on the generation of structural envelopes to assist in solving structures of metal-organic framework (MOF) materials from powder data. While structural envelopes have been widely used in zeolite materials, the extension to MOFs is not trivial, and it yields significant improvements in the ability to solve structures. Most powder diffraction lineshapes are modeled with empirical functions, but a complete understanding of optical aberrations depends on modeling the profile shape as a convolution of functions specific to the physical characteristics of the diffractometer. **Jim Cline**, NIST, described the use of this fundamental parameters approach in certifying standard reference materials with lattice parameters traceable to the International System of Units (SI) standard of length.

Pavol Juhas, Columbia, presented SrReal, a toolbox to analyze atomic pair distribution functions (PDFs) and other data. SrReal is distinguished from other PDF refinement programs by its library design, allowing users to incorporate purpose-built structure or lineshape models, as well as combining multiple structure criteria along side PDF refinements.

Peter Stephens

L to r: in back: Jonathan White, Sandra Reisinger, Jeffrey Lynn, Andrew Wills, Mark Green. In front: Manuel Perez-Mato, Feng Ye, Lisa DeBeer-Schmitt, Clarina dela Cruz, Songxue Chi. *Photo by the NO student AV crew.*

8.14: Diffraction Studies of Magnetic Materials

Jeffrey Lynn began the session with an overview of the *Magnetic and Structural Properties of Iron-based Superconductors* determined by neutron diffraction done at the NCNR, (NIST Center for Neutron Research). Rather than focusing, as most do, on the itinerant Fe in the system, Jeff spoke about the antiferromagnetic order of the rare earth moments and their substantial coupling to the Fe-subsystem. He also discussed recent results pertaining to the substitution of light rare earths for Ca in $CaFe_2As_2$ such that superconductivity was realized with a surprising TC reaching as high as 45°K. **Mark Green**, (NIST and U Maryland), talked about *Superconductivity in Iron Chalcogenides*. Although they possess simple layered tetragonal topology, the iron chalcogenides have a complex structural and magnetic phase diagram that is dependent on composition, ordered vacancies and occupancy of a secondary interstitial Fe site. Mark discussed in detail the role of the interstitial iron in the magnetism observed in the compounds as well as the process by which to control its occupancy.

Manuel Perez-Mato, Universidad del Pais Vasco, described the superspace symmetry of incommensurate magnetic structures. Motivated by the upsurge of intensive research on multiferroic materials of recent years, his group used superspace symmetry concepts to develop a systematic and comprehensive description of a multiferroic material's symmetry as fundamental information for explaining and classifying it's magnetoelectric effects.

The next talk by **Andrew Wills**, University College-London, on geometrically frustrated magnetism - a new route to room temperature spintronics described how the competing exchange interactions in magnetically frustrated systems such as Fe_3Sn_2 appear responsible for its giant anomalous Hall coefficient at room temperature, demonstrating how frustrated ferromagnets can provide new routes to RT spin-dependent electron transport properties that are suited to applications in spintronics.

Feng Ye, ORNL, presented his work using elastic neutron scattering techniques to investigate the magnetic properties of doped $Mn_{1-x}Me_xWO_4$ (Me: Fe, Zn, Co) and the correlation of magnetic with ferroelectric properties. Feng showed how magnetic neutron diffraction revealed a systematic evolution of the spin configurations that shows a flip of the spin spiral plane, changing the ferroelectricity from crystalline *b*-axis to *a*-axis.

Jonathan White, Paul Scherrer Inst., discussed spin wave excitation and neutron diffraction measurements designed to help understand the coupling of magnetic and ferroelectric hysteresis by a multi-component magnetic structure in Mn_2GeO_4. Two additional talks on magnetoelectric materials were: *Phase Separation and Phase Transitions in Multiferroic $K_3Fe_5F_{15}$* given by **Sandra Reisinger**, U St. Andrews, and *Elastic Neutron Scattering Study of Single Crystal $BaMnO_3$ and It's Doped Compounds* by **Songxue Chi**, ORNL.

Clarina dela Cruz

8.16: Earth Materials

There was some concern before the meeting that this session might not be well attended, because at the Salt Lake City meeting in 2007 there was a similar session that did not have great attendance. Happily, this time there were many people in the audience. One thing that helped was that there were few opposing sessions for non-bio people attending the conference, which meant that non earth-science people with an inorganic/materials background could also attend. Rather than the usual traditional crystallographic mineralogy, there was a nice mix of that aspect plus crystal chemistry; field work; the application of mineralogical studies to industrial problems such as acid mine waste from sulphide ores; and instrumentation (portable x-ray diffractometers destined for Mars or remote earth sites). Also, a talk on gas hydrates, an area where mineralogy intersects with solid state physics and chemistry studies, had broad appeal. The "materials" aspect was prominent in a talk on SANS and it's application to rock porosity evolution, with implications for metamorphism and oil sands recovery.

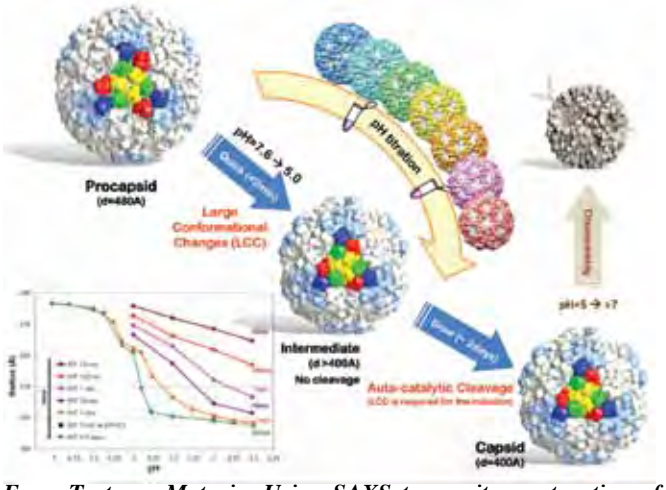
There were two last minute cancellations so after the conference started two other people were recruited to fill the talk slots and these speakers came up with 15 or 20 min talks with only 36 hours notice: **Claudia Rawn**, ORNL, described equipment and experiments aimed at gas hydrate formation inside real sediments, and **Claire White**, LANL, who spoke previously in the PDF session, discussed geopolymer concrete (an alternative low-CO_2 concrete) and the use of DFT (density functional theory) combined with PDF (pair distribution function) analysis to explore possible structures of amorphous and poorly crystalline mineral compounds such as metakaolin (dehydroxylatedkaolinite).

Ian Swainson & John Parise

8.17: Combined Techniques for Determining the Structure of Complexes & RNAS in Solution

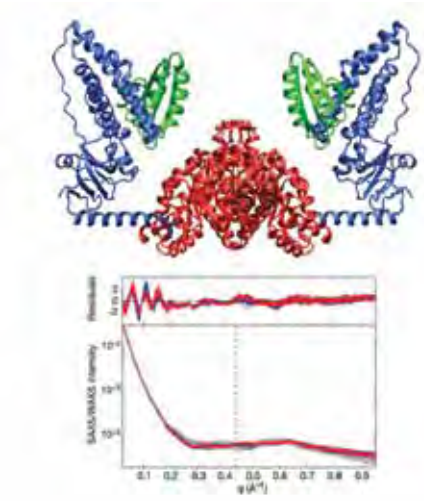
Combined use of SAXS and WAXS (wide angle x-ray scattering) together with x-ray crystallography, NMR and other biophysical tools is a new norm for structural biologists to tackle some of the most challenging problems facing biologists. All 8 speakers gave exciting talks, however we chose to highlight only four.

SAXS provides not only spatial information but temporal information as well. **Tsutomu Matsui** in Tsuruta's group at Stanford presented time-resolved SAXS studies that revealed crucial roles of autoproteolysis in viral maturation. The pH-dependent maturation of *Nudaurelia capensis* omega virus was studied in a variety of SAXS experiments aided by cryo-electron microscopic, crystallographic and biochemical techniques. The results showed strong pH-dependence of quaternary large conformational changes (LCC) and fast conformational changes (size reduction) were observed within 10ms after lowering the pH (see figure below). Interestingly, at pH=5.5, autocatalytic cleavage triggered by the LCC was required to achieve the final mature size, whereas particles below pH 5.0 did not require cleavage to achieve the final mature size. These results indicate that structural forces prior to cleavage counter the reduction in electrostatic force and that structural resistance is reduced when the autocatalytic cleavage occurs.



From Tsutomu Matsui: Using SAXS to monitor maturation of Nudaurelia capensis omega virus. Time-resolved SAXS was used to record the viral particle sizes in response to pH changes (the lower left panel). Matsui et el. (2010) Biophys J. 98, 1337-1343.

Often one can determine the 3D structures of individual components of a large complex relatively easily but fail to reveal how those components interact with each other. This challenge has become the one of next frontiers in structural biology. **Marius Clore**, Nat'l Inst. Diabetes, Digestive & Kidney Diseases, NIH, presented a structure determination of the solution structures of free Enzyme I (EI, ~128 kDa, 575 × 2 residues), the first enzyme in the bacterial phosphotransferase system, and its complex with HPr (~146 kDa), see figure below. Marius used prior structural knowledge combined with RDC (research data center) data, SAXS, WAXS, and neutron scattering (SANS) data.



From Marius Clore: The 3D structure of bacterium Enzyme I in complex with substrate HPr. The total molecular weight is 146 kDa, the largest complex structure solved in solution using combined SAXS, SANS and NMR.

Schwieters, Suh, Grishaev, Ghirlando, Takayama & Clore (2010) JACS 132, 13026-13045.

Comparison of the structures of EI and the EI-HPr complex with that of the phosphorylated EI intermediate reveal that a large (~70°) hinge body rotation of the EINα/β subdomain relative to the EIC domain

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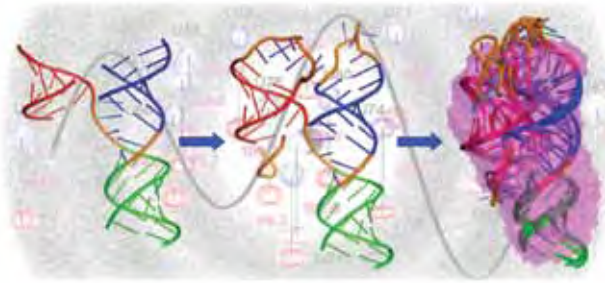
is required to allow the two subdomains to switch from the B state to the A state conformation. These large-scale interdomain motions shed light on the structural transitions that accompany the catalytic cycle of EI. The hybrid strategy Marius used makes optimal use of structural data from multiple sources to probe rigid body conformational transitions in large multidomain, multimeric proteins. This is the largest complex structure ever determined in solution. The SAXS/WAXS data were collected at the ID-12 beamline at ANL.

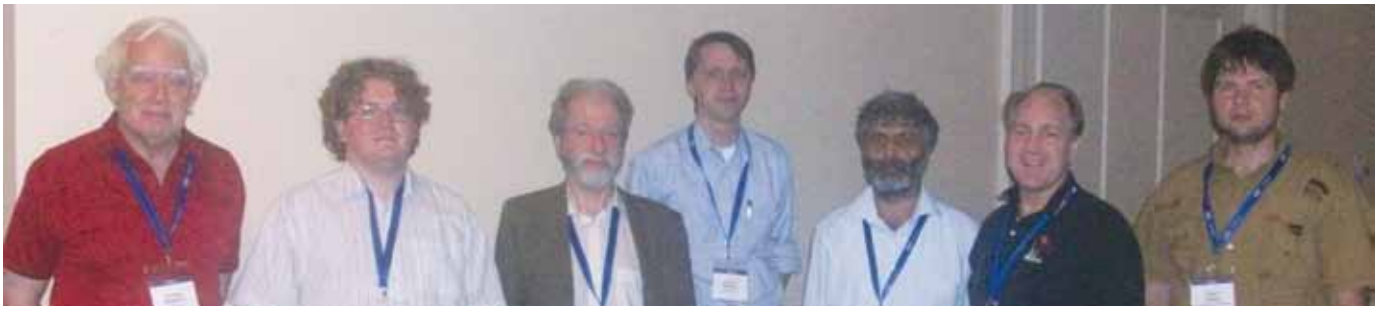
Our knowledge about the three-dimensional structures of RNAs and complexes is very limited due to technical difficulties with the current structural tools and methods. **Xiaobing Zuo**, ANL, described new methods for structure determination of RNA in solution using SAXS (for global shape) and NMR (for component orientation information). By combining both orientation and global shape restraints, one can determined the relative orientation and position of components in complexes. These methods were used to solve 3D structures of a 71-nucleotide (nt), and a 102-nt RNA (see figure at right) as well as a number of other protein or RNA complexes. 3D structures of large complexes or large RNAs, thought to be impossible to tackle using traditional x-ray crystallography or NMR spectroscopy alone are now accessible. The SAXS and WAXS data were recorded at the ID-12 at ANL.

There is no more straightforward and simple way than SAXS to characterize the flexibility and dynamic nature of biostructures, so important to any structural biology study. **Robert Rambo**, LBNL, presented a lecture on a novel use of the Porod-Debye law as a powerful tool for detecting and quantitating biopolymer flexibility in SAXS experiments. This law fundamentally defines a Porod-Debye region in the scattering intensities that captures information needed for distinguishing between folded and flexible particles. Proper application of the law in SAXS experiments can distinguish between discrete conformational changes and localized flexibility. This approach is more insightful and conclusive than traditional Kratky analyses of SAXS data with fully and partly flexible macromolecules.

Yun-Xing Wang & John Tainer



From Xiaobing Zuo: Combined use of SAXS and NMR spectroscopy for global structure determination of large RNAs in solution. The grey background is the two-dimension SAXS image, the red and blue contours (spots) are the in-phased and anti-phased (IPAP) spectrum that contains orientation information of imino 1H-15N bond vectors in the adenine RNA (RiboA) switch. The structure of the RiboA is shown in a backbone mode with the SAXS-derived molecular envelope of the molecule shown in magenta. Jinbu Wang, Xiaobing Zuo, Ping Yu, Huan Xu, Mary R. Starich, David M. Tiede, Bruce A. Shapiro, Charles D. Schwieters and Yun-Xing Wang; J. Mol. Biol., 2009, 393, 717–734. Xiaobing Zuo, Jinbu Wang, Ping Yu, Dan Eyler, Huan Xu, Mary R. Starich, David M. Tiede, Anne E. Simon, Wojciech Kasprzak, Charles D. Schwieters, Bruce A. Shapiro and Yun-Xing Wang; Proc. Natl. Acad. Sci. USA, (2010) 107, 1385-1390.



L to r: George Sheldrick, Thomas Womack, Gerard Bricogne, Clemens Vonrhein, Garib Murshudov, Ed Collins, Pavel Afonine.

Photo by the NO student AV crew.

8.19: Phasing & Refinement for Dummies

8.15: Data Processing with the Pros

This was the inaugural **Blackboard** session of ACA annual meetings. The goal was to merge educational and practical elements from a workshop-type format with the ease of timing of a general session. A large number of "pathogenic" data sets were made available in advance of the meeting including ***high mosaicity, many merged data sets, weak anomalous data, and ultra high resolution.*** During the session, the software experts offered their suggestions for how to process these data sets in order for the audience to get direct advice about how to deal with specific problems.

Kay Diederichs, U Konstanz, started by describing how to process data effectively using XDS; **Harry Powell**, MRC-LMB, discussed iMosflm features; **Jim Pflugrath**, Rigaku Americas, reported processing high-resolution data sets with d*TREK. **Matt Benning**, Bruker-AXS, showed some of the features of **Proteum** and **Wladek Minor**, U Virginia, finished with a presentation on HKL2000. Powerpoint, video and output files for comparison will be available through the **Blackboard Sessions** link at the ACA website, **www.AmerCrystalAssn.org**. The data sets are still available for download at the same site.

Many thanks to all of the contributors and speakers for donating data and their time to make the session a great success. We are also grateful for the kind donations made by **Rigaku, Rayonix, Dectris, ADSC, Bruker-AXS**, and **Global Phasing**.

Ed Collins

8.21 New Bio-Science from Emerging Opportunities and Sources

The Laser Coherent Light Source at Stanford, our newest thing, produces hugely bright, coherent, x-ray pulses that are only tens of femtoseconds long. Each pulse essentially destroys the specimen, but not before x-rays scatter to reveal its structure. **Petra Fromme**, ASU, reported a demo experiment on approximately 1μ crystals of the huge (36 proteins, 381 cofactors) Photo System I complex. Even though each crystal contained only about 103 molecules, the several tens of thousands of diffraction patterns, one per crystal, that were processed could reproduce the structure of the complex. This and another experiment: stimulating crystals with light pulses then recording x-ray diffraction patterns, suggest that the method will produce full time-resolved images – molecular movies – of photo-excited molecular species.

Lee Makowski, Northeasten, described coherent-beam measurements his group had done in collaboration with Ian Robinson, using the APS at ANL. Studying cellulose fibres from corn, they were able firstly to observe Bragg diffraction from nano-crystals of cellulose, and then to employ the 3D speckle-patterns about the Bragg reflections to reconstruct the shape of the crystals themselves. The jagged patterns they observe suggest that the crystals are bundles of 25nm cellulose filaments, with variations in size and shape that reflect the position of the crystal in the plant.

Julian Chen, ANL, described some of the procedures he and Steve Ginell have used at ANL to measure ultra-high resolution diffraction data from crystals of crambin. Modifications of typical procedures are: to employ very high energy photons (in their case 19keV); to put the planar detector very close; and to tilt the detector to record high-angle reflections. At resolutions ~ 0.6Å Julian observes that multiple high-quality crystals of crambin show clear non-isomophisms as revealed by Fo(1) - Fo(2) difference electron density maps. The non-isomorphism is less from duplicate measurements made on different parts of the same crystal.

Most single-crystal x-ray diffraction data measured from macrocmolecular specimens employs radiation in the 8-13keV (0.95-1.5Å) range. Some believe that there may be important advantages to stepping outside that range. **Roger Fourme**, Soleil synchrotron, reported tests at 18 and 33keV, and others have published data 6.5-56 keV, suggesting that a larger amount of data, and more accurate data, can be collected from a given cryocooled sample at higher energy. Exploiting the full potential of this approach would justify dedicated short-wavelength MX beamlines, and more efficient detectors, particularly pixel-array detectors based on elements heavier than Si.

The opposite approach is being taken by **Armin Wagner**, Diamond Light Source, to solve a different problem. The use of selenomethionine substitution in proteins has become an almost universal mechanism for one to determine the phase of diffraction, and thus solve the structures of macromolecules in crystals. Occasionally this simply isn't possible, and *then* one can revert to an old, almost alchemical approach and create isomorphous heavy atom derivatives. However, Armin has another alternative in mind; he is creating a diffraction station for Diamond Light Source that will produce photons in the 3-8 keV range. Tests show that at these energies the anomalous dispersion from the naturally occurring sulfur atoms in protein suffices to provide a strong enough signal to determine phases. Two challenges are: first, that this soft radiation is so strongly absorbed by matter -even air -that the whole experiment must be done in a vacuum and to mount and cryo-cool a macromolecular crystal in a vacuum will be difficult; and second, that the long wavelengths will limit the minimum d-spacing that can be observed. Armin will array area-detector elements in a cylinder around the crystal axis, inside the vacuum chamber, to get around this problem.

Alberto Podjarny, Institut de Génétique et de Biologie Moléculaire et Cellulaire, Strasbourg, is combining neutron single-crystal diffraction data, from the reactor at Institut Laue-Langevin (ILL), with equivalent x-ray data from the ESRF. He and his colleagues are studying details of the function of anti-freeze proteins, found in tissues of Antarctic fish. Previous studies revealed an ice-binding surface on the proteins, which had the confusing property that there were many hydrophobic patches. Neutron studies with D_2O in the solvent allowed them to show conclusively that the enigmatic hydrophobic residues bind inside the holes of the ice surface, which explains the specific strong binding to ice rather than to water.

Andrey Kovalevsky, LANL, seeks to understand the enzymatic pathway of xylose isomerase. This is the enzyme that can transform xylose, a sugar found in wood, to xylulose, which yeast will ferment. They use the ***quantum enzymology*** method in which many atoms around the active site (~1000) are treated solely with quantum chemistry. Knowing especially the positions of hydrogen atoms is critical to this study. The H locations are derived from joint RT x-ray/neutron crystallographic structures determined at the Los Alamos LANCSE facility and ILL, validating the method.

Finally, **Timothy Fenn**, Stanford, reported work done in collaboration with Paul Langan of ORNL and Axel Brunger and Vijay Pande of Stanford. They exploited the detailed information provided by explicit H-atom positions for combined x-ray/neutron refinement in crystals of Z-DNA. They employed a modern, polarizable, atomic multipole force field, specifically providing explicit electrostatic treatment of hydrogen atoms. The resultant models offer a sufficiently improved description of the true structure that x-ray and neutron diffraction data-derived Rfree values improve, suggesting improved agreement across varied experimental data.

Bob Sweet

22nd IUCr Congress, Madrid, Spain, 22-30 August

Before I describe the meeting itself, I would like to preface my comments with a few words about how pleasant it was to visit Madrid. Madrid as a city is a mixture of old and new, with a large number well-maintained buildings from many different time periods; these are combined with many modern homes and offices. English is not as widely spoken as in many other European cities, but I found the Spanish people to be exceptionally friendly and helpful. I am still puzzled, however, at how the Spanish survive without sleeping. Restaurants do not open for dinner until 9 pm or so. For me, the conference included two dinner meetings, in addition to the banquet. None started before 9 pm and all ran past midnight. Since a large fraction of participants needed to take 1-3 metro trains to reach their hotels, nights did end quite late, considering that morning talks started at 9 am. No complaints about the metro, however - not only was it convenient but an unlimited pass was included in the registration.

The weather was predicted to be hot, and my first few days in the city managed to match Chicago at its August worst, but once the Congress started, it moderated. Then again, I was spending all my days inside the large ***Palacio Municipal de Congresos de Madrid*** (Municipal Conference Centre), an attractive and welcoming building with large open spaces. By the second day I had figured out the building's topology, and found it convenient to move between presentation rooms, the exposition space, and meeting rooms. Alas, half of the meeting's posters were exiled to the sub-basement which was very poorly connected to the rest of the meeting space - the very long flights of stairs and inconvenient elevators discouraged many people from visiting that area. This was the only flaw in what was an otherwise exceptional meeting site - ***future meeting organizers take note!*** There was even a gallery of art, but this was blocked off from the conference.

Upper right: the Royal Palace of Madrid; the next photo down is the Basilica. The Palacio Municipal, where the IUCr congress was held is pictured below.

The conference was packed with presentations, -from 9 am to at least 9 pm. The usual IUCr format was followed: morning and late afternoon keynote/plenary sessions unopposed, and 6 simultaneous micro-symposia. Lunch was combined with poster sessions. Micro-symposia chairs were very good at keeping on schedule, and despite the several floors used for the meeting, the talks were in very close proximity making it easy to sample talks between different sessions and the Software Fayre. Despite my desire to attend the morning micro-symposia, the late nights kept my attendance down, so I have no comment on them. I think the high point for me was the plenary talk from Venki Ramakrishnan. My knowledge of biology has not progressed all that much since my high school biology course (except that lots that we knew then is known now to be ***not*** so), but I found his talk to be both clear and profoundly exciting.

A cultural highpoint of the meeting was a two-hour performance of folk dances by a troupe of 18 dancers. For the second half of their performance they were accompanied by four musicians and a singer. They performed choreographed versions of regional dances from all different regions of Spain. This went from 7:30 - 9:30 pm and the large auditorium remained filled, with only a few people trickling out. I was surprised that one could not hear rumbling of stomachs above the music.

After a tiring, but enjoyable meeting, I am looking forward very much to getting home, but by 2014 I should regain my enthusiasm for repeating the experience in a very attractive location: the **Palais des Congress de Montreal**.

Brian Toby

The leftmost two photos were taken at Buen Retiro Park in Madrid; at lower right, the Villanueva Pavilion in the Royal Botanical Gardens, with statue of Carl Linnaeus in foreground. All photos were taken by Victor and Carole Young.

Seated (L-R): Francine Belanger (UdeM), Erin Dodd (McGill), Alya Arabi (Dalhousie), Berline Mougang-Soume (UdeM), Fatima Helzy (UdeM), Michel Simard (UdeM) Standing (L-R): Debakanta Tripathy (UdeM), Jim Britten (McMaster), Shawn Shadkarmi (Toronto), Chuck Campana (Bruker-AXS), Hein Schaper (UdeM), Ilia Korobkov (Ottawa), 'visitor' (McGill), Zhijie Chua (McGill), Andreas Decken (New Brunswick), Christopher Allan (Windsor), Tiffany Smith (Syracuse), Jonathan Gooch (Syracuse), Mourad Intissar (UdeM), Brandon Groves (Dalhousie), Jessica Virdo (Toronto), Robert Giacometti (UdeM), Thierry Maris (UdeM), Hakan Dal (Anadolu), Thao Nguyen (Concordia), Louis Cuccia (Concordia), Yuan Fang (Concordia), Sima Mehrpajouh (Concordia) Not pictured: Adrien Côté (Xerox RCC)

3rd Canadian Chemical Crystallography Workshop, Université de Montréal, 10-14 June

This workshop was a satellite to the annual **Canadian Society for Chemistry** meeting, organized by the CNCCr, with local arrangements handled beautifully by UdeM crystallographers **Francine Bélanger, Thierry Maris, Hein Schaper**, and **Michel Simard**. Rumour has it that Francine did most of the work. The local crew was joined for the week by **Chuck Campana**, Bruker-AXS, **Adrien Côté**, Xerox Research Centre of Canada, **Andreas Decken**, U New Brunswick, and **Jim Britten**, McMaster U.

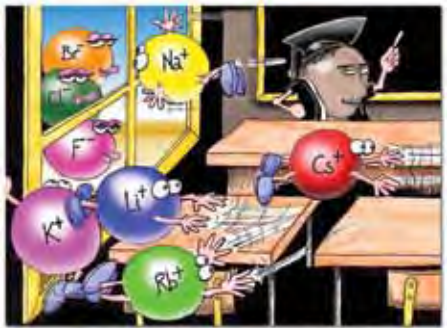
A flexible program of lectures on general, practical, and a few advanced topics were presented in the mornings **www.canadian-crystallography.ca/cccw/program.html**, and in the afternoons the students worked on data sets (canned, collected at UdeM, or brought from home) with the help of the instructors. The afternoon sessions, which often extended beyond the scheduled closing time, were most appreciated by the students. "I really loved how many instructors were there!" "It gave a lot of us a chance to get one-on-one attention with structures. I also loved the enthusiasm that all of the instructors had!" "It was awesome that we could ask any questions about crystallography and have instructors be happy to answer them!" "This was an amazing experience and I learned so much, I am so grateful that I was able to attend this workshop!"

The CCCW is in its third year, and this travelling workshop is showing some very interesting side effects. Chemistry departments are more than willing to host the meeting and allow their crystallographers to spend five straight days working with students. Word gets around, and students come not only from Canadian provinces, but also from around the world (Hakan travelled from Anadolu University in Turkey to attend!). Locally, new lines of communication are opened in the community – students and faculty from Concordia and McGill Universities now know the talented crystallographers at l'Université de Montréal (and the powerful instrumentation!). The instructors all learned a few new tricks, and the depository of teaching material is growing. A few of the workshop students always get hooked, and will become proficient crystallographers. Local industrial researchers are given an opportunity learn some crystallography and get to know the characterization capabilities of the university x-ray labs.

The workshop is not intended to be a comprehensive school for crystallographers – there are a number of very good offerings by the ACA and the IUCr - but more a training session for interested chemists, geologists, etc. who want to have a better understanding of the techniques. In order to keep workshop registration fees to a minimum, the CNCCr has started a workshop fund which will be used solely to support various crystallography workshops in Canada, including chemical, powder, and macromolecular schools. Donations can be made to **CNC for IUCr, c/o Marie Fraser, Treasurer, Dept of Biol. Sci., U Calgary, 2500 University Drive NW, Calgary, Alberta, T2N 1N4, Canada**. Many thanks to Sid Pharasi, Bruker-AXS, for providing the opening donation! These workshops are an important part of the scientific education of our students, and it looks like the demand will be increasing. If you are in a position to offer support, it will be greatly appreciated (and acknowledged).

The ***4th CCCW will be held in Calgary, Alberta in late May, 2012***. Watch for it!

Jim Britten



"Perhaps one of you gentlemen would tell me just what it is outside the window that you all find so attractive ...?"

Cartoon courtesy of Nick D. Kim. See www.lab-initio.com/index.html

The 43rd crystallographic course on electron diffraction and the 44th crystallographic course on powder diffraction took place simultaneously at the **Ettore Majorana Centre**, Erice, Italy June 2-12, 2011.

The course organizers were **Annalisa Guerri** (left), and **Paola Spadon**, on the right with **Bo Song**. **Paola** can also be seen --at 2 o 'clock in the circle dance photo below. **Annalisa** might or might not be the one at 11 o'clock. Bo is holding a **Lodovico Prize** certificate. The **Lodovico Prize** is new and meant to recognize both scientific ability and interpersonal engagement, in and out of the lecture hall. It is given to *the most dynamic student* in memory of **Lodovico Riva Di Sanseverino** (1939-2010).

As is traditional at Erice, there were many social events: **Celebrating Lodovico in San Domenico,** followed by **Welcome Buffet & Sicillian Evening in San Francesco**; **Excursion to Selinunte and Segesta Temple (with dinner in front of); *or* San Vito lo Capo beach**; **Pasta Party; Boat trip to Mothia Island (extensive walk, Phoenician archaeology); *or* Trapani beach; Pizza Party & Disco; Goodbye Buffet.**

There were, of course, many lectures, and posters and poster prizes. **Chris Gilmore**, directly above, was one of the distinguished powder diffraction scholars to lecture.

Boston 2012
AMERICAN CRYSTALLOGRAPHIC ASSOCIATION

ACA 2012 July 28 - August 1

Westin Boston Waterfront Hotel

Abstract Deadline: March 31, 2012
Student and Young Scientist Travel Grant Applications: March 31, 2012
Advance Registration Deadline: May 31, 2012
Advance Hotel Registration Deadline: July 5, 2012

Register online and see Call for Papers at
www.AmerCrystAssn.org

Meeting website: ***www.amercrystalassn.org/2012-meeting-homepage***

Exhibits website: ***www.amercrystalassn.org/2012-exhibits***

The 2012 Meeting will have a 4-day, 5 concurrent session pattern. The meeting will start with workshops on Saturday, July 28, and scientific sessions on Sunday, July 29; meeting will end on Wednesday, August 1, after the Awards Banquet.

Program Chairs

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Poster Chair
Ilia Guzei
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NOVEMBER 2011

28-2 **MRS Fall Meeting & Exhibit,** Hynes Conventio Ctr, Boston, MA. Registration begins mid September.

2-4 **69th Annual Pittsburgh Diffraction Conference & 5th Annual Ohio Valley Crystallography Symposium,** Case Western Reserve U, Cleveland, OH. Registration and abstract deadline: September 30, 2011. See **www.pdsconference.org**

MARCH 2012

8-16 **32nd Berlin School on Neutron Scattering**. Lise Meitner Campus/Helmholtz-Zentrum Berlin fur Materialien und Energie. See **nschool@helmholtz-berlin.de**

11-15 **Annual Pittcon Conference & Exposition,** Orange County Convention Center, Orlando, Fl. (Expo March 12-15). This is the world's largest laboratory science conference & exposition. 1,000 exhibitors, 2,000 presentations, and attendees from > 90 countries are expected. Program Chair: Joe Grabowski **joeg@pitt.edu**

13-14 **Select Biosciences Structural and Computational Chemistry Conference**, Munich, Germany, as part of the Discovery Chemistry Congress.

19-23 **ICDD Spring Meeting,** ICDD Headquarters, Newtown Square, PA.

26-27 **Small Molecules in Interactions Internat'l Symposium,** Ruhr Universität Bochum, Germany **www.rub.de/smi** Contact Vera Vasylyeva: **vv-smi@rub.de** or Sebastian Marquardt: **sm-smi@rub.de**

JULY 2012

28-2 **ACA2012, Westin Boston Waterfront Hotel, Boston, MA. See previous page for info.**

AUGUST 2012

7-11 **ECM27 27th European Crystallography Meeting**, Bergen, Norway. See **ecm27.ecanews.org/**

SEPTEMBER 2012

9-13 **EMC 2012, European Mineralogical Conference**, at Johann Wolfgang Goethe-University, Frankfurt, Germany.

JANUARY 2013

8-13 **10th NCCR Pratical Course & 3rd Winter School: Introduction to Biomolecular Modelling,** Kandersteg, Switzerland. Contact Sraboni Ghose, U Zurich Winterthurerstrasse 190 CH - 8057 Zurich.

DECEMBER 2012

20-24 **AsCA'12.** Adelaide, Australia. See **www.asiancrysassn.org**

AUGUST 2014

5-12 **XXIII Congress and General Assembly of the IUCr,** Montreal, Quebec, Canada. See IUCr website: **www.iucr2014.org/**

Ludo Frevel Scholarship Awards

Application Deadline: 26 October 2011

To encourage promising graduate students to pursue crystallography-oriented research, the International Centre for Diffraction Data (ICDD) has established the **Ludo Frevel Crystallography Scholarship Fund.** Multiple recipients are selected on a competitive basis, each receiving an award of $2,500. **Applications must be received by the ICDD by 26 October 2011.**

Qualifications: The applicant should be enrolled in a graduate degree program during the 2012 calendar year with major interest in crystallography, for example crystal structure analysis; crystal morphology; modulated structures; correlation of atomic structure with physical properties; systematic classification of crystal structures; phase identification; and materials characterization. Students who graduate prior to 1 July 2012 are not eligible for the 2012 scholarship award. There are no restrictions on country, race, age or sex. The term of the scholarship is one year.

Visit **www.icdd.com/resources/awards/frevel.htm** for information on the new on-line application procedure.

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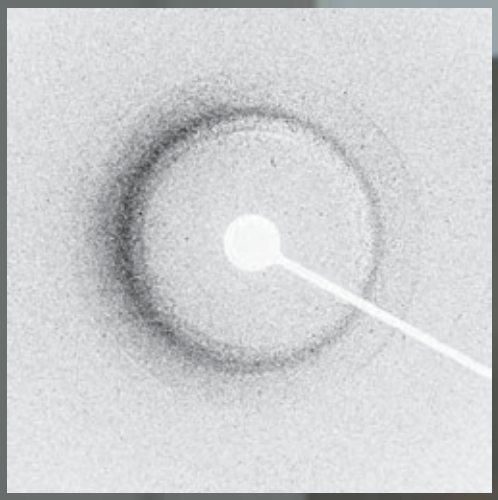

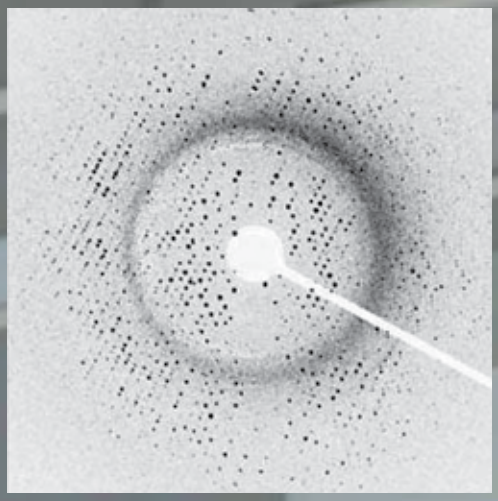

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