

Virginia Tech Crystallography Laboratory



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2006 Annual Report

The Virginia Tech Crystallography Laboratory is a co-located facility of the Departments of Biological Sciences, Chemistry, and Geosciences, within the College of Science. The mission of VTX is three-fold:

1. **Education** – We teach crystallography courses to VT students, and we hold an annual summer crystallography workshop for faculty and students from other Universities.
2. **Service** – We provide crystallographic services (structure solution and refinement) to research groups at Virginia Tech and other universities in the region. We organize and host the Virginia Tech Structural Biology symposium.
3. **Research** - Members of VTX pursue their own research programs in crystallography and structural biology.

This has been an exciting year for the Virginia Tech Crystallography Laboratory, with some major new developments that will allow us to pursue all three aspects of our mission.

In the field of structural biology, Florian Schubot joined us as an assistant professor in the Department of Biological Sciences. The laboratory then organized and hosted the first VT Structural Biology Symposium over two days in March. The symposium attracted 150 registrants, with guest speakers Prof. Dr. Rolf Hilgenfeld (Universitaet Lubeck, Germany) and Prof. Andrew Bohm (Tufts University, USA), and several VT faculty speakers. The symposium was sponsored by Oxford Diffraction, the College of Science, the Department of Biological Sciences and the Department of Biochemistry, and several biotech companies exhibited products. A second symposium is planned for March 2007.

Later in the year Oxford Diffraction moved their US corporate headquarters to Blacksburg to co-locate with VTX. This brought new diffractometers and upgrades worth \$1,600,000 for a cost to VT of only \$250,000 funded through the Commonwealth Research Initiative. A dual-source Gemini diffractometer from Oxford Diffraction was installed to provide improved facilities for small-molecule service crystallography. This is the first true dual-source diffractometer to be sold in the USA. The Xcalibur-2 diffractometer was converted to full-time high-pressure usage to relieve the over-demand

on Xcalibur-1. And the protein diffractometer was upgraded to a PX-Nova with a much higher intensity “microsource” Xray system to enable us to measure much smaller protein crystals than before. The last part of these upgrades was the installation in October of a PX-scanner system from Oxford Diffraction, which is used to pre-screen protein crystals by diffraction while they are still growing in well plates. The PX-scanner is temporarily located at the “VTX-outstation” in the Fralin Institute on campus.

The move of Oxford Diffraction and the expansion of the facilities in VTX were announced at a press conference in October:

“Oxford Diffraction’s decision to locate its North American office in Blacksburg is a testimony to the energy and world-class scholarship of our faculty, who have created this exciting partnership for scientific discovery,” said Virginia Tech President Charles Steger. “It also signifies Virginia Tech’s position as one of the leading materials science research universities in the United States”. “The partnership with Oxford Diffraction is an excellent example of the type of relationship we hoped the CRI funding would encourage,” said Del. Harvey Morgan, chairman of the House Appropriations Higher Education Subcommittee. “This partnership leverages state dollars to provide increased private sector and federal funding and promises to generate intellectual property that may well result in jobs for Virginians.”

During 2006 the departments and the College decided to improve the facilities for the laboratory by moving it to the new *Integrated Life Sciences Building* which is currently being constructed on the Corporate Research Center adjacent to campus. The improved environment for the instruments, including being able to house all of them in a single laboratory in a building that will also house many of the structural biology programs and which is immediately adjacent to the headquarters of Oxford Diffraction Inc, will allow us to further improve our research and services. The move is scheduled for Fall 2007.

The laboratory continued its regional leadership role by providing service crystallography facilities to research groups from several smaller colleges in south-west Virginia, and by holding the fourth of our summer crystallography workshops for faculty and students from colleges throughout the Commonwealth. The 2006 workshop attracted 25 people, of whom 10 were women and 4 were minorities, from these colleges as well as several students from outside of Virginia who had enrolled for the summer in the NSF REU program in the Chemistry department.

In its service chemistry role, led by Dr. Carla Slebodnick, the laboratory collected about 120 datasets in support of the research programs of the groups of Professors T. Long, G. Yee, K. Brewer, B. Hanson, P. Deck, R. Gandour, H. Gibson, H. Dorn, D. Kingston, N. Castagnoli, and E. Marand. Papers containing the structures are listed below.

More information is available at www.crystal.vt.edu or by contacting Prof. Ross Angel, email: rangel@vt.edu.

People

Staff, students and faculty are appointed to the individual departments, and work together in the laboratory. Among the post-doctoral researchers who started 2006 in the laboratory, Dr. Fabrizio Nestola moved to a faculty position at the University of Padua in Italy, Dr. Husin Sitepu to a faculty position at the American University in Riyadh, and Dr. Carine Vanpeteghem moved to a position at Georgia Tech. Jason Burt successfully defended his PhD thesis on the crystal chemistry of the Al_2SiO_5 polymorphs and has become a post-doc at the University of Arizona. Dr. Jinyuan Yan joined the laboratory as the programmer for the CEAD project, a community project to develop automated processing of powder diffraction data. Dr. Jing Zhao was promoted to Senior Research Associate to acknowledge his leading role in the operations of the laboratory. A full list of affiliated staff and faculty follows:

Ross Angel	VTX Director, Professor of Crystallography in Geosciences
Mike Berg	Instructor in Chemistry
Phil Burcham	Technical staff, Geosciences
Jason Burt	Graduate student, Geosciences (graduated June 2006)
Theresa Detrie	Graduate student, Geosciences
Carla Finkielstein	Assistant Professor, Biological Sciences
Liangming Hu	Graduate student, Chemistry
Eleda Johnson	Graduate student, Geosciences
Fabrizio Nestola	Postdoctoral associate, Geosciences (to March 2006)
Nancy Ross	Associate Dean, Professor of Mineralogy in Geosciences
Florian Schubot	Assistant Professor, Biological Sciences
Husin Sitepu	Compres Research Fellow (to summer 2006)
Carla Slebodnick	VTX Associate Director, Instructor in Chemistry
Carine Vanpeteghem	Postdoctoral associate, Geosciences (to June 2006)
Nancy Vogelaar	Postdoctoral associate, Biological Sciences
Jinyuan Yan	Postdoctoral associate, Geosciences
Jing Zhao	Senior Research Associate, Geosciences

During the year the laboratory also hosted research visits by the following scientists:

Tiziana Boffa-Ballaran	Bayerisches Geoinstitut, Bayreuth, Germany
Maciej Bujak	University of Opole, Poland
Diego Gatta	University of Milan, Italy
Fabrizio Nestola	University of Padua, Italy

Publications

- [1] Angel RJ, Bujak M, Zhao J, Gatta GD, Jacobsen SD (2007) Effective hydrostatic limits of pressure media for high-pressure crystallographic studies. *Journal of Applied Crystallography* 40:26-32.
- [2] Angel RJ, Zhao J, Ross NL, Jakeways CV, Redfern SAT, Berkowski M (2006) High-pressure structural evolution of a perovskite solid solution $(\text{La}_{1-x}\text{Nd}_x)\text{GaO}_3$. *Journal of Solid State Chemistry* submitted:
- [3] Brown JM, Abramson EH, Angel RJ (2006) Triclinic Elastic Constants for Low Albite. *Physics and Chemistry of Minerals* 33:256-265.
- [4] Bujak M, Angel RJ (2006) High pressure and low temperature induced changes in $[(\text{CH}_3)_2\text{NH}(\text{CH}_2)_2\text{NH}_3][\text{SbCl}_5]$. *Journal of Physical Chemistry, part B* 110:10322-10331.
- [5] Bujak M, Angel RJ (2007) $[(\text{CH}_3)_2\text{NH}_2]_2[\text{SbCl}_5]$ at low temperature and high pressure - X-ray and Raman studies. *in preparation*
- [6] Burt J, Ross NL, Angel RJ, Koch M (2006) Equations of State and Structures of Andalusite and Sillimanite to 10 GPa. *American Mineralogist* 91:319-326.
- [7] Burt J, Ross NL, Gibbs G, Rossman G, Rosso K (2007) Potential protonation sites in the Al_2SiO_5 polymorphs based on polarized FTIR spectroscopy and properties of the electron density distribution. *Physics and Chemistry of Minerals* submitted:
- [8] Burt JB, Gibbs GV, Cox DL, Ross NL (2006) ELF isosurface maps of the Al_2SiO_5 polymorphs. *Physics and Chemistry of Minerals* 33:138-144.
- [9] Cai T, Slebodnick C, Xu L, Harich K, Glass TE, Chancellor C, Fettinger JC, Olmstead MM, Balch AL, Gibson HW, Dorn HC (2006) A pirouette on a metallofullerene sphere: Interconversion of isomers of N-tritylpyrrolidino I-h $\text{Sc}_3\text{N}@C\text{-}80$. *Journal of the American Chemical Society* 128:6486-6492.
- [10] Carlier PR, Zhang YQ, Slebodnick C, Lo MMC, Williams ID (2006) Effect of 2,6-disubstituted aryl groups on acyclic conformation: Preference for an antiperiplanar orientation of the geminal and vicinal hydrogens. *Journal of Organic Chemistry* 71:8835-8841.
- [11] Deck PA, McCauley BD, Slebodnick C (2006) Transition metal cyclopentadienyl complexes bearing perfluoro-4-tolyl substituents. *Journal of Organometallic Chemistry* 691:1973-1983.
- [12] Fan H, Slebodnick C, Hanson BE (2006) A tetranuclear zinc cluster, $(\text{bipy})_4\text{Zn}_4(\text{HPO}_3)_4$, that stabilizes a hydrogen bonded chain of water molecules: transformation of a zinc phosphite 4-ring to a cluster. *Inorganic Chemistry Communications* 9:103-106.
- [13] Fan J, Yee GT, Wang G, Hanson BE (2006) Syntheses, structures, and magnetic properties of inorganic-organic hybrid cobalt(II) phosphites containing

- bifunctional ligands. *Inorganic Chemistry* 45:599-608.
- [14] Gatta GD, Angel RJ (2006) Elastic behavior and pressure-induced structural evolution of nepheline: implications for the nature of the modulated superstructure. *American Mineralogist* accepted:
- [15] Gibbs GV, Cox DF, Crawford TD, Rosso KM, Ross NL, Downs R (2006) Classification of metal-oxide bonded interactions based on local potential- and kinetic-energy densities. *Journal of Chemical Physics* 124:084704.
- [16] Gibson HW, Clifton-Dickson J, Lecavalier PR, Wang H, Berg MAG, Merola JS (2007) Reissert Compounds in Stereoselective Synthesis: Enantioselective Synthesis of 1-Isoquinolyl t-Butyl Carbinols. *Journal of Organic Chemistry* submitted:
- [17] Gibson HW, Wang H, Slebodnick C, Merola J, Kassel S, Rheingold AL (2007) Isomeric 2,6-pyridino-cryptands based on dibenzo-24-crown-8. *Journal of Organic Chemistry* submitted.
- [18] Han Q, Robinson H, Gao YG, Vogelaar N, Wilson SR, Rissi M, Li J (2006) Crystal structures of *Aedes aegypti* alanine glycoxylate aminotransferase *Journal of Biological Chemistry* 281:37175-37182.
- [19] Hu L, Fan J, Slebodnick C, Hanson BE (2006) Structural Diversity in 4,4'-Trimethylenedipyridine-zinc phosphite Hybrids: Incorporation of Neutral Guest Molecules in Hybrid Materials. *Inorganic Chemistry* 45:7681-7688.
- [20] Huang F, Slebodnick C, Switek KA, Gibson HW (2007) Inclusion [2] Complexes Based on the Cryptand/Diquat Recognition Motif. *Journal of Organic Chemistry* submitted.
- [21] Huang FH, Slebodnick C, Switek KA, Gibson HW (2006) Bis(meta-phenylene)-32-crown-10-based cryptand/diquat inclusion [2] complexes. *Chemical Communications* 1929-1931.
- [22] Nestola F, Nemeth P, Angel RJ, Buseck PR (2007) Equation of state and crystal structure behavior of the new germanate post-titanite phase in excess of 9 GPa. *American Mineralogist* submitted:
- [23] Ross NL, Hoffman C (2006) Single crystal neutron diffraction. In R Wenk (ed.) *Neutron Scattering in Earth Sciences*, Mineralogical Society of America: Washington DC, Reviews in Mineralogy and Geochemistry, vol. 63
- [24] Stocksdale MG, Berg MAG, Slebodnick C (2007) *S*-(2,2-Dimethyl-5-oxo-[1,3]dioxolan-4-yl)acetic acid. *Acta Crystallographica, Section C: Crystal Structure Communications* submitted.
- [25] Sugandhi EW, Macri RV, Williams AA, Kite BL, Slebodnick C, Falkinham JO, Esker AR, Gandour RD (2007) Synthesis, determination of critical micelle concentrations, and antimycobacterial properties of homologous, dendritic amphiphiles. Probing intrinsic activity and the "cut-off" effect. *Journal of Medicinal Chemistry* submitted.
- [26] Vanpeteghem CB, Angel RJ, Ross NL, Jacobsen SD, Litasov KD, Ohtani E

- (2006) Al, Fe substitution in MgSiO₃ perovskite structure: a single X-ray diffraction study. *Physics of Earth and Planetary Interiors* 155:96-103.
- [27] Vanpeteghem CB, Zhao J, Angel RJ, Ross NL, Bolfan-Casanova N (2006) Crystal structure and equation of state of MgSiO₃ perovskite. *Geophysical Research Letters* 33:L03306, doi:10.1029/2005GL024955.
- [28] Wang GB, Zhu HF, Fan JA, Slobodnick C, Yee GT (2006) Coordination complexes with cis-TCNE radical anion ligands. Models of M[TCNE]₂ magnets. *Inorganic Chemistry* 45:1406-1408.
- [29] Zhao J, Ross NL, Angel RJ (2006) Structural evolutions of perovskites under high pressure and high temperature (in chinese). *Wuli (Physics)* 35:461-465.
- [30] Zhao J, Ross NL, Angel RJ (2006) Estimation of polyhedral compressibility and tilting in GdFeO₃-type perovskites through compression of unit-cell axes. *Acta Crystallographica B* 62:431-439.