BROOKHAVEN NATIONAL LABORATORY

NEWSLETTER

national synchrotron light source

November 2003

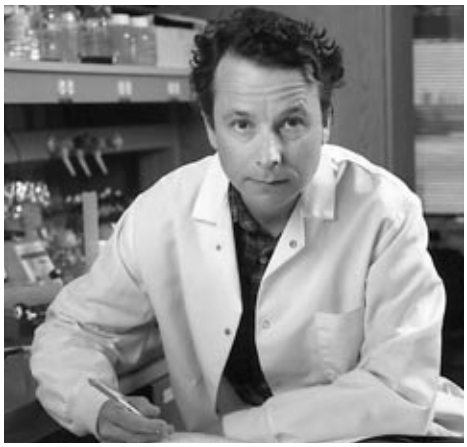
2003 Nobel Prize in Chemistry Awarded to NSLS User Roderick MacKinnon

Karen McNulty Walsh, BNL Media and Communications Office

Roderick MacKinnon, M.D., a frequent NSLS user, shares this year's Nobel Prize in Chemistry for work explaining how a class of proteins helps to generate nerve impulses -- the electrical activity that underlies all movement, sensation and perhaps even thought. The work leading to the prize was done primarily at the Cornell High Energy Synchrotron Source (CHESS) and the NSLS. "That has been a very important resource," MacKinnon told Newsday. "Without the synchrotrons, none of the work I do would be possible."

The proteins, called ion channels, are tiny pores that stud the surface of nearly all living cells. These channels allow the passage of potassium, calcium, sodium and chloride molecules called ions. Rapid-fire opening and closing of these channels releases ions, moving electrical impulses from the brain in a wave to their destination in the body.

Starting in 1998, after 10 years studying the biophysics of ion channels, MacKinnon and his research team published the very first potassium channel structure, which revealed the way that positively charged potassium ions flow easily through a protein's pore, spanning the cell membrane. In the five years following, the Rockefeller scientists have revealed the inner workings of sodium and potassium channels, or the whys and hows of ion movement through a cell's membrane. This series of structural solutions, determined by x-ray crystallography, are high-resolution molecular-level "snapshots" of ion channels that literally show the scientific community how electrical signaling occurs.



Rod MacKinnon

These structures not only portray an elusive ion channel structurally and mechanistically, but also bring history full circle by showing, for the first time, the natural molecular mechanism that underlies the theory of the action potential demonstrated in a mathematical formulation by Alan Hodgkin and Andrew Huxley in 1952. MacKinnon and colleagues have eclipsed their own already formidable contributions to the field of ion channel research — contributions that widen a foot trail into an avenue for an entire new area of medical study on ion channel diseases.

MacKinnon, a biophysicist and self-taught x-ray crystallographer, is a professor at Rockefeller University and an investigator at the Howard Hughes Medical Institute. He shares this year's chemistry Nobel with Peter Agre, M.D., of Johns Hopkins University School of Medicine.

Photo courtesy of Robert Rathe for the Howard Hughes Medical Institute, ©2003.

INSIDE THIS EDITION

Chairman's Introduction 2
Science Highlight 3
UEC News 4
Awards 5
Safety 6
Focus On 7
X-Ray Long Range Schedule 8
VUV-IR Long Range Schedule 9
Science Highlight 10
Ring Status 12
Facility Report 13
Events 14

Letter from the Chairman

Steve Dierker, NSLS Chairman

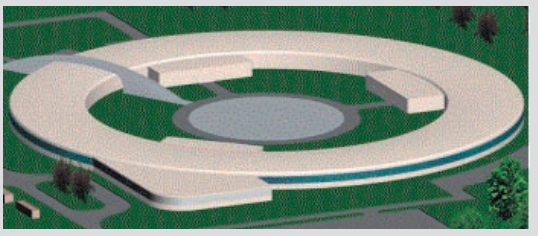
The NSLS and its user community continue to thrive and have a well-deserved reputation for very high scientific productivity and impact. A recent example is the co-awarding of this year's Nobel Prize in Chemistry to NSLS user Roderick MacKinnon for explaining the structure and function of ion channels, based in substantial measure on measurements carried out at NSLS. We strive, in partnership with our users, to provide state-of-the-art facilities to enable the best science to be done at NSLS. Space permits mention of only a few examples: a new undulator based protein crystallography beamline, X29, will become operational in 2004 through a partnership between AECOM, BNL's Biology Department, and the NSLS; a new undulator based x-ray micro-diffraction beamline, X13B, recently funded by DOE, will also become operational in 2004; a new high-throughput curved photon sensitive detector developed by NSLS is enabling users to conduct time-resolved and high-throughput experiments.

In the 21 years since NSLS began operations in 1982, we have also made extraordinary improvements to the accelerator systems, e.g., improving the x-ray brightness by a factor of 100,000. However, we have now reached the inescapable limits imposed by the lattice design and circumference, and significant

gains from the existing machines are no longer possible. To meet the urgent needs of the large user community who demand very high average brightness and flux with exquisite stability in order to tackle the increasingly difficult 'grand challenge' scientific problems, we are proposing to DOE that the NSLS be replaced with a new state-of-the-art medium energy storage ring, called NSLS-II. By taking advantage of the latest developments in storage ring technology, we intend to deliver world-leading performance that will set a new standard.

Over the summer, many of you worked to organize and participate in a series of focused workshops that explored the scientific opportunities that would be enabled by NSLS-II. From these ideas and contributions, a proposal articulating the scientific need for, the requirements to be met by and the pre-conceptual design of NSLS-II has been developed for submission to DOE. BNL has proposed that the design and engineering of NSLS-II begin in 2005, construction in 2008 and operations in 2012. While submission of the proposal is a tremendous milestone, much remains to be done!

It is vital that the user community and other stakeholders continue to provide input and feedback on NSLS-II. To facilitate this, we will host a major workshop for NSLS-II at BNL this coming December 12 (see insert for more information). We have assembled an exciting program, including perspectives by representatives from Congress, DOE, New York State and BNL, plenary lectures by distinguished scientists, breakout sessions to provide participants an opportunity for input on the design and direction of NSLS-II, its beamline characteristics, and instrumentation concepts, and a poster session to highlight your exciting current science and ideas for future science enabled by NSLS-II. I strongly urge you to attend, to show your support for this essential project and to voice your needs and ideas, to be sure NSLS-II will enable outstanding science for decades to come.



NSLS-II Workshop

December 12, 2003

For Agenda, Poster Session and Registration information, go to:

http://www.nsls2.bnl.gov/newsroom/workshops/2003/NSLS-II/

Very Small But Useful Anomalous Signal of Sulfurs

Udupi Ramagopal[1], Miroslawa Dauter[2] and Zbigniew Dauter[1]

[1]Synchrotron Radiation Research Section, National Cancer Institute; [2]SAIC-Frederick, Inc., Basic Research Program

The recent advancements in X-ray data acquisition techniques and phasing algorithms made possible the successful use of a very small anomalous diffraction signal for solving crystal structures of macromolecules. Two crystal structures, the 44 kDa glucose isomerase containing 9 sulfurs and 33 kDa xylanase containing 5 sulfurs, have been solved from a single-wavelength anomalous dataset. These two enzymes contain less sulfur than most proteins in the bacterial or eukariotic proteomes, providing the Bijvoet ratio of about 0.6%. The S-SAD approach relies on the anomalous signal of sulfur, naturally occurring in proteins, and alleviates all need for sample derivatization. It may therefore be applicable to all protein crystals and able to provide accurate diffraction data.

The anomalous scattering contribution of sulfur at 1.54 Å wavelength is 0.56 electrons, rising to 0.70 electrons at 1.74 Å and to 0.90 e at 2.0 Å. The practical use of a small anomalous signal for phasing requires that the X-ray data be measured very accurately. The first successful attempts of phasing macromolecular structures based on the anomalous scattering signal of sulfur date from the solution of the structure of crambin[1], with 5 sulfurs in 45 amino acids, providing the Bijvoet ratio $<\Delta F^{anom}/F>$ of 1.5%. From theoretical simulations[2], concluded that a signal as small as 0.6% may be sufficient for the successful structure solution.

We collected X-ray data on crystals of two proteins, glucose isomerase with 9 sulfurs in 388 amino acids using 1.54 Å wavelength at beamline X9B to 2.0 Å resolution, and xylanase with 5 sulfurs in 302 residues using 1.74 Å wavelength at beamline X9A to 1.63 Å resolution. The high redundancy of measurements ensured very high accuracy of the estimated intensities. The amount of the anomalous signal in both data sets was less than 0.7 %.

All sulfur sites in both proteins could be easily located from anomalous differences by the direct methods program SHELXD[3] with data truncated to different resolution limits, including as low as 3.0 Å. The coordinates of sulfur sites were subsequently used for phase estimation by the single-wavelength anomalous diffraction (SAD) approach.

Initial SAD estimation of phases and their subsequent improvement for glucose isomerase was executed by programs MLPHARE and DM from the CCP4 suite[4].

Zbigniew Dauter

The resulting electron density was easily interpretable and an almost complete atomic model of the protein was built automatically by wARP[5]. The phases for xylanase were estimated by SHELXE[6] and wARP automatically constructed the complete protein consisting of 302 amino acid residues.

If the anomalous signal weaker than 1% is to be used for the estimation of phases, the errors in the estimation of intensities cannot on average exceed 2% (since $\sigma I/I \sim 2\sigma F/F$) and in practice should be considerably lower. Such a level of accuracy can be achieved only at well-performing data collection facilities with crystals diffracting strongly enough and by averaging multiple measurements of the same or symmetry-equivalent intensities. Any X-ray beam instabilities, crystal misalignment, bad beam shutter synchronization, deficient detector calibration, etc., will lead to systematic errors, precluding the appearance of such a sub-percent amount of anomalous signal.

The results presented here prove that even a very weak, below 1%, anomalous signal of sulfur, naturally occurring in proteins, can be successfully used for solving crystal structures. They also show that the macromolecular data collection facilities at NSLS beamlines are able to provide X-ray diffraction data of unprecedented accuracy.

*For more details of this work see: U.A. Ramagopal, M. Dauter and Z. Dauter, "Phasing on Anomalous Signal of Sulfurs: What is the Limit ?" Acta Crystallogr., **D59**, 1020-1027, (2003).*

References:

[1] Hendrickson, et al. (1981). *Nature* **290**, 107-113.

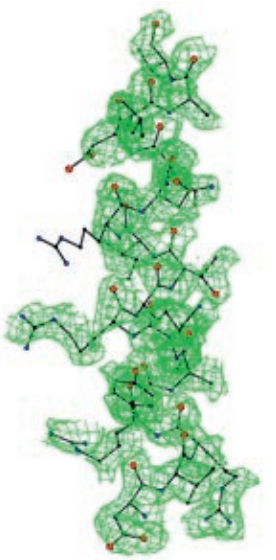
[2] Wang (1985). *Methods Enzymol.* **115**, 90-112.

[3] Schneider, et al. (2002). *Acta Crystallogr.* **D58**, 1772.

[4] CCP4 (1994). *Acta Crystallogr.* **D50**, 760-763.

[5] Perrakis, et al. (1999). *Nat. Struct. Biol.* **6**, 458-463.

[6] Sheldrick (2003). *Z. Kristallogr.* **217**, 644-650.



A helical region of the electron density maps after density modification for glucose isomerase at 2.0 Å, contoured at 1 σ level.

Notes from the UEC

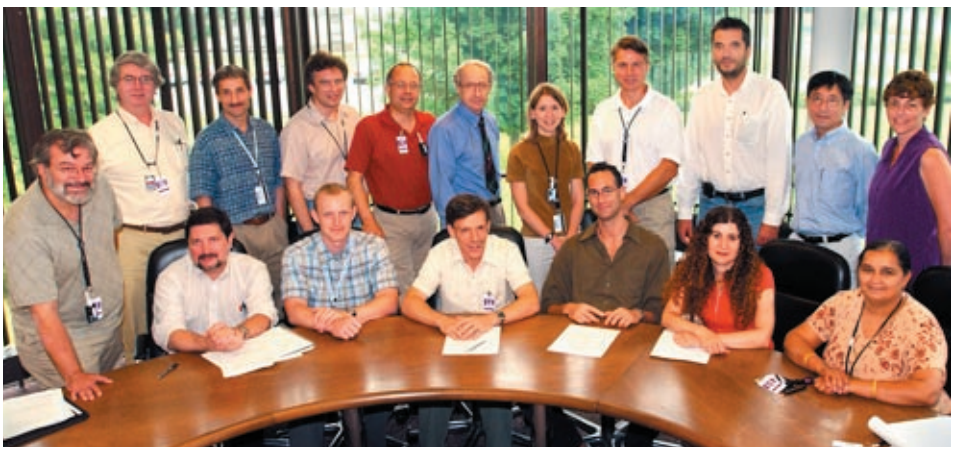
Tony Lanzirotti, Users' Executive Committee Chair
University of Chicago

First, on behalf of all the users at the NSLS, I congratulate Prof. Roderick MacKinnon from Rockefeller University on winning the 2003 Novel Prize in Chemistry. A portion of Prof. MacKinnon's prize-winning research was conducted at the NSLS.

In my last Users Update I wrote about many of the changes that you as users would be seeing at the NSLS over the next few months and one of the topics I focused on was user safety. You know, it's great when we get some 'good' safety news, rather than something about somebody poking their eye out. As I'm sure most of you are aware of by now, as of October 1st most short-term users at the NSLS will not need to wear a thermoluminescent dosimeter to work on the experimental floor. Not only is this an added convenience to the user, but this is a big reduction in the burden on the NSLS safety staff. This would never have happened if not for the NSLS safety staff's diligent efforts over the past 4½ years to demonstrate to DOE that the radiological exposure to users on the experimental floor is negligible, well below what DOE requires for personal monitoring. As I said, this is a great example of how, in my opinion, the attention to safety at the NSLS has paid off for users. On the part of the UEC I'd like to warmly thank the NSLS safety staff for their efforts on this. Remember though, that all personnel on the floor are still required to have appropriate NSLS facility specific and radiological training. Keep you training up to date!

The other topic I want to discuss in this issue is a push that the UEC, in conjunction with user organizations at other DOE facilities, is making to try and raise awareness within DOE and the federal government about issues important to you, the user. It's been clear to all of us who act as your representatives in these organizations that we have more clout as a united group than we do individually. One effort we're making is organizing a joint meeting of User Facility Administrators and User Executive Committee Chairs to discuss the policies for non-US citizen access into the US and the Foreign Visits & Assignments Program for national laboratories. We're going to have discussions with representatives from the Office of Security, Department of State, Office of Science and Technology Policy and the American Physical Society. I'm sure many of you have horror stories about how confusion regarding the existing policy has impacted your experiments while visa questions and notification requirements were being resolved. I also have serious concerns about the burden that has been placed on our User Administration Office. This will be a great opportunity for us to get these issues some attention.

The UEC continues to be active on your behalf to raise the level of awareness within the federal government about the importance of funding for the continued success of research at the NSLS, such as the recent Nobel Prize-winning work of Prof. Roderick MacKinnon. Over the last few years we have lobbied members of Congress on your behalf and occasionally we come to you for help. This is another one of those times. In a multi user-facility effort, coordinated by the chair of the user organization at the Advanced Photon Source, Steve Durbin, in collaboration with the Office of Public Affairs at the American Physical Society, a letter-writing campaign has been initiated. As users of DOE-BES facilities,



UEC Members and SPiG Representatives (standing from left); Tony Lanzirotti (U. of Chicago); Jerry Delaney (Rutgers U.); Richard Reeder (Stony Brook U.); Anatoly Frenkel (Yeshiva U.); Peter Stephens (Stony Brook U.); John Sutherland (BNL, Biology); Lisa Miller (BNL, NSLS); Simon Bare (UOP LLC); Fred Dyda (NIH); Chi-Chang Kao (BNL, NSLS); Mary Anne Corwin (BNL, NSLS), (sitting from left); Larry Carr (BNL, NSLS); Aaron Celestian (Stony Brook U.); Daniel Fischer (NIST); Larry Shapiro (Columbia U.); Leemor Joshua-Tor (Cold Spring Harbor Laboratory); and Kumi Pandya (North Carolina State U.). Missing from photo: Vivian Stojanoff (BNL, NSLS); Mahbub Khandaker (Thomas Jefferson National Laboratory); Michael Dudley (Stony Brook U.); Dean Hesterberg (North Carolina State U.).

you are in the ideal position to tell both Congress and the Administration about the importance of the DOE Office of Science and to help reverse a decade-long decline of the DOE SC budget. Congress is now determining the FY04 budget for the Office of Science. The House proposed larger increases for the Office of Science than did the Senate Appropriations Committee. Soon, the Senate and House will negotiate a compromise bill, making this an excellent time to weigh in with your opinion. Meanwhile, the Administration is working on the FY05 budget and is considering a significant physical science, engineering and computing initiative for FY05. These are important moments; please write to both now! Everything has been prepared for you; the whole process will take 3 minutes! Template letters have been written but feel free to edit them to suit your personal point of view. Using the convenient webpage link below, your letters can be sent electronically within minutes. Click here: http://www.congressweb.com/cweb4/index.cfm?orgcode=APSPA&hotissue=13.

NSLS UEC Website:

http://www.nslsuec.org

for information on all ongoing Users' Executive Committee activities

AWARDS

NSLS Physicist Wins 2003 Free Electron Laser Prize

Diane Greenberg, BNL Media and Communications Office

Li-Hua Yu, a physicist at the NSLS, has won the 2003 Free Electron Laser (FEL) Prize sponsored by the 25th International Free Electron Laser Conference. Yu received the award, which consists of $3,000, a certificate and a plaque, at the FEL conference held this year in Tsukuba, Japan.

Yu's award was given "in recognition of his outstanding contributions to FEL science and technology." Over the last 20 years, Yu and colleagues from Brookhaven contributed significantly in developing two types of lasers that are important for scientific investigations: the self-amplified spontaneous emission free electron laser (SASE FEL) and the high gain harmonic generation free electron laser (HGHG FEL).

In the SASE process, the light the laser emits for experiments starts from noise, or random signals. In contrast, in the HGHG process, the output light starts from fast-moving electrons interacting with a seeding laser that shifts the light to a higher frequency and makes it significantly more coherent, meaning electrons move in a coordinated way to emit light. The intense light of the HGHG FEL reveals the fine details of atomic interactions inside materials and the very fast motions of molecules in chemical reactions, all with an unsurpassed precision.

Yu explained, "The HGHG FEL combines the intensity and coherence of a laser with the broad spectrum of light available in a synchrotron. The invention of the laser provided a revolutionary source of coherent light that created many new fields of scientific research. The development of the HGHG FEL extends the reach of lasers to much shorter wavelengths, thus opening new research opportunities."

Yu continued, "I am very happy to receive this award, and I am grateful for Brookhaven Lab's support and the excellent team who worked with me to make the HGHG FEL at Brookhaven the first and only one of its kind in the world."

At Brookhaven's Accelerator Test Facility in 1999, Brookhaven scientists, in collaboration with Argonne National Laboratory researchers, verified the theoretical foundation of the HGHG FEL operating in the infrared region of the light spectrum. In 2002, the technique was further developed to enable the HGHG FEL at Brookhaven to produce shorter wavelength light in the deep ultraviolet spectral region (DUV-FEL). This enabled researchers to perform new chemistry experiments.

The HGHG FEL may be a complementary research tool to synchrotrons around the world, including the NSLS.

Brookhaven's Laboratory Directed Research and Development Program, the U.S. Naval Research Laboratory and the U.S. Air Force funded Yu's research on the DUV-FEL.

Li-Hua Yu earned his undergraduate degree from Jilin University in China in 1970. He earned both an M.S. and Ph.D. in physics from Stony Brook University in 1980 and 1984, respectively. In 1984, he joined Brookhaven Lab as a research associate and rose through the ranks to become a senior physicist in 2000. With a team of eight scientists and engineers from Brookhaven, Yu won an R&D 100 Award from *R&D Magazine* in 1989 for inventing the Real-Time Harmonic Closed-Orbit Feedback System, which stabilizes the orbit of electron beams in synchrotrons.

Changes in TLD Requirements and Laser Safety Review

Bob Casey, Associate Chair for ESH/Q

Changes in TLD Requirements

On Oct. 1, 2003 the "Controlled Area" postings on all doors to the experimental floor at the NSLS were revised to remove the requirement for TLDs for access. As a result, most users will not need to wear a TLD to work on the experimental floor. There are some users (see below) who are still expected to wear a TLD.

Everyone should be aware that all other access requirements remain in effect. It is particularly important to remember that **all** personnel on the floor must have appropriate NSLS facility-specific and radiological training unless escorted as described in the NSLS escort policy. There are no exceptions to this requirement.

This new access policy will reduce the number of persons wearing TLDs from approximately 2000 per year to 300. However, it is not intended to limit dosimetry for personnel with special needs or concerns. Please contact NSLS ESH staff if you desire dosimetry while at the NSLS or would like to discuss this policy further.

Laser Safety Under Review at BNL

Some of our users operate lasers while conducting research at the NSLS. Some of our beamlines use low-power lasers for alignment or other similar activities, and a few beamlines utilize higher power lasers for various purposes. Recently, a researcher in another facility at BNL suffered a significant eye injury when he was accidentally exposed to a pulsed high power laser beam. As the result of the accident, all lasers at BNL were shutdown to permit a detailed review of operating procedures and training. In addition, all laser users participated in discussions with senior management regarding the incident and laser safety.

At the NSLS, all of our high-power lasers (Class 3b/4) should be back in operation during the fall. However, as I write this article, only 1 high-power laser within the NSLS facility has been restarted. All of our low-power lasers (Class 2/3a) are back in service.

Although the accident did not take place at the NSLS, it has resulted in extensive discussion of our laser safety program. There are some important points that our users should remember:

- The use of every laser must be reviewed and approved by the NSLS Safety Officer. When you submit your Safety Approval Form, be sure to identify any lasers that you intend to bring or use while at the NSLS.

- Review of low-power (Class 2/3a) lasers is straightforward and is normally easily accomplished.

- Review of high-power lasers (Class 3b/4) is considerably more involved and will require the development of operating procedures and a training program. Any user who wishes to bring a high-power laser should anticipate extended reviews and allow time for the process.

- If you plan to use a class 3b/4 laser that is installed at one of the NSLS beam lines, you are required to complete a medical eye exam, BNL laser training and laser-specific training developed by the responsible laser owner at the NSLS.

Who will still need to wear a TLD?

- NSLS and beamline staff and long-term users routinely working on the floor throughout the year are required to wear a TLD.

- There are several special cases that will require a person working on the floor to wear a TLD:
  - Work with radioactive materials (including sealed sources) or other work as required through the safety approval process
  - Work in a location on the experimental floor posted as requiring a TLD (there are few such locations at the NSLS)
  - Women with a declared pregnancy working on the experimental floor
  - Minors (i.e. < 18 years old) working on the experimental floor
  - Personnel escorting minors on a tour of the experimental floor

The NSLS Accelerator Division

James B. Murphy, Associate Chair for Accelerators

NSLS Accelerator Division (AD)

James Murphy, Division Head

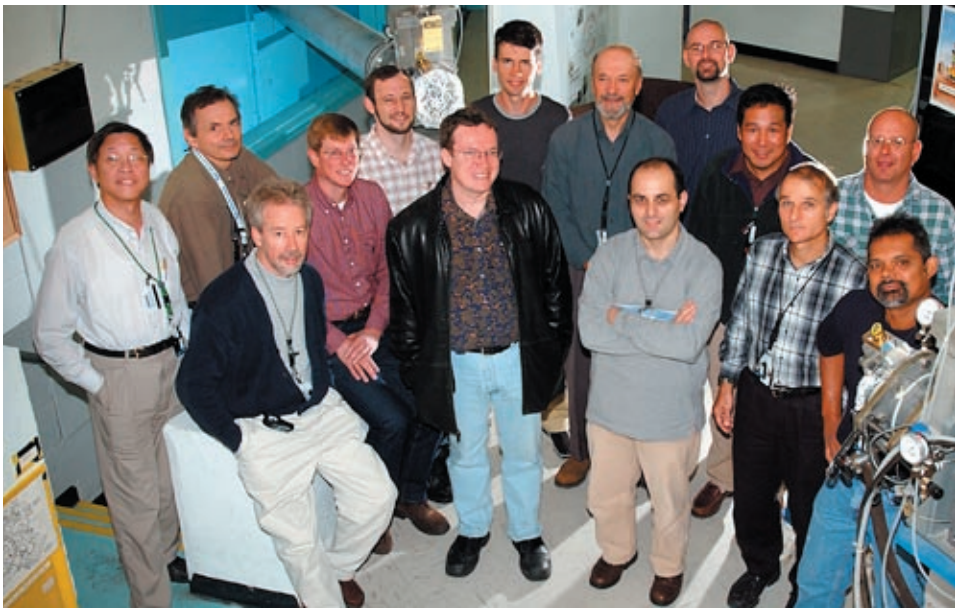
The NSLS Accelerator Division (AD) is the home of the accelerator physics staff. The AD has responsibility for the "quality and improvement of the electron beam" in all the accelerators in the NSLS complex as well as the R&D for future light sources. In particular, the AD scientific staff is heavily involved in the R&D work for the NSLS-II machine design.

Storage Ring & Insertion Device Section

Boris Podobedov, Section Head

The Storage Ring & Insertion Device Section, with its staff of 4 scientists, 1 engineer and 1 technician is responsible for the beam quality in the X-ray and VUV rings and for the operations of the Magnet Measurement Lab. The staff's responsibilities on the rings include providing support in case of beam physics related problems, participating in commissioning, maintaining performance histories, initiating machine improvements, running ring studies program and interfacing with the NSLS User Meetings. These duties are mainly handled by Stephen Kramer and Boris Podobedov, who are the designated machine physicists for the X-ray and VUV rings, respectively. Theoretical support is provided by Jiunn-Ming Wang.

The Magnet Measurement Lab, headed by George Rakowsky and assisted by David Harder (electrical engineering) and mechanical technician Michael Lehecka, provides quality assurance, acceptance testing and calibration of magnets prior to placement in the rings and/or the Deep Ultra Violet Free Electron Laser (DUV-FEL). The Lab also designs and constructs state-of-the-art magnets and insertion devices, such as a very successful NSLS mini-gap undulator, presently operational at beamline X13 and another one coming on line at beamline X29.



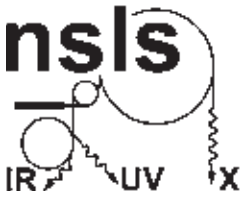
From left: (back) Li-Hua Yu, Steve Kramer, James Rose, Timur Shaften, Boris Podobedov, George Rakowsky, Henrik Loos, Xijie Wang, and Michael Lehecka, (front) James Murphy, Brian Sheehy, Adnan Doyuran, Dave Harder, and Boyzie Singh. Missing from photo: Jiunn-Ming Wang, Zilu Wu and Joe Greco.

Linac Section

Xijie Wang, Section Head

The Linac Section, with its staff of 4 scientists, 3 postdocs and 2 technicians, is responsible for maintaining and improving the NSLS accelerator injection system beam quality and operating the DUV-FEL. Jim Rose, the machine physicist for the NSLS Booster and Linac, directed the recent successful completion of the upgrade of the low level RF system and the high voltage modulators of the NSLS injection linac.

The DUV-FEL is a platform for single-pass high-gain FEL R&D and applications. The main focus at the DUV-FEL is to develop and explore laser-seeded high-gain harmonic generation (HGHG) FEL technology, both theoretically and experimentally. This work is supported by our staff scientists, Li Hua Yu and Timur Shaftan, and by our talented postdoctoral research associates, Adnan Doyuran, Henrik Loos and Zilu Wu. In addition to ensuring first class operation of the DUV-FEL, our two technicians, Boyzie Singh and Joseph Greco, provide support for the NSLS user community to explore the potential of the DUV-FEL for chemical science applications. The saturation of the DUV-FEL and completion of the first chemistry user experiments in the last year have been a tremendous success for the entire division. In partial recognition of these successes, Li Hua Yu was awarded the 2003 International FEL Prize for his seminal work in the field of FELs.



X-Ray Ring Long Range Schedule

January 2004

Sun	Mon	Tue	Wed	Thu	Fri	Sat
				1 Lab Holiday 0000 Maint.	**2** 0000 Maint.	**3** 0000 Maint.
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February 2004

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29 0000 Ops.						

March 2004

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April 2004

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				1 0000 Ops.	**2** 0000 Ops.	**3** 0000 Ops.
4 0000 Ops.	**5** 0000 Ops. 1200 Studies	**6** 0000 Studies 0600 Maint.	**7** 0000 Maint.	**8** 0000 Studies 1200 Ops.	**9** 0000 Ops.	**10** 0000 Ops.
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VUV Ring Long Range Schedule

January 2004

Sun	Mon	Tue	Wed	Thu	Fri	Sat
				1 Lab Holiday 0000 Cond.	**2** 0000 Cond.	**3** 0000 Cond.
4 0000 Cond.	**5** 0000 Cond.	**6** 0000 Cond.	**7** 0000 Cond.	**8** 0000 Cond.	**9** 0000 Cond.	**10** 0000 Cond./Ops.
11 0000 Cond./Ops.	**12** 0000 Ops.	**13** 0000 Ops.	**14** 0000 Ops.	**15** 0000 Ops.	**16** 0000 Ops.	**17** 0000 Ops.
18 0000 Ops.	**19 Lab Holiday** 0000 Ops. 1800 Timing	**20** 0000 Ops.	**21** 0000 Ops.	**22** 0000 Ops.	**23** 0000 Ops. 1800 Studies	**24** 0000 Ops.
25 0000 Ops.	**26** 0000 Ops.	**27** 0000 Studies	**28** 0000 Studies	**29** 0000 Ops.	**30** 0000 Ops.	**31** 0000 Ops.

February 2004

Sun	Mon	Tue	Wed	Thu	Fri	Sat
1 0000 Ops.	**2** 0000 Ops. 1800 Timing	**3** 0000 Ops.	**4** 0000 Ops.	**5** 0000 Ops.	**6** 0000 Ops. 1800 Studies	**7** 0000 Ops.
8 0000 Ops. 1800 Studies	**9** 0600 Maint.	**10** 0000 Maint.	**11** 0000 Ops.	**12** 0000 Ops.	**13** 0000 Ops. 1800 Timing	**14** 0000 Ops.
15 0000 Ops.	**16 Lab Holiday** 0000 Ops. 1800 Timing	**17** 0000 Ops.	**18** 0000 Ops.	**19** 0000 Ops.	**20** 0000 Ops. 1800 Studies	**21** 0000 Ops.
22 0000 Ops.	**23** 0000 Ops.	**24** 0000 Studies	**25** 0000 Studies	**26** 0000 Ops.	**27** 0000 Ops.	**28** 0000 Ops.
29 0000 Ops.						

March 2004

Sun	Mon	Tue	Wed	Thu	Fri	Sat
	1 0000 Ops. 1800 Timing	**2** 0000 Ops.	**3** 0000 Ops.	**4** 0000 Ops.	**5** 0000 Ops. 1800 Studies	**6** 0000 Ops.
7 0000 Ops. 1800 Studies	**8** 0600 Maint.	**9** 0000 Maint.	**10** 0000 Ops.	**11** 0000 Ops.	**12** 0000 Ops. 1800 Timing	**13** 0000 Ops.
14 0000 Ops.	**15** 0000 Ops. 1800 Timing	**16** 0000 Ops.	**17** 0000 Ops.	**18** 0000 Ops.	**19** 0000 Ops. 1800 Studies	**20** 0000 Ops.
21 0000 Ops.	**22** 0000 Ops.	**23** 0000 Studies	**24** 0000 Studies	**25** 0000 Ops.	**26** 0000 Ops.	**27** 0000 Ops.
28 0000 Ops.	**29** 0000 Ops. 1800 Timing	**30** 0000 Ops.	**31** 0000 Ops.			

April 2004

Sun	Mon	Tue	Wed	Thu	Fri	Sat
				1 0000 Ops.	**2** 0000 Ops. 1800 Studies	**3** 0000 Ops.
4 0000 Ops. 1800 Studies	**5** 0600 Maint.	**6** 0000 Maint.	**7** 0000 Ops.	**8** 0000 Ops.	**9** 0000 Ops. 1800 Timing	**10** 0000 Ops.
11 0000 Ops.	**12** 0000 Ops. 1800 Timing	**13** 0000 Ops.	**14** 0000 Ops.	**15** 0000 Ops.	**16** 0000 Ops. 1800 Studies	**17** 0000 Ops.
18 0000 Ops.	**19** 0000 Ops.	**20** 0000 Studies	**21** 0000 Studies	**22** 0000 Ops.	**23** 0000 Ops.	**24** 0000 Ops.
25 0000 Ops.	**26** 0000 Ops. 1800 Timing	**27** 0000 Ops.	**28** 0000 Ops.	**29** 0000 Ops.	**30** 0000 Ops. 1800 Studies	

Investigating a Surface Science Mystery: The Case of the Disappearing Monolayer

Kevin S. Schneider,[1] Thomas M. Owens,[1] Daniel R. Fosnacht,[1] Bradford G. Orr,[2,3] & Mark M. Banaszak Holl[1,3]

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A recent X-ray photoemission spectroscopy (XPS) and scanning tunneling microscopy (STM) investigation of an alkylsilane-based monolayer has yielded intriguing chemical and physical phenomena. In particular, oxidation of an octylsilane ($C_8H_{17}SiH_3$) monolayer chemisorbed to Au(111) via ambient atmosphere exposure yields two surprising results. First, the Au(111)-23×√3 surface reconstruction typical of a clean gold surface spontaneously regenerates underneath the oxidized (alkylsiloxane) monolayer. Furthermore, the physisorbed alkylsiloxane monolayer is completely transparent to STM imaging.

Frequently, the STM image contrast mechanism of organized organics—such as alkanethiols—on Au(111) is explained as a consequence of hydrocarbon chain crystallization and/or variations in chain angle or orientation. Similarly, subtle variations in alkyl chain angle or orientation may yield differing apparent heights of alkylsilane monolayer features as observed by STM. However, a recent XPS and STM investigation of an alkylsilane monolayer prior to and following oxidation suggests the alkyl chains are "transparent" to STM imaging and impart a negligible contrast contribution to the STM images.

A STM image of clean Au(111) displays the parallel striped features intrinsic to the 23×√3 surface reconstruction (**Figure 1a**). Monolayer formation via exposure of Au(111)-23×√3 to a saturating gaseous pressure of octylsilane ($C_8H_{17}SiH_3$) in ultrahigh vacuum (UHV) yields a complex pattern of interwoven, sinuous ridge features containing numerous interstitial Au islands 20 – 40 Å in diameter (**Figure 1b**). The presence and quantity of the Au islands (~7% area coverage in **Figure 1b**) indicates the underlying 23×√3 surface reconstruction has fully relaxed to the unreconstructed Au(111)-1×1 phase.

Oxidation of the octylsilane monolayer via ambient atmosphere exposure results in the disappearance of monolayer features from the STM image (**Figure 1c**). The resulting substrate terraces are indistinguishable from clean Au(111)-23×√3 (having identical lateral and vertical dimensions) under the imaging conditions employed. STM image features of the exposed monolayer do not vary with changes in tunneling current (0.01 – 2 nA) or applied sample bias (± 2 V). However, exposure to additional octylsilane does not regenerate the image shown in **Figure 1b**. Instead, an image identical to clean Au(111)-23×√3 remains (**Figure 1d**). Therefore, the "clean gold" surface displayed in **Figure 1c** does not have the identical chemical properties of the authentic clean gold surface illustrated in **Figure 1a**. On the basis of STM data alone, this set of results was mysterious!

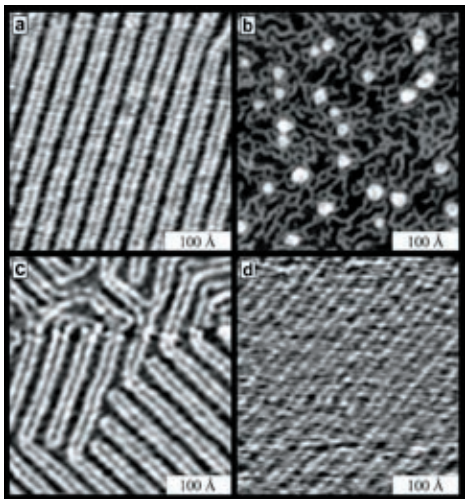

Figure 1. *UHV-STM images of the same Au(111) sample (different sample areas), following successive experimental steps. All images are 35 nm × 35 nm. (a) Clean Au(111) 23×√3. (b) Chemisorbed octylsilane monolayer formed on (a) following exposure to 50 L (L = langmuir = 1 × 10^{-6} torr·s) gaseous octylsilane in UHV. (c) Oxidized physisorbed alkylsiloxane monolayer formed following exposure of (b) to ambient atmosphere for 15 minutes. (d) Oxidized physisorbed alkylsiloxane monolayer in (c) following exposure to 50 L gaseous octylsilane in UHV.*

Authors (top photo, from left) Kevin S. Schneider and Bradford G. Orr, (bottom photo, from left) Thomas M. Owens, Mark M. Banaszak Holl and Daniel R. Fosnacht.

Direct chemical analysis of oxidized sample surfaces have been performed using soft X-ray photoemission spectroscopy (SXPS) at beamline U8B at the National Synchrotron Light Source at Brookhaven

National Laboratory. Beamline U8B is perfectly suited to obtaining the high-resolution Si 2p core-level needed for this study as well as the valence band region. Note that, due to the presence of an Au plasmon trailing the Au 4f core level, conventional XPS of the Si 2p core-level for these monolayers provides no information. Following exposure to ambient atmosphere, the Si 2p core level of the unoxidized octylsilane monolayer (**Figure 2a**) shifts by 2.3 eV to higher binding energy and the peak full width at half-maximum (fwhm) increases (**Figure 2b**). The binding energy shift and peak broadening indicate formation of a cross-linked $RSiO_3$ and/or $(ROSiO_{1.5})_n$ network. The C 2s features arising from the octyl chain are retained in the valence band data (-12 to -20 eV), indicating the presence of intact alkyl chains in the oxidized monolayer (**Figure 2d**). In summary, the XPS data indicate that exposure to ambient atmosphere effectively oxidizes the silicon head-groups; however, all of the silicon and alkyl chains are retained within the oxidized monolayer. Thus, the STM image displayed in **Figure 1c** does indeed have the monolayer present, albeit it in an oxidized form.

The combination of SXPS and STM data suggest the original octylsilane monolayer image in **Figure 1b** is the result of significant mixing of Au and Si states. Oxidation of the Si head-groups removes this interaction, causing the underlying substrate to regenerate the Au(111)-23×√3 surface reconstruction, leaving only the Au states to image. In this case, the alkyl chains are "transparent" in both **Figures 1b** and **1c** and it is only the Au-Si interaction that changes upon oxidation. Since monolayer oxidation severs all Au-Si bonds, a completely "transparent" physisorbed alkylsiloxane layer remains on top of the reconstructed Au(111)-23×√3 surface.

*For more details of this work see: Kevin S. Schneider, Thomas M. Owens, Daniel R. Fosnacht, Bradford G. Orr and Mark M. Banaszak Holl, "The Case of the Disappearing Monolayer: Alkylsilane Monolayer Formation, Oxidation, and Subsequent Transparency to Scanning Tunneling Microscopy," ChemPhysChem, **4**, 1111-1114, (2003).*

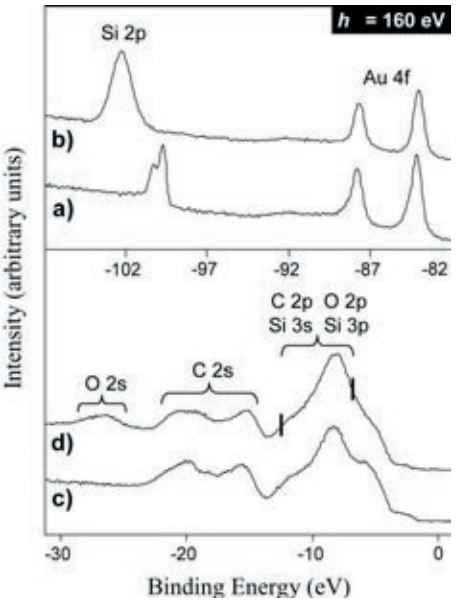

***Figure 2**. Soft X-ray photoemission spectra. (a) Si 2p and Au 4f core level spectrum of an octylsilane monolayer on Au(111). (b) Si 2p and Au 4f core level spectrum of the monolayer in (a), following exposure to ambient atmosphere for 15 minutes. (c) Valence band spectrum of an octlysilane monolayer on Au(111). (d) Valence band spectrum of the octylsilane monolayer in (c) following exposure to ambient atmosphere for 15 minutes.*

Weekly NSLS Activities

TUESDAYS

Bi-Monthly Symposia: 10:30 to 11:30 a.m., Seminar Room See URL below for schedule:
http://www.nsls.bnl.gov/newsroom/events/seminars.htm

WEDNESDAYS

Joint VUV and X-Ray Users' Meeting: 11:30 a.m., Conference Room A. Experimenters and staff meet weekly to decide on any proposed short-term schedule changes, to make announcements and to discuss issues of relevance to operations. To subscribe to the email list for meeting minutes and schedules, follow the instructions at the URL below:
http://www.nsls.bnl.gov/newsroom/events/weekly_meetings.htm

Coffee for Users and Staff: 3:30 p.m., NSLS Lobby. The NSLS hosts a coffee break as an opportunity for users to meet one another and NSLS staff.

FRIDAYS

Student/Postdoc Pizza Get-Together: Every other Thursday, 4:00 p.m, NSLS x-ray ring kitchen (across from vending machines). Funded by the Users' Executive Committee (UEC). All local and visiting students and postdocs are invited to attend.

Friday Lunch Seminars: 12:00 to 1:00 p.m., Seminar Room. Learn about the exciting research being done at the NSLS. Two unannounced, informal, half-hour presentations are made weekly by experimenters. Attendees can bring their own lunch or can place a sandwich order by contacting Lydia Rogers at (631)344-4746 or lrogers@bnl.gov by 10:00 a.m. on Friday. Orders must be paid upon delivery.

NSLS Accelerator Complex Update

Erik Johnson, Associate Chair for Operations and Engineering

Big surprises continued to impact our machine availability during the last quarter of the fiscal year. We had been operating nearer our historic performance, achieving over 99% reliability in July for both VUV and X-ray. Then in August the NSLS, like much of the northeast, was hit by the blackout, which took more than 60 hours out of scheduled operations. In fact, just three rather unusual major events accounted for more than 60% of our down-time. For the year, reliability of VUV remained high at 98%, but the major downtime events brought the overall reliability of X-ray down to 89%.

In looking back over the 2003 performance, we continue to evaluate the more routine types of problems. While they don't account for substantial down-time, they can be extremely annoying, especially for users with short runs. Our goal is to minimize both the number of faults and their impact on operations. In addition, we are looking more carefully at our ability to recover from the types of unusual events we experienced this year.

Looking ahead, preparations for the winter shutdown are proceeding briskly. The completion of the X29 insertion device beamline is the leading activity on X-ray. This includes the completion of the electrical connections for the insertion device itself, implementation of the active interlock system for protecting the vacuum chamber from the high power undulator beam and the completion of the installation of the X29 front end. Some conditioning of the machine will be required, as well as commissioning the active interlock system. Therefore, significant commissioning time is included in the turn-on schedule coming out of the x-ray shutdown. On the VUV ring, the major pacing item is the repair of the U4IR extraction mirror assembly. This delicate operation requires venting a portion of the ring for up to two days, so ample conditioning time has been scheduled.

Installation of the last new RF cavity on the x-ray had been in the original shutdown planning. However, during the testing and preparation of the cavity, a gross vacuum leak developed, which requires rework from the vendor. The failed part (the cavity cover plate) was removed and sent back to the vendor in September. When the repair is completed, testing will resume and the installation of the cavity will be rescheduled. If the cavity cannot be installed during this shutdown, preparations have been made to allow the installation of the second high power circulator to improve the overall RF system stability.

Finally, thanks to all who provided input for the Winter 2004 schedule. Some adjustments were made to satisfy user requests, including uninterrupted operations to coincide with the 2004 RapiData workshop in April. Also, based on suggestions from NSLS staff, the sequence of the maintenance days was altered to facilitate a smoother turn-on of the machines. Don't forget that the Summer 2004 schedule firms up in February. If there are any special scheduling considerations, please let me know by January.

NSLS Fiscal Year 2003 Operations

Month	VUV Ring			X-Ray Ring			Major Events	
	Planned Hours	Reliability	Availability	Planned Hours	Reliability	Availability		Downtime Hours
October	617	99.8%	104.7%	532	96.2%	114.9%		
November	521	98.2%	101.3%	457	96.0%	101.7%		
December	0	-	-	0	-	-	Winter Shutdown	
January	440	99.3%	122.6%	214	16.0%	24.4%	Sextupole Failure	177.8
February	546	99.6%	106.6%	492	96.5%	108.3%		
March	617	99.4%	107.9%	556	99.0%	107.2%		
April	595	97.3%	106.2%	552	83.3%	94.6%	Stripline Leak	35.3
May	338	95.1%	112.3%	256	65.4%	65.4%	Spring Shutdown	
June	597	98.6%	106.2%	535	95.6%	101.8%		
July	515	99.4%	111.4%	510	99.9%	105.3%		
August	615	90.5%	103.5%	547	82.1%	96.3%	Northeast Blackout	63.5
September	592	99.9%	115.0%	495	94.6%	112.0%		
Fiscal 2003	**5993**			**5146**				**276.7**
Delivered	***5871***	98.0%		***4575***	88.9%			
Available	***6498***		108.4%	***5109***		99.3%		

Notes: Delivered Hours are only those accumulated during scheduled operations
Unscheduled operations do not contribute to this total
Available hours are Delivered plus Unscheduled
Underlined numbers fall below NSLS target of 95% Reliability

NSLS Facility Report

Gerry Van Derlaske, NSLS Building Manager

Safety First

With the Laboratory's continuing commitment to working safely, let me remind everyone to be constantly vigilant of their surroundings when performing daily tasks in and around the Laboratory. Do not hesitate to properly report unsafe acts or situations that could escalate into a more serious event if intervention does not take place.

Beamline X29

The Albert Einstein College of Medicine/ BNL Biology/ NSLS joint effort to construct a new beamline at X29 is nearing completion. Lead shielding has been installed and painted. Experimental vacuum chambers are presently surveyed in place. Electrical installation and Personnel Safety interlock systems are currently being installed. Commissioning is expected to begin soon after the Winter 2003 shutdown.

New User Network

A new, unsecured network connection is available for users not wishing to route their connection through the BNL Firewall. Some of the advantages for connection to this network (nslsusers.org) are that it: 1) Does not require full compliance with the BNL Cyber Security policy's; 2) Eliminates the need for conduit exception requests; 3) Allows hosting web servers when using a registered static IP address; 4) Does not prevent use of FTP and Telnet services; 5) Opens VPN's to home institutions without need to open a conduit through BNL firewall; 6) Minimizes the administration requirements for wireless access points (WAPs); 7) NSLS provides network jacks to interested parties.

Access to BNL mail on the Exchange server for computers connected to nslsusers.org can be done remotely via OWA (Outlook Web Access) using a web browser, BNL SSH Gateway (must have a valid account), or via a BNL VPN account.

Contact Alan Levine at (631) 344-4707 or by e-mail at alanl@bnl.gov for additional information.

Roof Repairs

The Plant Engineering Carpenter crew spent numerous hours repairing the NSLS roof. Mechanical Equipment Room-2 (MER2), which has been plagued with leaks of varying degrees, and the incoming power feeders penetrating the roof near the RF penthouse, were both sealed and proof-tested during intense summer downpours. As the roof repair season nears an end for this calendar year, Plant Engineering will continue to address recurring roof leaks within the facility.

New Arrivals

Dr. Christie Nelson, Dr. Elaine Dimasi and Dr. Dario Arena have joined the User Science Division. Heather O'Kula will be working with Ken Koebel in the Budget/ Admin Section.

Bicycles

For those who have signed out a bicycle from the Quality of Life Office and wish to return the bike prior to the winter season and obtain a refund of your deposit, contact Erin Rogers at (631) 344-8481 to make the arrangements.

Identification and Tagging of Equipment

The Department of Energy requires that all non-expendable property at BNL have bar codes or tags to indicate ownership. If your organization does not have tags (logo's, etc.) we will supply blank tags (see sample on bottom).

Tags are available at the NSLS stockroom free of charge. Please obtain tags, fill in your organization in the space provided and apply to all unidentified equipment belonging to your organization. The serial numbers on the blank tags are for your optional use in recordkeeping.

BNL's Supply & Materiel Division will be conducting periodic inspections to ensure proper identification of all equipment.

If you have any questions or need assistance, please call Wendy Morrin at (631) 344-4884.

EXAFS Data Collection and Analysis Short-Course has Another Successful Year

Simon Bare, UOP LLC

A hands-on EXAFS Data Collection and Analysis Course was held July 14-17, 2003 at the NSLS. The course was co-organized by Bruce Ravel (Naval Research Laboratory) and Simon Bare (UOP LLC), with superb administrative support by Lisa Tranquada (SFA, Inc.). Twenty-eight eager participants (graduate students, postdocs and institution and industrial scientists) representing universities, national laboratories, research institutes and industry attended the course. Of these, there were ten new users to the NSLS. The participants had diverse research interests across a broad spectrum of scientific fields (materials science, geological and environmental sciences, catalysis and biology) and attended to learn how XAFS may be applied to their research program.

The four-day course was divided into morning lectures, with two afternoons of hands-on data collection using seven different NSLS spectroscopy beamlines (X9B, X11A, X18B, X19A, X23A2, X23B, and X26A) and two afternoons of data analysis. The instructors on the beamlines were Faisal Alamgir, Wolfgang Caliebe, Scott Calvin, Syed Khalid, Tony Lanzirotti, Nebojsa Marinkovic and Kaumudi Pandya.

The eight morning lectures were: "Introduction to XAFS," given by Matt Newville (University of Chicago), "Basics of sample preparation" by Scott Calvin, "XANES measurement and interpretation" by Simon Bare (UOP LLC), "Detectors and synchrotron radiation" by Peter Siddons (BNL), "Basics of data processing" by Shelly Kelly (Argonne National Laboratory), "Introduction to theory" by John Rehr (University of Washington), "Introduction to analysis" by Anatoly Frenkel (Yeshiva University) and "Applying XAFS into a research program" by Rich Reeder (SUNY Stony Brook). The time allotted for the lectures allowed ample time for stimulating discussion, which often developed.

For the first two days of the course, after attending the morning lectures, the participants were divided into small groups by research discipline to conduct the experimental part of the course. Each student became familiar with beamline operation and collected real XAFS data on representative samples from their own individual research projects. On the last two days, following the morning lectures, the participants learned data analysis techniques using the data they had just collected. The participants also enjoyed ample time for informal discussion over coffee, and in the evenings, over the excellent dinners and drinks that were included in the course fee.

There was a tremendous amount of information disseminated over the four days. All the participants left the course with new friends and armed with the basic tools to apply x-ray absorption spectroscopy to their own research programs.

We plan to offer the course again in 2004 – check the NSLS website for updated information.

The course was sponsored by the NSLS, with support from the Center for Environmental Molecular Science at SUNY Stony Brook.

Participants of the 2003 NSLS EXAFS course

NSLS Summer Sunday Draws a Record-Breaking Crowd

Lisa Miller, Information and Outreach Coordinator

On Sunday August 3, 2003, over 750 visitors toured the NSLS as part of Brookhaven National Laboratory's Summer Sunday tour series. Thirty-five NSLS staff members, students and users volunteered their time for the event, which was organized by NSLS scientist Lisa Miller.

Tours of the NSLS included presentations, demonstrations and hands-on exhibits. At Berkner Hall, visitors watched an introductory video about how a synchrotron works, narrated by NSLS Chairman Steve Dierker. After a short bus ride and tour of the Lab, visitors were dropped off at the NSLS. In the seminar room, NSLS scientists presented an introduction to "Science at the NSLS" by describing the many ways the NSLS is used to study scientific problems that affect everyday life. Improvements in biomedical imaging techniques, drug design, catalytic converters, environmental cleanup and computer storage media were just a few of the topics discussed.

Visitors then toured the NSLS lobby, which was transformed into an exhibit area for numerous light and synchrotron-related demonstrations. Visitors were able to experience "total internal reflection" as a laser beam was guided through a stream of falling water. A display on the principles of a vacuum demonstrated its effect on a ringing bell, a balloon, a feather and a marshmallow. The technique of diffraction was demonstrated using tiny metal grids and compact disks. Visitors had the opportunity to build their own "crystals," using gumdrops, and "see" the synchrotron light (at least the visible part of the spectrum) which was transported to the lobby through a fiber optic. But perhaps one of the all-time favorite features in the NSLS lobby was the view of the experimental floor from the display windows, which continues to amaze visitors year after year.

NSLS scientist, Vivian Stojanoff, shows how much fun it can be to build crystal models out of gumdrops and toothpicks.

NSLS Annual Awards Ceremony and Picnic

Lisa Miller, Information and Outreach Coordinator

On Wednesday, September 17, 2003, the NSLS had its annual Awards Ceremony and Picnic. Despite the impending arrival of Hurricane Isabel, the weather was spectacular and the pig roast was another big success. The picnic was coordinated by Laura Miller and executed by a number of NSLS staff members, including Charlie Nielson, Boyzie Singh, Bob Best, Joe Greco, Paul Humbert, Jim Lacy, Jim Newburgh, John Burke, Gerry Van Derlaske and Bob Kiss, along with Ken Sutter "the pigman" (MSD).

This year, Service Awards were given to 24 NSLS staff members: Al Almasy, Sam Krinsky and Bob Casey (30 years); Roy D'Alsace, Walter De Boer, John Gallagher, Rick Greene, Chris Lanni, Payman Mortazavi, Jack Tallent, Frank Terrano and Gerry VanDerlaske (25 years); Diane Hatton, Steve Hulbert, Jim Murphy and Florin Staicu (20 years); Peter Gross, Alan Levine, Paul Montanez, Pauline Pearson, Eva Rothman, Brian Sheehy, Chris Stelmach and Xijie Wang (10 years).

NSLS Chairman, Steve Dierker, presented the Service and Spotlight Awards at the Annual Picnic.

Spotlight Awards were presented to NSLS staff members for the completion of extraordinary accomplishments that were of significant benefit to the Department, Division or Laboratory. This year's Spotlight Award winners were: (1) Brian Kushner for getting the X-ray ring's digital vertical feedback up and running, (2) Jim Newburgh for outstanding radio frequency cavity installation efforts during the winter 2002 shutdown, (3) Jack Tallent for designing a new beam profile monitor for the X-ray ring, (4) John Burke for completely rebuilding the fire-damaged Pulse Forming Network (PFN) section of the SDL modulator-A and (5) Gary Nintzel for the preparation, dismantling and shipping of the U6 beamline.

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Call for NSLS General User Proposals

For Beam Time in Cycle	Deadline
May-August 2004	Monday, February 2, 2004

General User Proposal and Beam Time Request Forms including instructions can be found at:
http://www.nsls.bnl.gov/users/procedures/proposals/general_user.htm
Proprietary Proposal Forms including instructions can be found at:
http://www.nsls.bnl.gov/users/procedures/proposals/proprietary.htm

Safety Approval Forms

Safety Approval Forms (SAFs) are required for every experiment. Your SAF must be submitted online **at least one week before** your scheduled beam time. To submit, go to:

http://130.199.76.84/safety/default.asp

NSLS User Administration Office

User Information, Registration, and Training:
Phone: (631) 344-user Fax: (631) 344-7206

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For additional information about the NSLS (including this Newsletter in electronic format) see the NSLS Home Page on the World Wide Web at:

http://www.nsls.bnl.gov/