July 1999 NSLS Newsletter

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July 1999 NSLS Newsletter

A Research Resource for Macromolecular Crystallography at the NSLS

R.M. Sweet and M.Capel, BNL-Biology Department

L. Berman, BNL-NSLS

The NSLS is a popular x-ray source for macromolecular crystallography (PX). Seven beamlines are used regularly for PX studies. Some 766 researchers visited the NSLS during last year for these types of measurements. A complete summary of these facilities is available at: **http://www.x12c.nsls.bnl.gov/x12c/nsls_px.html** . Five beamlines have been organized loosely under a new Research Resource funded by a grant from the National Institute of Health (NIH): X8C, X12B, X12C, X25, and X26C.

There are several components to the project. There are five major research and development projects, a number of collaborative projects that extend and apply the R&D projects, and efforts in training and service. We can discuss each of these in turn.

One of the R&D projects is to improve the quality of support for NSLS Beamline X25 for PX. Beamline X25 is the premier NSLS X-Ray beamline. Driven by a 27-pole wiggler magnet, it produces the hottest beam in the x-ray regime used for PX of any beamline at the NSLS. During the last six years, it has been used increasingly for PX. Recently, a four-module CCD-based x-ray detector was provided by a grant from the National Science Foundation to the detector-development group at Brandeis University and the BNL Biology Dept. We have installed this 20cm. square detector on a four-circle diffractometer that is under the control of the software described below. A new scientific staff member, Hal Lewis, has joined the Biology Department to work full time in development and operation of the beamline.

A second R&D project focuses firstly on upgrades to the detection system at Beamline X12B and secondly on development of a shared computing and file server for macromolecular crystallography at the NSLS. We are pursuing a noncommercial path toward development of the resource's shared computational server, that of the Beowulf cluster, developed by the Beowulf Consortium and administered by the staff of NASA's Center of Excellence in Space Data and Information Sciences (CESDIS), Goddard Space Flight Center. The greatest advantage of the Beowulf concept is the low price/performance ratio. Using relatively inexpensive, high performance commercial PC's, a Beowulf cluster can be constructed to provide computational power rivaling that of massively-parallel commercial multiprocessors (e.g., the Intel Parallax, Cray XMP) at a fraction of the capital and operating cost. Our existing Beowulf cluster consists of 18 off-the-shelf Intel Pentium-based PC's linked via a 100-base-T switched ethernet ILAN, containing ~1Gbyte of RAM and 50 Gbyte of magnetic disk space. This system was assembled for less than $50K to provide a sustained 1 Gflops (billions floating point operations per second) performance. We have begun to port data-reduction and structure-solving software to the cluster for use by all PX users at the Resource Beamlines.

A third R&D project is development of integrated beamline-control, data-collection, and data-reduction software for PX. A major goal of those of us who operate these facilities is continually to improve the interface between the user and the instruments. We take very seriously the role that good instrument-control and data-reduction software can play in making reliable use of the facilities and in making it as user friendly as possible.

The beamline software we present to our users now has several aspects of the experiment integrated into one package, is fairly easy for the users to learn to use, and is filled with checks to prevent mis-steps and to guide the user in making measurements. The software easily drives a diffractometer system like that shown in **Figure 1,** and by June of 1999, three essentially identical systems will be in operation at Beamlines X12C, X25, and X26C. The diffractometer is controlled by a many-process system that handles the diffractometer, the graphical user interface (GUI), and the four

PCs that drive the detector itself. Control of the beamline comes from another combination of a GUI with an existing algorithmic program. With this program and the GUI, we have reduced most beamline-control operations to a few button pushes. For example, ordinary beamline alignment requires only a button push to launch a few linked scans of the motorized table that carries the diffractometer, with optimisation logic providing the final position.

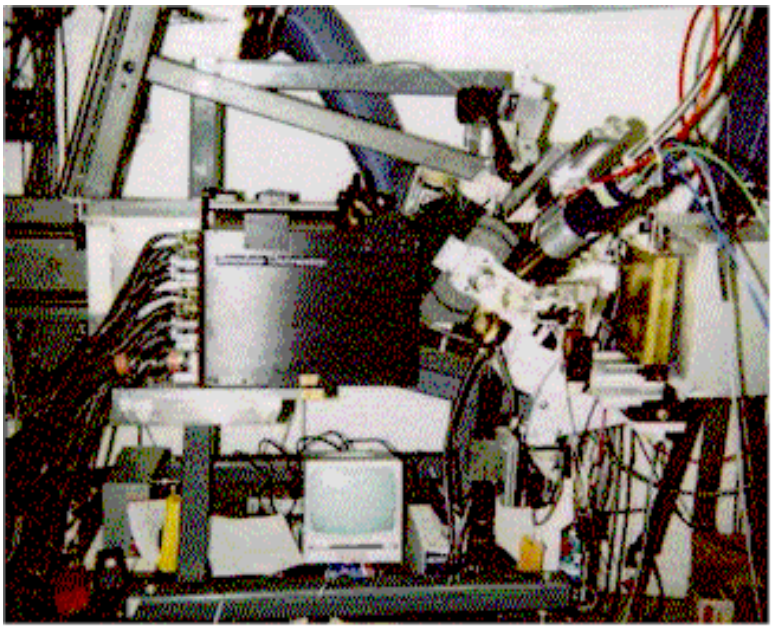

Figure 1: The Brandeis B4 detector on the Nonius diffractometer at beamline X25.

The ultimate in beamline and experimental control is represented in **Figure 2**, where the system has measured the x-ray absorption spectrum of a heavy atom in the crystal of the protein, has optimized the x-ray wavelength to the rising edge of that spectrum, and now is measuring a sweep of diffraction data. We are working to allow remote operations and monitoring of beamline experiments. We have already begun to use the web-based software for this kind of operation.

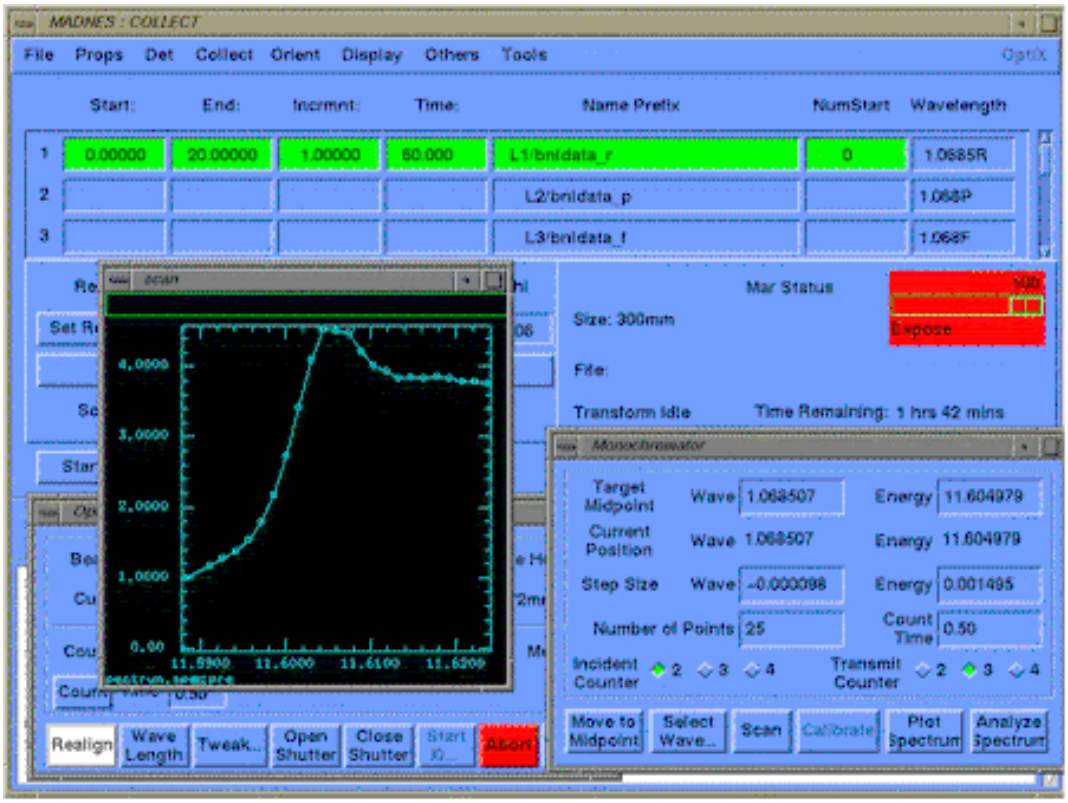

Figure 2: Automatic MAD data collection in progress with the monochromator-control box and a absorption edge for a heavy atom displayed. Reproduced with permission from Acta Crystallographia.

The fourth R&D project involves use of multilayer monochromators for macromolecular crystallography. Crystallographic studies of weakly-diffracting biological samples using bending-magnet beamlines at the NSLS

frequently are limited by the low intensity of the focused, monochromatic x-ray beam striking the sample. Acknowledging the difficulty in simply building more wiggler beamlines, one would like to find an alternative means to provide dramatically more x-ray flux at the NSLS so that one can approach these difficult problems.

Most experimental applications of synchrotron radiation require monochromatic beams, which usually are produced by perfect silicon or germanium crystal monochromators. Such monochromators diffract a photon energy bandwidth of typically 10^{-4}. Often, such fine resolution is unnecessary for monochromatic beam applications, such as small-angle scattering and some crystallographic experiments. Broader bandwidth diffractive optics, such as layered synthetic microstructures (multilayers), consisting of a periodic film of alternating heavy and light layers grown atop a substrate for which the resultant bilayers form "Bragg planes" can be designed to diffract a photon energy bandwidth of 10^{-2} or greater. This results in a "monochromatic" beam that is at least 100 times as intense as a beam monochromated by a perfect silicon or germanium crystal, making more efficient use of the continuous synchrotron radiation spectrum.

In the case of multilayers used for x-ray monochromators, silicon and tungsten (or B_4C and W) typically are applied in roughly 25Å bilayers to a depth of some 200 repeats. The increased bandpass translates to an enhancement of flux delivered on the sample by about a factor of 100 (see **Figure 3**). The broad bandpass leads to a limit in relative d-spacing resolution, d/a, and typically they are equal. For 1% bandpass, 2Å resolution data can be measured from a unit cell with a longest axis of 200Å. We intend to develop a high-intensity, multilayer-based double monochromator system for macromolecular crystallography. The first of these will be mounted at NSLS Beamline X12B.

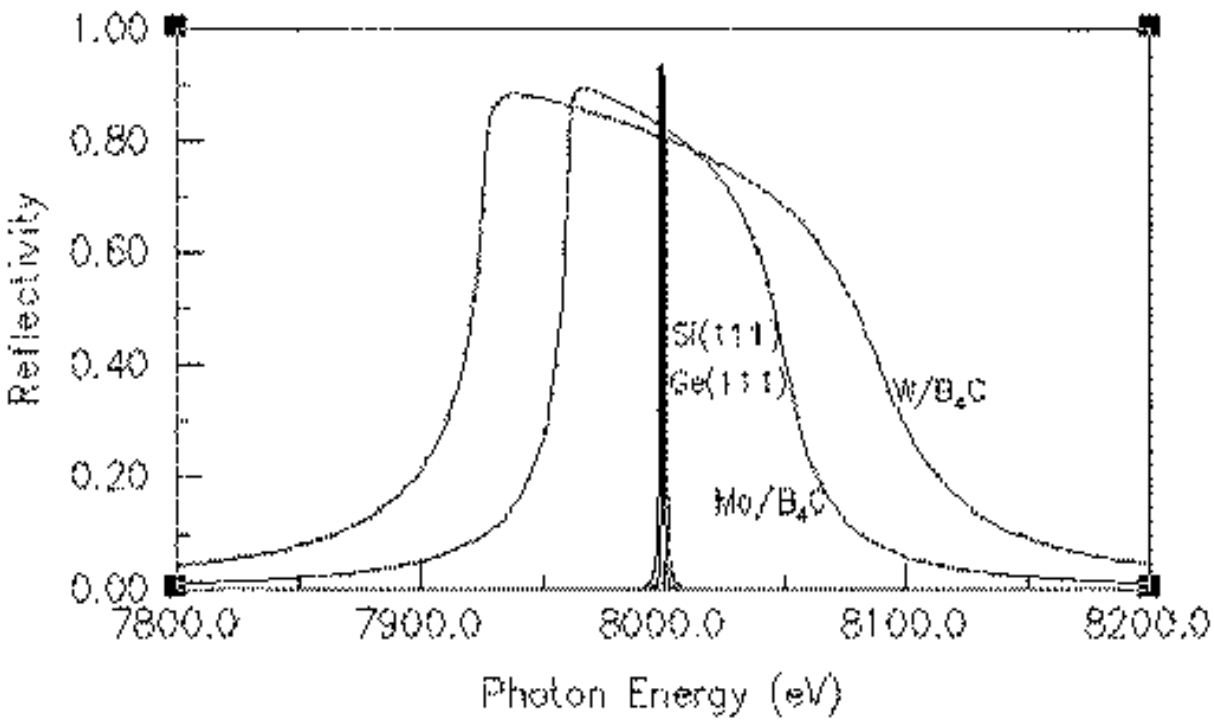

Figure 3: Reflectivity of four monochromator types. Total intensity is the integral of the curve. Notice the broad bandpass for the two multilayer reflectivity curves.

The final R&D project involves phase determination for macromolecular crystallography through the use of three-beam diffraction. This work is being done in collaboration with Edgar Weckert, Institut für Kristallographie, Universität Karlsruhe. A technique exists wherein one may measure directly the phase of combinations of reflections. This becomes possible when a crystal is oriented in an x-ray beam so that two diffracted rays (or waves) are excited simultaneously. When this happens, there will be an interaction between the second reflection **g** and the first reflection **h** via a third reflection **h-g**; each diffracted beam becomes the source for at least one more reflection. The result of the interactions among these three beams is that the phase and amplitude of each diffracted ray is modulated in a way that can be interpreted in terms of the phase of a "triplet" of reflections, that is, the sum of three phases. The indices of the triplet have the relationship that $h_1 + h_2 + h_3 = 0$. In this case $h_1 = g$, $h_2 = h\text{-}g$, and $h_3 = -h$.

The geometry of the three-beam case is shown in **Figure 4**, taken from Weckert and Hümmer. The three vectors of the triplet in this figure are drawn as coplanar. One can see that since reciprocal lattice (r.l.) vectors **h** and **g** lie on the surface of the Ewald sphere, the source wave vector K(0) could be reflected simultaneously in directions K(h) and K(g). If **h** and **g** are r.l. vectors, then h-g must be also. Therefore, there is a set of Bragg planes (**h-g**), which, in this case, are in position to reflect the wave vector K(**h**) in the direction of K(**g**), or alternatively K(**g**) in the direction of K(**h**).

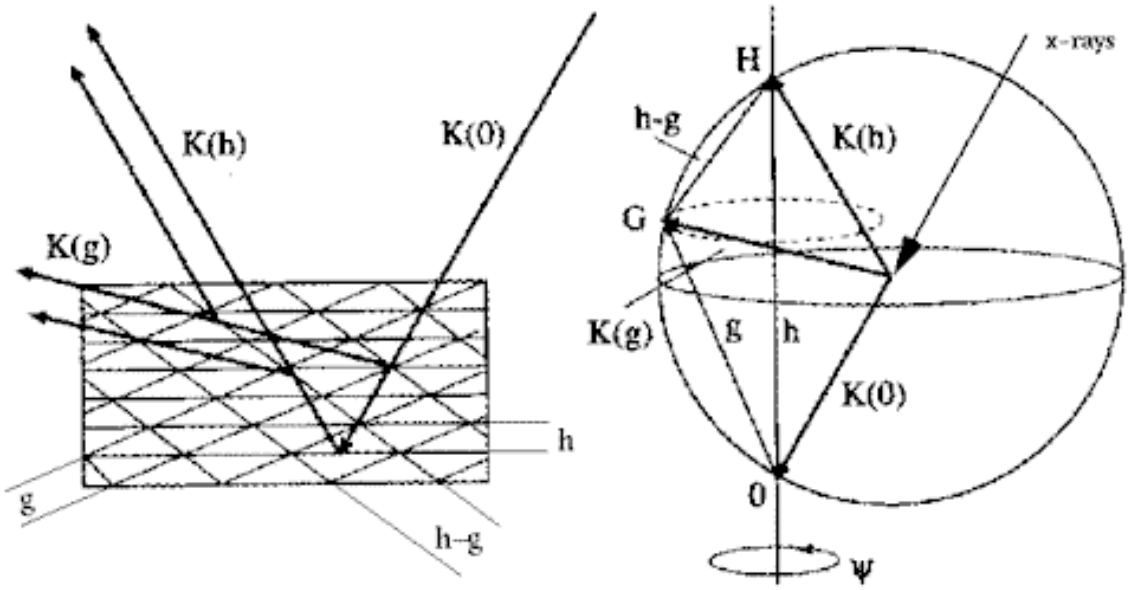

Figure 4: Geometry of three-beam diffraction. Reproduced with permission from Acta Crystallographica.

Notice that in the direction K(**h**), there are two superimposed rays. The phase of this combined wave will be the sum of the phases (relative to an arbitrary origin) from the two reflections plus a resonance phase shift: theta(**g**) + theta(**h-g**) + DELTA theta . Therefore, the phase difference between K(**h**) and the doubly-reflected wave will be $theta_3$= theta(**g**)+ theta(**h-g**)-theta(**h**)+ DELTA theta = $theta_{triplet}$+ DELTA theta . There should be a shift in the intensity of K(**h**) when it interferes with the doubly-reflected ray. Therefore, by observing the intensity shift that occurs in reflection h when the r.l. point **g** is scanned through the diffraction position, one should be able to measure the phase f3, hence $theta_{triplet}$.

For this method to work properly the specimen crystals must be high quality, the beam crossfire must be low, the unit cell should not be too large (in the range of 10^5 $Å^3$), and one needs an easily changed monochromator. We are in the process of establishing a facility to put the method into practice for crystals of biological macromolecules at Beamline X26C. A more complete description of the method can be found at:**http://www.x12c.nsls.bnl.gov/hoelzer/3BD/3beam_diffraction.html.** As a major effort in the "training" component of the Research Resource, we held a course, "Rapid Data Collection and Structure Solving at the NSLS: A Practical Course in Macromolecular X-Ray Diffraction Measurement." It was held at the Biology Department and National Synchrotron Light Source at BNL from 18-23 April 1999.

Forty-seven students attended the four-and-a-half day lecture and laboratory course. A summary of the course can be found at the web site **http://www.x12c.nsls.bnl.gov/rr_course/**. Two days were devoted to lectures on fundamentals and practical details of use of software and specimen handling. On the evening of the second day, the whole course (including some thirty instructors) moved to the NSLS for last-minute training and to begin data collection. Half of the students had won admission to the course by having proposed their own experimental problem, and they brought specimens with them for data collection. The rest of the students attended as observers and helpers. Some students brought crystals already frozen in liquid nitrogen; some brought them in delicate crystal-growing apparatus.

Six NSLS dipole beamlines were dedicated for use by students in the course; they were used for about 60 hours, with the students working in shifts around the clock. Nearly all of the projects accomplished useful data collection as part of the learning process. One of the goals of the course was to teach the students that modern synchrotron and

computational methods can lead to the production of an atomic-resolution map of the structure on the time scale of the data-collection process itself. Indeed, one of the teams of students had produced such an electron density map within about 16 hours of having started data collection. Two other groups accomplished the same thing by the end of the course.

Further information about macromolecular crystallography at the NSLS can be found at **http://www.x12c.nsls.bnl.gov/x12c/nsls_px.html** . Additional information about the Resource can be found at **http://www.x12c.nsls.bnl.gov/rr_summary brief_intro/brief_intro.html** and **http://www.x12c.nsls.bnl.gov/rr_summary/rr_summary.html.**

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July 1999 NSLS Newsletter

A Users' Perspective

Barbara Illman, UEC Chair

USDA FS Forest Products Lab, University of Wisconsin

After the 1999 User's Meeting, I take up the reins from John Parise as Chair of the User's Executive Committee (UEC). Newly elected UEC members and the Special Intrest Group (SPIG) representatives also start new terms on the committee. The UEC is an independent representative body of the users. Please contact any of us regarding user concerns and issues.

We wish to thank John Parise for his efforts over the past year. On our behalf, John has worked diligently on several issues that can be highlighted by the work with Brookhaven Science Associates (BSA) concerning (1) the closing of the cafeteria on weekends and resolving an alternative for weekend food service to all BNL User facilities and (2) UEC members on the Community Advisory Council (CAC) which is a forum intended to advise BNL about the Laboratory's policies and operations related to environment and public health issues. He has worked on the national level to coordinate science-specific interest groups to promote the use of synchrotron radiation research to NSF, DOE, DOD and NIH. Special thanks also go to dedicated UEC members rotating off the committee, Paul Stevens and Joel Brock. A special thanks goes to Eva Rothman for her many contributions over the years, and a welcome to Mary Anne Corwin, the incoming Ex-Officio User Administrator on the UEC.

At the last UEC meetings, members spent a lot of time discussing the options for NSLS Beam Energy. Months of discussions with users had gone into the final UEC recommendations: (1) the NSLS be operated at one ring energy and that the energy be 2.8 GeV, (2) for the present, the 2.5/2.8 modes be alternated, as is currently the case (3) exclusive 2.8 GeV operations be implemented when the low-emittance lattice parameters have been tested and found to be satisfactory (in one to two year's time), and (4) this recommendation be disseminated widely, for example, through the 10-day e-mail news and in the NSLS Newsletter. News messages from the 10-day e-mail news can be found at **http://www.nsls.bnl.gov/Intro/News/News-msg.html** and the NSLS Newsletter can be found online at **http://www.nsls.bnl.gov/Pubs/NewsLtrs.html**

The 1999 User's Meeting and Workshops were a big success. I offer my congratulations and personal thanks to everyone involved, particularly Paul Stevens, the Meeting Chair, and Linda Feierabend, Meeting and Workshop Coordinator and also to the organizing committee members, Mark Chance, Steven Hulbert, Sue Wirick, Nancye Wright, and Eva Rothman. They represent the coordinated work of the NSLS User Administration Office and the program committee of users. Over the next year, we expect to report accomplishments by the NSLS administration and users working together to maintain the historical nature of NSLS as a state-of-the-art user facility. The users have available to them a team of dedicated individuals in the NSLS administration and staff.

NSLS users have a unique position and responsibility for maintaining the excellence of the NSLS. Users need to be more aware and responsive to outside interests that can deter our use of large scientific

facilities. We encourage users to voice opinions about `hot' issues that threaten our ability to conduct science. In order to aid the involvement of NSLS users in many timely issues, the UEC has instituted an independent home page on the World Wide Web that does not violate lobbying rules, **http://www.buoy.com/~nsls_uec/**

The web page keeps membership informed about important issues affecting NSLS users and provides resources that allow users to communicate. Additional vehicles of communication are:

- Selecting a special interest group with the User Administration and contacting your SPIG Representative
- Contacting UEC members
- Attending and participating in town meetings

The *next generation of synchrotron users* will come from diverse areas of science. Numerous examples can be given of synchrotron users who have pioneered work at NSLS that has impacted their field. These users need a facility and synchrotron administration that will be responsive to new requirements and special needs. I think that the NSLS is such a facility and the administration, lead by Chairman Michael Hart, have proven to be capable of meeting the challenge. The Birgeneau Report also supports this view. The Birgeneau Committee was the blue ribbon DOE-appointed panel that lauded NSLS for productivity, user support and innovation. We need to find out how we can assist in the implementation of the Birgeneau Report recommendations that would allow NSLS to build on its innovative approach to serving all scientific communities, how we can assist the NSLS participating research teams (PRTs) and general users to meet their needs.

More biological scientists will integrate synchrotron radiation into their research programs in the next century and may apparently have the most numerous user groups at NSLS in the coming year. This has in part resulted in the user-driven $4 million NIH grant given to NSLS to support beamline infrastructure for research on molecular biology and protein crystallography. This successful coalition of users prompted many of us to propose this group as a model from which we can all learn. We need to pool our subject matter needs to apply for block grants from our specific funding agencies. The UEC is committed to assisting in this endeavor, providing input to user groups and granting agencies.

To summarize, I want to stress that the UEC provides a forum for users' interests; it is the users' representative and interface with the NSLS and a coherent voice to express these interests to the NSLS and BNL administrations, DOE, funding agencies and special outside interest groups.

Please send comments and questions to me at billman@facstaff.wisc.edu.

Users' Executive Committee		
Chair:	Barbara Illman	USDA/FS Forest Products Lab, U. of Wisconsin
Vice-Chair:	Mark Chance	Albert Einstein College of Medicine
Past Chair:	John Parise	SUNY @ Stony Brook
Secretary:	Lisa Kelly	U. of Maryland Baltimore County

Member:	Carol Hirschmugl	University of Wisconsin
Member:	John Hill	Brookhaven National Lab, Physics Dept
Member:	Kenneth Evans-Lutterodt*	Bell Laboratories, Lucent Technologies
Member:	Chris Jacobsen*	SUNY @ Stony Brook

1999 SPIG Election Results		
Bio/Cryst	Malcolm Capel	Brookhaven National Laboratory, Biology
Imaging	George Cody	Carnegie Institute of Washington
Industry	Jean Jordan Sweet	IBM Research Division
Infrared	Lisa Miller	Brookhaven National Laboratory, NSLS
Nuclear	Mark Lucas	Ohio University
Scattering	Steven Ehrlich	Purdue University
Timing	Larry Carr	Brookhaven National Laboratory, NSLS
Topography	Michael Dudley	SUNY @ Stony Brook
UV Photo Emission	Robert Bartynski	Rutgers University
XAFS	Dale Sayers	North Carolina State University

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X-Ray Diffraction Studies of Adsorbed Films

J. Z. Larese, *M. Sprung*, *A. Freitag* and *J. Hanson*

BNL-Chemistry Department

The use of x-ray diffraction to study the behavior of thin films adsorbed on solid surfaces can be traced back to the work of Horn and Birgeneau in the mid 1970's[1]. Since then, literally hundreds of investigations have been performed to explore such things as commensurate-incommensurate phases and transitions, Kosterlitz, Thouless, Halperin, Nelson and Young (KTHNY) melting in two dimensions, orientational and compositional ordering, layer-by-layer growth/melting and wetting[2]. These studies have employed single crystal surfaces and crystalline powders with large surface-to-volume ratios, like exfoliated graphite. The principal challenge in the diffraction measurements is to separate the signal coming from the surface film from the large, often overwhelming, signal from the substrate. This usually requires making a difference map by subtracting the signal from the bare substrate from those coming from the adsorbate coated one, keeping in mind that the difference in photo absorption in the two cases may be significant. We have recently employed x-ray image plate technology in our investigations of the structure, dynamics and reactivity of molecular films on surfaces. The following article describes recent measurements using the Chemistry Department's diffraction Beamline X7B. These studies are one component of a larger program in Materials Chemistry that includes materials synthesis, elastic and inelastic neutron scattering, thermodynamics, scanning probe microscopy and computer simulation methods.

A number of new components have recently been installed at X7B. These include a new variable-curvature pre-mirror, a detector/sample holder trolley outfitted with a MAR 345 Image plate based detector system, a commercial gas-cabinet and venting system for use with in-situ, time resolved reactivity studies, an automated gas-handling system, gas flow devices and a mass spectrometer based flow reactivity gas system. Some of these upgrades were introduced as part of the new Chemical-Materials Sciences combined initiative known as Chemical Interfacial and Catalysis Effort (CICE) based in the BNL Chemistry Department.

Figure 1 is a schematic view of the experimental setup recently developed for use at X7B to investigate adsorbed films. A copper sample cell has been fabricated with two replaceable, indium-sealed beryllium windows and a heated stainless-steel capillary used as the gas fill line. The cell is mounted on the second stage of a closed cycle helium refrigerator that can be regulated to ±5mK using a Neocera Model LTC-21 temperature controller. Sample gases are introduced to the sample cell using an automated gas handling system. The heart of the setup is a MAR 345 Image plate detector system. This device has several advantages over conventional discrete image plate and separate plate reader systems. Most notably are the two orders of magnitude enhancement of the dynamic range, the automated single-detector, read-erase feature and the exact repositioning of the image plate. The experiments described below have been performed using this apparatus in various stages of it's development.

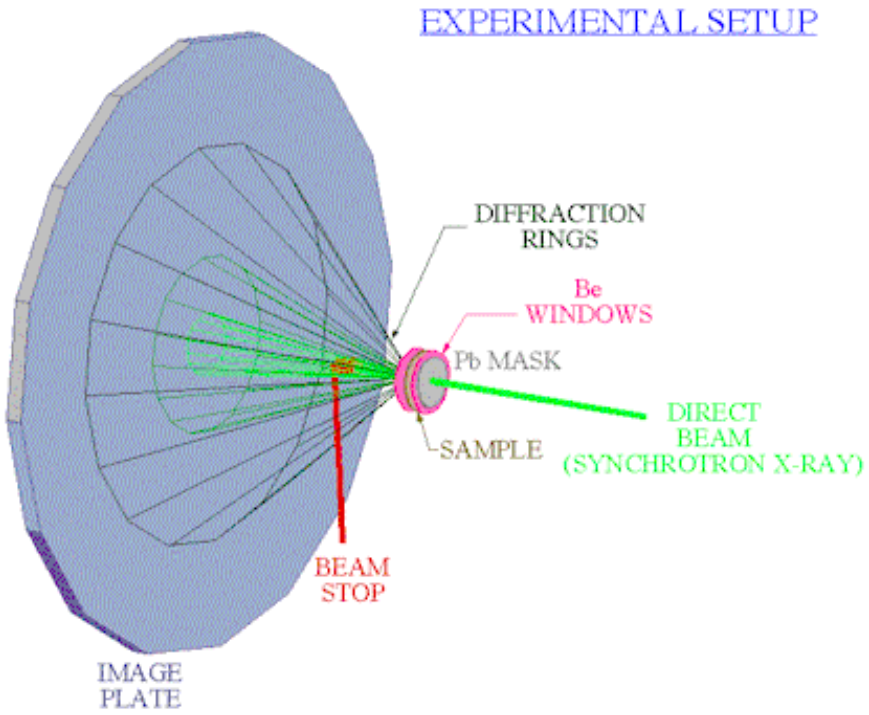

Figure 1

To characterize the performance of the image plate system, it was decided to investigate a well-characterized system first. We chose to examine the adsorption of Xe on Graphite near monolayer completion[3]. A lightly compressed, vermicular form of crystalline graphite known as graphite "foam" was used as a substrate. **Figure 2** illustrates the coverage dependence of the diffraction pattern for an isotherm performed at 112K with Xe films between 0.83 = X(mnl) = 1.30. The fluid-to-solid phase transition is clearly visible within this coverage range. The two diffracted peaks near 1.6Å^{-1} and 2.9Å^{-1} can be indexed as the (10) and (11) peaks of a triangular lattice formed by the Xe film. These peaks move toward higher Q with increasing surface density in response to the compression of the film. These data represent difference plots derived by subtracting the signals recorded for the bare graphite substrate from those covered with the Xe films. The individual Debye-Schirrer rings recorded on the image plate were integrated over the entire image plate face using the Fit2D software package developed by Hammersley *et.al.* from the ESRF[4]. Furthermore, a combination of an automated gas handling system and the MAR 345 detection system is ideally suited for this type of measurement. Data for this type of study can be recorded over the entire Q range in 2 minutes even on a bending magnet line.

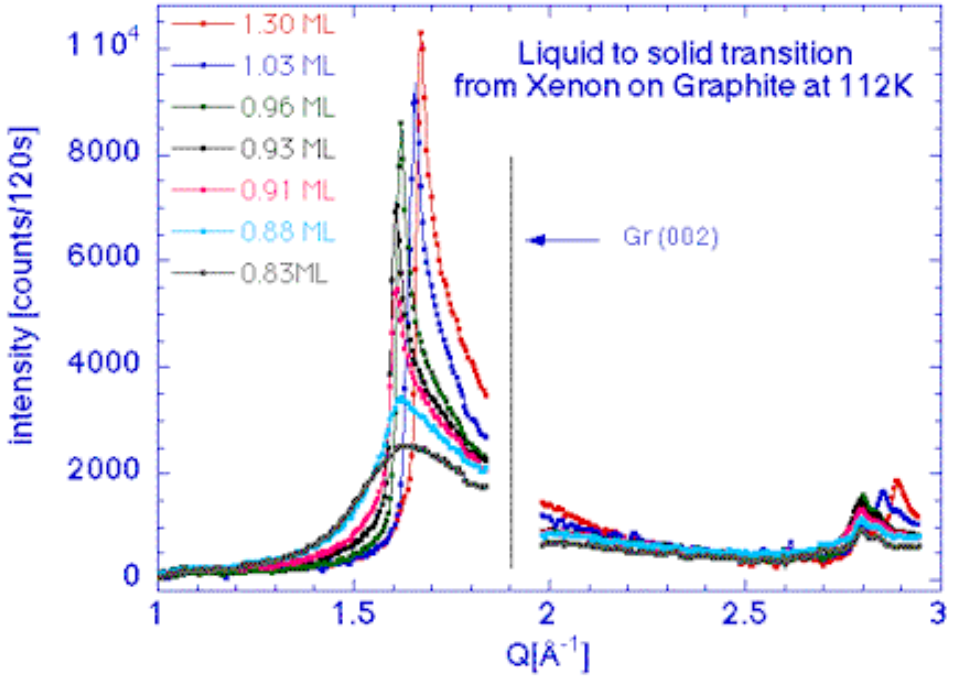

Figure 2

The interaction of the methyl halides CH_3R (R=F, Cl, Br, I)with MgO(100) surfaces have also been examined. The MgO powders were synthesized at BNL using a novel, gas-phase based method for producing pure and doped metal oxides[5]. The MgO powders used in this study exhibit a surface area of about 10 m^2/gm and can easily be prepared in 100 gm quantities. The goal here is to determine the structural properties of the monolayer films including the low temperature orientational ordering and dynamics using complementary elastic and inelastic neutron methods. Our interest in this system originates from the possibility that electrostatic (dipolar) ordering might take place and from an ongoing interest in the wetting properties of small polar molecular films on solids[6,7]. As is usually the case, a set of adsorption isotherms is first collected to identify interesting regions in the phase diagram. **Figure 3** shows a set of adsorption isotherms for CH_3Cl on MgO. Wetting is "incomplete" i.e. only a finite number of uniform layers form before the bulk phase appears. A layering transition for the 2^{nd} layer is found near 160K.

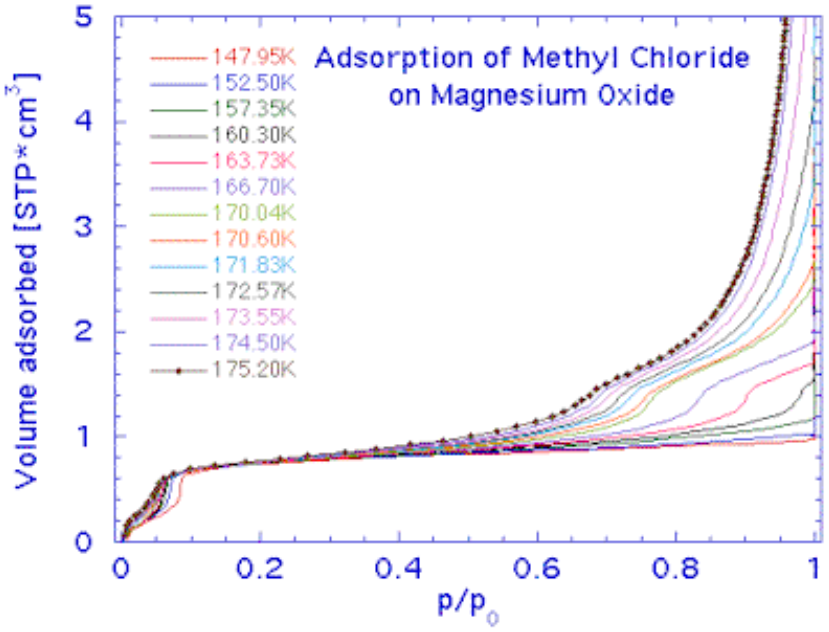

Figure 3

Figure 4(a) is a snapshot of the diffraction pattern taken directly from the MAR 345 while **Figure 4(b)** is a difference map recorded for the submonolayer phase of Methyl Iodide, CH_3I, at 120K. Preliminary analysis of these data indicates that CH_3I forms a centered rectangle solid structure, commensurate with the MgO substrate. There are eight molecules in the unit cell. The dimensions of the cell are a=8.42 Å and b= 25.27 Å. This areal density determined from earlier adsorption isotherms is consistent with value based on the structural study[8].

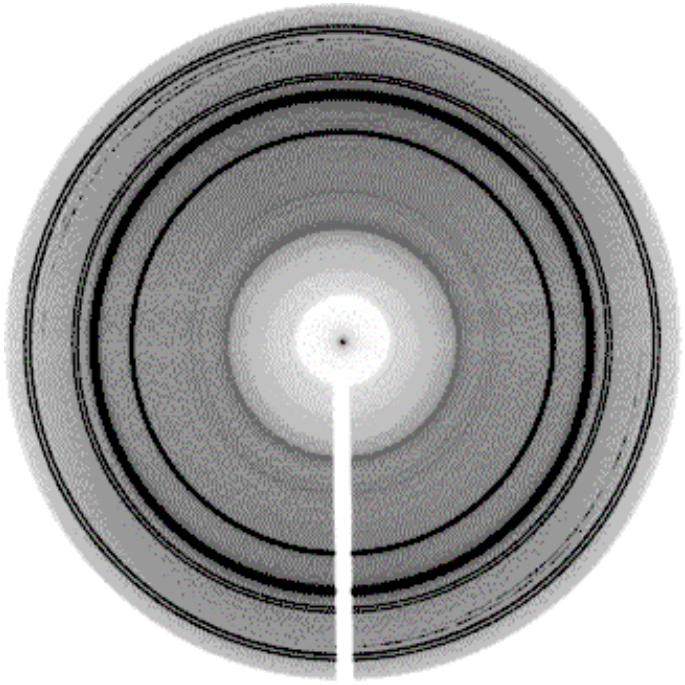

Figure 4a

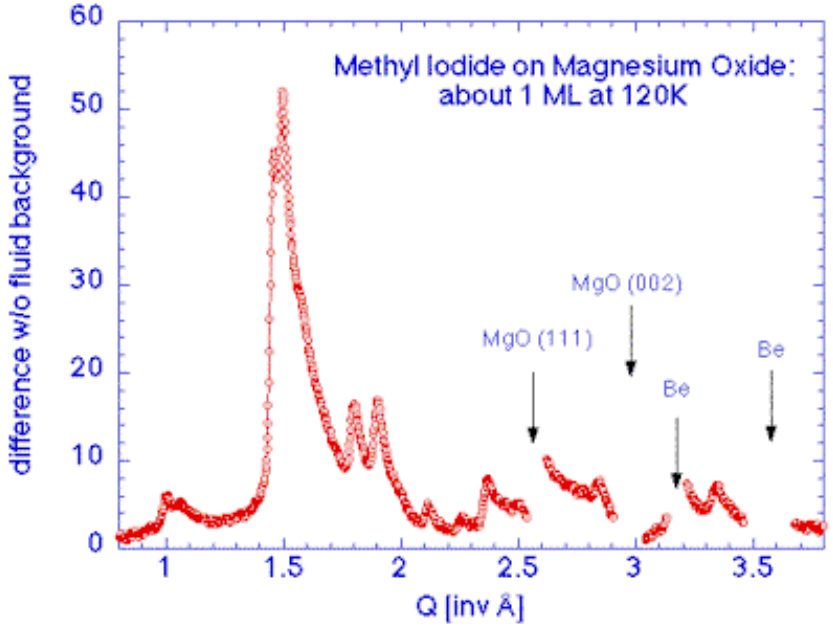

Figure 4b

Migone and co-workers[9] have recently collected an extensive set of adsorption isotherms characterizing the thermodynamic properties of several rare gases and small molecules adsorbed on hexagonal powders of Boron Nitride (BN). BN has an in-plane lattice constant that is about 2% larger than that of graphite in the basal plane. Little is known about the structural properties of adsorbed films on BN although strong similarities in the adsorption behavior are expected. The adsorption of methyl halides on graphite has been well-characterized [10]. For example, CH_3Cl forms both the low-density (LD) and high-density (HD) phase on graphite. **Figure 5** shows the difference maps recorded for submonolayer CH_3Br and CH_3I on BN recorded at 50K. Preliminary analysis indicates that on BN monolayer films of CH_3Br sit with carbon-halogen axis pointing perpendicular to the surface plane (i.e., like the HD phase of CH_3Cl on graphite) while CH_3I lies with the carbon-halogen axis parallel to the surface plane (i.e., just like CH_3I does on graphite). Future work will focus on expanding the thermodynamic studies of the phase diagram to identify areas where phase transitions occur.

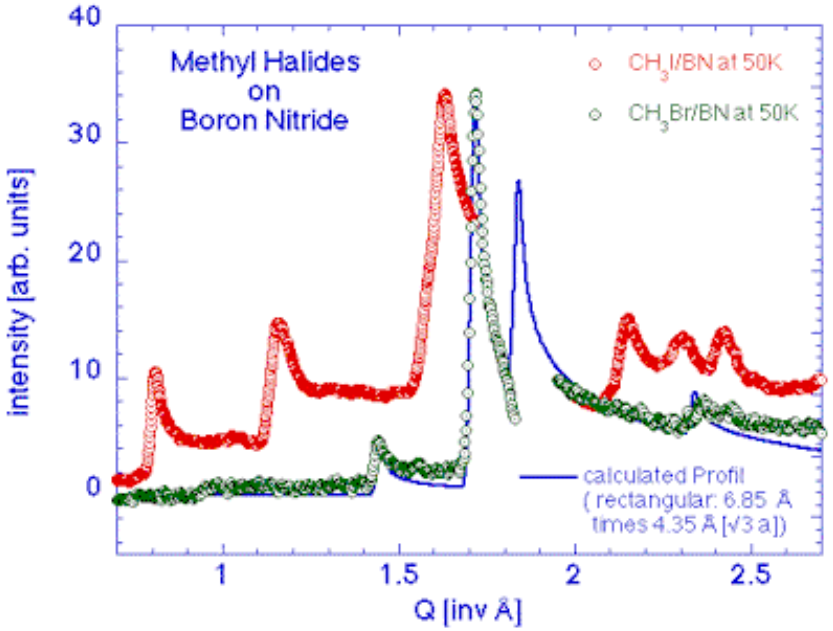

Figure 5

As noted above, another goal of the program is to elucidate the role of the surface in mediating chemical reactions. Here the interaction of SO_2 with MgO has been investigated using x-ray diffraction and XANES. The release of nitric (NO_x) and sulfur (SO_x) compounds during the burning of fuels constitutes a major environmental problem. Increased regulations affecting environmental pollution require new, more efficient catalysts to be developed for the removal or destruction of NO_x and SO_x. It has been suggested that catalyst based on metal promoted magnesium or cerium oxides can accomplish this goal. Using *in situ*, time resolved x-ray diffraction methods we have begun to address these issues. **Figure 6** illustrates the application of the MAR 345 to elucidate the interaction of SO_2 with MgO. When monolayer quantities of SO_2 are adsorbed on MgO at temperature near 200K signals are observed in the Q range between 1.4 $Å^{-1}$ and 2.2 $Å^{-1}$ indicative of the formation of a disordered (liquid like?) film of SO_2on the MgO surface. Temperature programmed desorption studies show that heating to temperatures in excess of 350°C is required for removal of the SO_2 from the MgO surface. Using x-ray near edge spectroscopy (XANES) at the sulfur K edge it can be shown that both SO_3 and SO_4 species exist at temperatures below 400°C (**see Figure 7**)[11]. Future work will focus on the stability and the relative concentrations of SO_x species as a function of temperature on various pure and doped metal oxide samples.

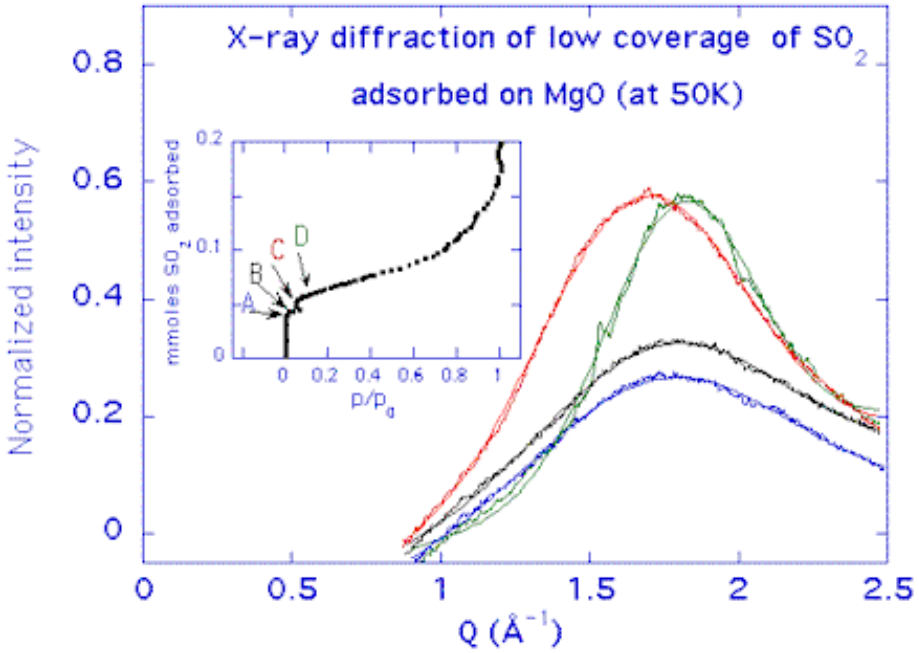

Figure 6

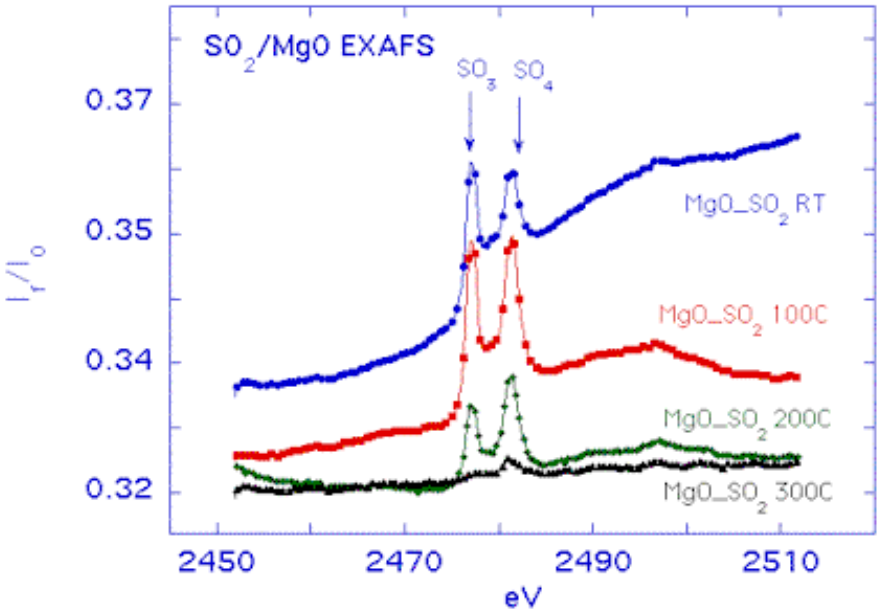

Figure 7

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July 1999 NSLS Newsletter

Lessons-Learned from Recent Events

Bob Casey

Associate Chairman ESH&Q

There is an old saying that if we don't learn from history, we are condemned to repeat the same mistakes again. An important part of our ESH program is to share operating experiences with all personnel and ensure that these lessons-learned are discussed and applied to our program where appropriate. Reported in this Newsletter are three recent events that we can learn from.

The Modulator Fire

Tom Dickinson

Safety Officer

The linac modulator fire in January certainly got our attention. The fire resulted in a dollar loss of $95,000 and program downtime of seven days. The fire occurred shortly after midnight on January 25 and originated from intense electrical arcing within one of ten large capacitors contained within the modulator power supply. The arcing continued for about 90 seconds before power to the supply was secured. At this point, there were no observable signs that a fire was developing within the power supply cabinet. The operators returned to the control room to call in technical support to evaluate the nature of the malfunction. About 10 minutes later, the first smoke alarm sounded, and all three operations personnel returned to the modulator power supply, leaving the control room unmanned.

A heavy smoke condition quickly developed in the immediate vicinity of the fire and resulted in smoke in many other areas of the building as well. The dense smoke quickly forced NSLS personnel to exit into the lobby of the building, where the building evacuation signal was sounded about 10 minutes after the first smoke alarm.

The BNL Fire/Rescue Group responded to building alarms and extinguished the small but intense fire using hand-held portable fire extinguishers. The fire was out about 25 minutes after the first smoke alarm sounded. The contents of the power supply cabinet were totally consumed by the fire; however, the fire did not spread beyond the cabinet. Two sweeps of the building were conducted by Fire Rescue personnel after the fire was out to ensure that no one was left in the building following the evacuation.

The Investigation Committee, chaired by Richard Heese, identified a number of issues that are important to the operation of the machine and to personnel on the NSLS floor. These items involve topics such as:

- early detection of modulator malfunction,
- combustible loading of components installed on the NSLS floor,
- fire detection/protection in components,

- ability to quickly secure electrical power during emergencies,
- emergency response procedures used by NSLS personnel,
- training and drills for emergency situations for operations and user personnel, and
- design review of new or modified systems.

Work is in progress on all issues.

There were some important lessons-learned for personnel on the experimental floor. In interviews after the event, several users indicated that they were uncertain about whether they should evacuate the building; and some personnel returned to their beamlines while the evacuation bells were ringing. One user attempted to leave the building by the front door and was nearly overcome by smoke as he approached the linac area. He retreated and found an emergency exit very near to his beamline. Concerns were also raised about accounting for occupants of the building and whether they had all gotten out. This confusion convinced us that we need to improve our procedures, information, and training about emergency evacuation of the building.

All occupants need to know exactly what to do in an emergency and what their responsibilities are. Each occupant should know:

- which exits are available to them, from wherever they may be working in the building,
- where to assemble outside the building (new postings on all doors),
- when they are allowed to re-enter the building, and
- what their responsibilities are, if any, with regard to providing assistance when dealing with an emergency.

Emergency Exits: For visiting users, this information is provided as part of the BLOSA training. We plan to improve this training to include a walk-down for each user to the two closest exits. This exercise will reinforce the correct behavior during the stress of a real emergency.

NSLS staff and resident users should check out emergency exits near places where they work. This is a good habit to get into, not only at your workplace, but also when checking into a hotel, going to a movie, or boarding a plane.

Assembly Area: This information is provided on the new yellow signs at all building entrances. The Assembly Area for people who evacuate the building is near the main entrance on Brookhaven Avenue. If the weather is bad, the alternate is in Building 726, (the shop building about 200 meters east on Brookhaven Avenue). It is important that everyone check in at the Assembly Area so that all building occupants may be accounted for. Otherwise, Fire/Rescue personnel may have to search for people in the building rather than fighting the fire.

Permission to Re-Enter Building: This information will be provided at the Assembly Area. If in doubt as to whether or not you may re-enter the building, ask a member of the operations crew.

Special Responsibilities: Anyone at the NSLS who has special knowledge about equipment or chemical or radiological hazards associated with their work is expected to offer assistance in dealing with these safety issues in the event of an emergency. These matters may have been addressed during the experiment safety review, but during an emergency, an offer to help should be made.

For our part, NSLS Operations and Safety personnel are working on ways to provide better information to occupants and to make our emergency procedures work more efficiently. We expect to implement a recorded message that will repeat every 30 seconds or so when there is a building evacuation. We may be able to silence the bells during the message so you can actually hear it. We will be running evacuation drills regularly, so we can all stay in practice.

The Laser Incident

Andrew Ackerman

Industrial Hygienist

We recently had an incident involving work with a Class IV laser that everyone at the Light Source should know about and learn from. Class IV lasers have sufficient power to cause eye or skin injury and can ignite combustible materials. For those reasons, several controls are required to work with these devices including proper posting, training, baseline eye exams, and enclosure of the beam with interlocks to the power supply of the instrument which will trip if the enclosure is removed. The laser work involved in this incident had been reviewed and planned, and appropriate controls had been identified and documented. The beamline hutch served as the required enclosure and the device was operated from outside the hutch. A small switch was attached to the hutch door as part of the required interlock. This experiment has been ongoing for some time and the scientists involved are experienced with laser operation.

The users arrived at the Light Source, set up their equipment and found that the laser was not operating properly. To determine the problem, they needed to work on the device while inside the hutch. Since the laser controls were located outside the hutch, work on the device required two people (one inside the hutch with the door closed and a second person outside the hutch at the controls). This was an awkward arrangement, so the users chose to override the interlock by taping the door switch closed, thereby giving unimpeded access to their equipment. They realized the potential hazards involved and placed a filter at the exit slit of the beam to attenuate the beam power and reduce the risk. Although they knew they were violating Light Source established requirements as well as their own, they were concerned about their schedule and were anxious to fix their laser problem. Later, NSLS ESH personnel observed the taped interlock switch during a routine tour of the floor. They investigated the incident and reported it to NSLS management shortly thereafter.

The work was terminated and was not allowed to resume until procedures were modified and additional personnel were qualified to operate the laser. Written reprimands were issued and NSLS research privileges were revoked for a three-month period for two individuals.

There are some important lessons-learned in this incident. Obviously, the scientists involved in this event had no intention of injuring anyone and took precautions to avoid that. Their mistake was not involving the Light Source staff in the solution to their problem. Had they approached us with this problem, we would have worked with them to determine what was needed to proceed in a controlled manner.

We rely on this partnership with the user community to ensure safe operations at NSLS. We strive to work with you to accommodate your needs within the ESH requirements that we are all expected to comply with. The users involved in this incident realize their mistake and have taken steps to ensure that

there will be no reoccurrences. Let's work together to ensure that all experimental work at NSLS is conducted safely.

Overheating Beamline Component Creates Smoke

Nicholas Gmür

ES&H Coordinator

Part of a custom-made experimental setup in use on the experimental floor involved directing heated (200°C) nitrogen gas onto a sample. The gas was warmed by a heating jacket around the gas line and controlled by an Omega unit. The temperature of the heated gas was monitored by thermocouples at the mouth of the gas discharge line. When the N2 cylinder ran out of gas, the gas flow stopped and the thermocouples cooled down. This apparent reduction in observed temperature resulted in the controller providing more current to the heater. The heating jacket eventually overheated and generated smoke locally. An Operations Coordinator detected this and the experiment was shut down. Although no fire resulted and smoke evolution was not severe, there are obvious implications that should be considered.

- The apparatus should have an adequate high temperature electrical power shut off with a sensor at the heater rather than being located at the gas discharge.
- Nationally recognized testing laboratory (UL, etc.) com ponents should be used when possible. Where custom- made equipment is used, then applicable standards (ANSI, IEEE, etc.) must be followed. The equipment must be listed as part of the Experimental Safety Approval Form.
- The N2 gas system should have proper pressure gauging, a flow volume and direction sensor as well as the capability of removing electrical power to the apparatus should gas flow cease.
- Use of house air would be an improvement; a cylinder contains a finite amount of gas.
- Spokespersons and Local Contacts should maintain over sight to minimize experimental hazards at their beamlines.

The NSLS provides a variety of support services to the Users. **Contact:** John Gallagher (x5770, pager 0875) for experimental electronics support.

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July 1999 NSLS Newsletter

A New Method for Examining the Dynamics of Macromolecules: Time-resolved Synchrotron X-ray "Footprinting"

Mark Chance[†‡], *Michael Brenowitz*[‡], *Michael Sullivan*[‡], *Simin Malenia*[‡] *and Corie Ralston*[‡]

[‡]*Center for Synchrotron Biosciences, Department of Physiology and Biophysics and Department of Biochemistry, Albert Einstein College of Medicine of Yeshiva University and* [†]*BNL- NSLS*

Time-Resolved Structural Biology

Achieving an understanding of conformational dynamics of macromolecules and their complexes during processes as diverse as ligand binding, folding and catalysis is a challenging and important step toward providing detailed molecular descriptions of biological systems. The flourishing use of synchrotrons in structural biology studies for examining the structural dynamics of biomolecules in real time is a response to the need for such information for a wide range of biological processes[1]. Nuclear Magnetic Resonance (NMR) spectroscopy and crystallography have high structural resolution but are sometimes difficult to use in the study of conformational dynamics. However, inventive approaches in the NMR and crystallography fields have enabled kinetic structural analysis. For example, deuterium exchange quench studies of protein folding by NMR have yielded major insights into conformational dynamics of macromolecules with high structural resolution.[2,3] Time-resolved crystallographic studies have provided snapshots of the chemical reaction steps for the hammerhead ribozyme and even nanosecond dynamics of myoglobin ligand interactions subsequent to photolysis.[4,5]

Of course, the major drawbacks, even for these powerful methods, include: 1) the limited size of the proteins or complexes that can be analyzed by NMR; 2) the need for quenching methods to "freeze" the exchange in NMR; 3) the typical necessity for significant quantities of soluble materials; 4) difficulties in crystallization of large macromolecules or complexes; and 5) the limitations imposed on dynamics by the crystal lattice. Thus, there is clearly room for new techniques to monitor the macromolecular dynamics of nucleic acids, proteins, and their biologically relevant complexes. Time-resolved synchrotron hydroxyl radical (•OH) footprinting provides a method that probes the solvent accessible surface of nucleic acids, proteins and their complexes on millisecond timescales with high structural resolution at nanomolar to micromolar concentrations of materials. Exposure of macromolecules to a "white" x-ray beam results in stable modifications of covalent bonds after only millisecond time exposures. We have recently shown that synchrotron •OH footprinting provides a unique time-resolved probe of nucleic acid dynamics and can answer fundamental questions relevant to the structural biology of RNA folding.[6] We are also developing related methods to analyze protein structure that will be equally powerful in probing the dynamics of these macromolecules.[7,8]

What is "Footprinting?" "Footprinting" refers to assays that examine ligand binding and conformational changes by determining the solvent accessibility of selected structural regions (e.g. the backbone, particular amino acids, or bases etc.) of macromolecules through their sensitivity to chemical or enzymatic cleavage, solvent exchange or modification reactions. The development of quantitative protocols for the conduct of footprinting experiments was a significant breakthrough in the use of these methods. Quantitative footprinting can determine thermodynamic[9] and kinetic[10] constants that describe macromolecular interactions. The individual-site binding isotherms and individual-site kinetic progress curves determined from quantitative thermodynamic and kinetic footprinting studies, respectively, allow a detailed energetic and temporal "portrait" of the macromolecular transition to be painted.

Radiolysis and •OH chemistry. Synchrotron x-ray footprinting is a melding of stopped-flow mixing technology and radiolytic chemistry using a high flux x-ray source to generate the footprinting re-agent, the hydroxyl radical.

Radiolysis generates •OH according to **Equation 1**.

$$H_2O \xrightarrow{h\nu} H_2O^+ + e^-_{dry} \rightarrow H_3O^+ + {\bullet}OH + e^-_{aq} \quad (1)$$

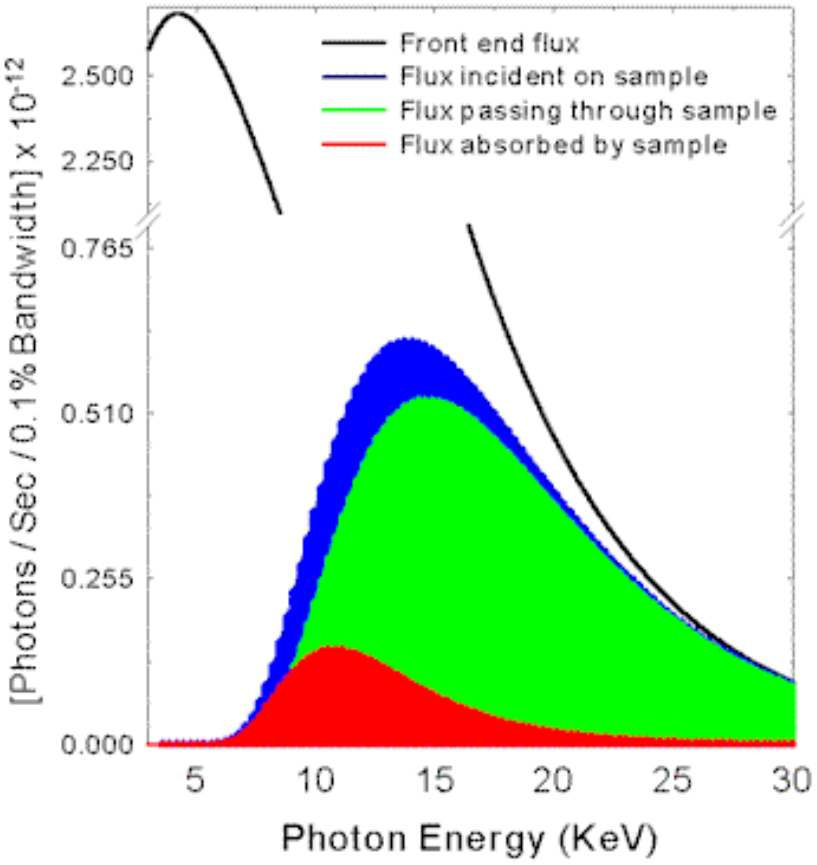
As photons interact with water and generate a water ion and an electron,[2,8] the ionized water molecules react with water to produce •OH according to the reaction outlined in **Equation 1**. The initial interactions of ionizing radiation with water occur in "spurs" localized around the radiation absorbing water molecules. The chemistry within a spur involves the reactants on the right-hand side of **Equation 1** and is complete within 100 nsec, yielding the major products in **Equation 2**.

$$4.14\ H_2O \xrightarrow{100\ eV} 2.7e^-_{aq} + 2.7\ H^+ + 0.61H{\cdot} + 2.87\ {\cdot}OH + 0.43H_2 + 0.61H_2O_2 + 0.03{\cdot}HO_2 \quad (2)$$

As illustrated in the detailed stoichiometry of **Equation 2**, for every 100 eV of energy absorbed, 2.87 •OH are produced.[11] Thus, the yield of •OH is 0.3 mmole Joule^{-1} of absorbed energy. Irradiation of aqueous solutions with various forms of radiation (such as gamma-rays, ß-particles or fast neutrons) result in basically the same ionizations of water.[11]

Figure 1 shows the flux emanating from a bending magnet port (impinging on a sample typical for a synchrotron footprinting experiment) at the National Synchrotron Light Source Beamline X9A when the ring energy is 2.584 Gev, as a function of energy. Taking into account losses due to the current Beamline X9A optics, which includes two Be windows (254mu and 500mu) and an Al window (200mu) gives the flux incident on a typical 10 microliter sample as a function of energy. We can also calculate the transmitted flux subsequent to the 1-mm water path, the difference between the two curves is, of course, the absorbed dose. Note that total absorbency is small, thus the dose is homogeneously delivered through the 1-mm path. Previously published calculations suggest that 5 x 10^{-4} M hydroxyl radicals are produced per second under these conditions[11]. Considering the radical kinetics, millisecond timescale exposures are required to provide substantial modification and cleavage of macromolecules when present at micromolar or lower concentrations.[12,13]

Figure 1: Photon flux intercepted by a 1-mm pathlength sample of ~10 microliters



(3-4 mm diameter) at beamline X9A, National Synchrotron Light Source. Beam energy of 2.584 GeV, 250 ma beam current. The figure indicates the front-end flux available for the specified geometry, the flux absorbed by the beamline optics incident on a typical sample, the flux passing through and absorbed by the sample. Calculated from Center for X-ray Optical website; ***http://www-cxro.lbl.gov/optical_constants/*** *(valid as of 8-20-98).*

Structural Biology of RNA Folding

Our first "proof of principle" of the method consisted of an analysis of the folding of an RNA catalyst, the Tetrahymena ribozyme. During the folding of both proteins and RNA, dramatic changes in solvent accessibility occur. The conformation changes of RNAs, in fact, can limit the speed of chemical reactions carried out by RNA catalysts[14]. As a result, the process by which RNA molecules fold into their native conformation has received much attention. Early investigations into the folding of tRNA established approximate timescales for the formation of secondary (10^{-4}-10^{-5} s) and tertiary interactions (10^{-2} - 10^{-1} s), with reorganization of incorrect secondary structure occurring more slowly (0.1 - 1 s). Recent work has shown that folding of large RNAs is more complex, involving multiple pathways and that individual domains of an RNA may form at rates that differ by orders of magnitude, with some transitions requiring minutes to reach completion[14]. Our synchrotron footprinting method, with the ability to monitor changes in the solvent accessibility throughout the RNA and monitor processes occurring from milliseconds to minutes, is ideally suited to investigate the complex folding of large RNAs.

The 385 nucleotide ribozyme derived from the Tetrahymena group I intron (**Figure 2**) folds into a well-defined tertiary structure in the presence of Mg^{2+}, which is required for activity[15].

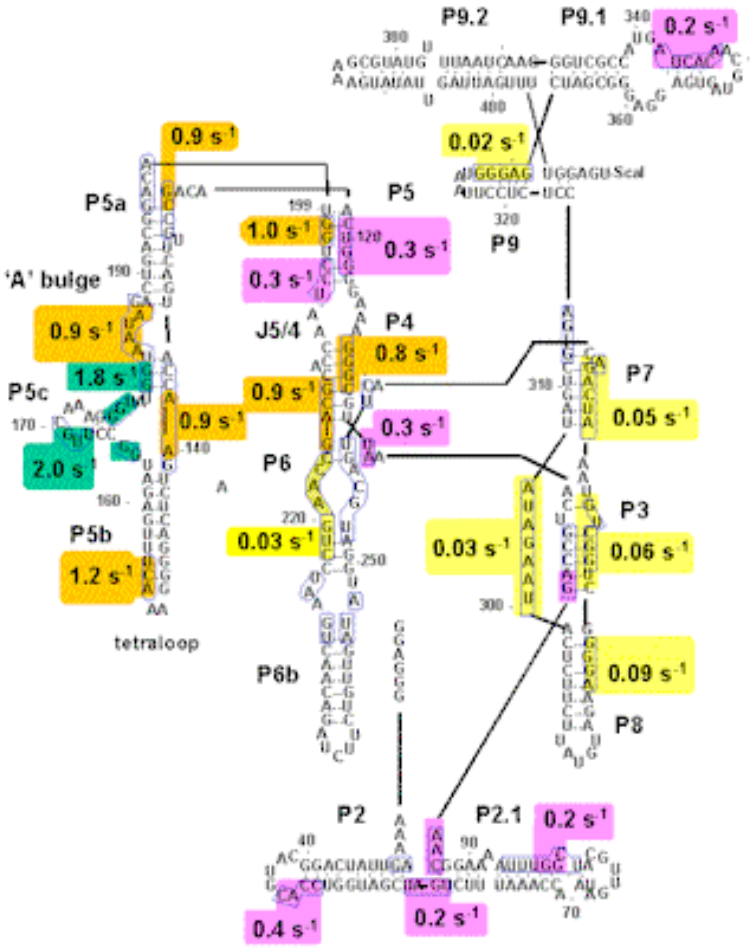


Figure 2: Secondary structure of the Tetrahymena ribozyme and rate constants for folding of 22 separate regions of the ribozyme that were determined by x-ray footprinting. Regions that fold on similar timescales are highlighted in the same color. P5c, the first region to fold, is highlighted in green. Immediately after, 7 regions within the P4-P6 domain fold simultaneously (orange). Subsequently, P2 and P9 make contacts (purple) with the pre-formed P4-P6 scaffold. After several minutes, protections appear in the P3 and P7 domains indicating formation of the native, active structure (yellow). [Editor's Note: NSLS Newsletters are printed in black and white. To view the true colors of this figure, please refer to the online version of this Newsletter through our website, ***http://www.nsls.bnl.gov****.]*

Biochemical and genetic methods have identified at least three domains of tertiary structure that, when separated, can re-associate to form the active ribozyme[15]. The Mg^{2+}dependent folding kinetics of the ribozyme were quantitated by determining the changes in solvent accessibility of individual sites as a function of time after the addition of magnesium ions. In **Figure 2**, we show the changes in solvent accessibility that occur for a specific set of nucleotides within the P4-P6 domain that became protected from hydroxyl radical cleavage within 100 ms after addition of Mg^{2+}. A powerful advantage of the footprinting method is that changes in protection in sites throughout the ribozyme can be monitored in a single experiment. **Figure 2** summarizes the kinetic determinations of folding for 22 separate regions of the ribozyme that show significant changes in protection upon addition of Mg^{2+}. The rapidly protected sites corresponded to nucleotides that are excluded from solvent by folding of the P4-P6 domain upon itself; the tertiary interactions within the domain are established in a concerted manner at a rate of about 1 s^{-1} at 42 C (colored in orange). A subset of nucleotides in P5c (**Figure 2**) were protected about twice as rapidly as other regions in the P4-P6 domain (colored in green). This region is a Mg^{2+}-rich subdomain suggesting that formation of a "metal ion core" in P5a-P5c could serve as a nucleation site for additional tertiary structure formation. Other regions highlighted in **Figure 2** show that tertiary contacts with the other domains of the ribozyme occur more slowly (colored in purple).

These synchrotron footprinting results yield a complete kinetic "map" for the folding of the ribozyme allowing detailed questions about the folding mechanism to be answered for this and other large RNAs. The methods used for analysis of

RNA structure are immediately translatable into examining the evolving protections that occur upon binding of proteins to both DNA and RNA, and experiments to probe such interactions in real time are underway. The footprinting of proteins, to examine the time-resolved changes in protection of a protein molecule, either in a folding reaction or in macromolecular assembly processes, is also being addressed through synchrotron applications. Although detailed discussion of the technical challenges of these applications are beyond the scope of this article, the modification of proteins by hydroxyl radical occurs on timescales equivalent to those of nucleic acid cleavage, use of advanced analytical techniques, such as mass spectrometry, can monitor these modifications and provide new probes of macromolecular structure.[7,8]

Please address any correspondence concerning this article to Dr. Mark Chance, at email address, mrc@aecom.yu.edu. Dr. Chance may also be contacted by telephone, (718) 430-4136 or by fax, (718) 430-8587.

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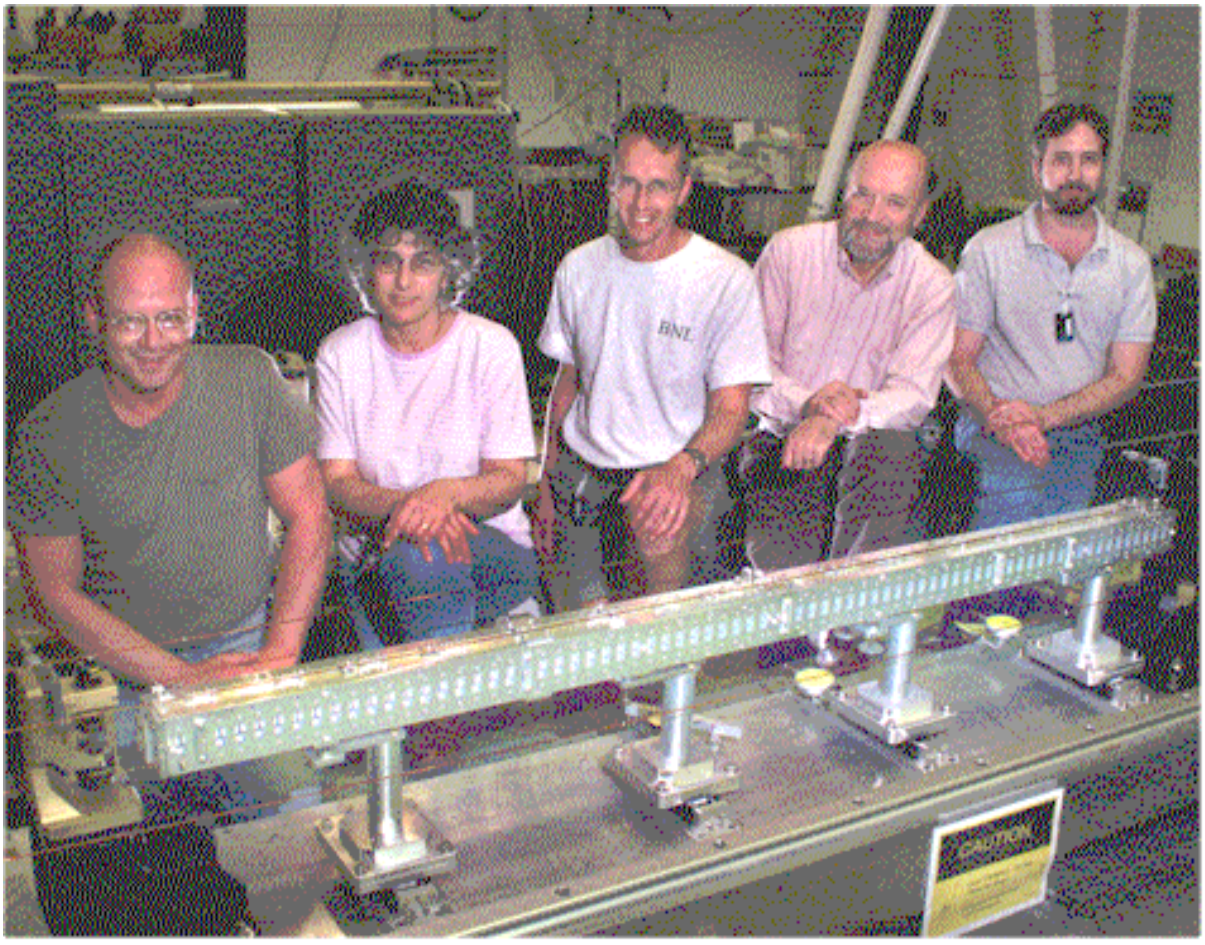
July 1999 NSLS Newsletter

FOCUS ON . ..... Magnetic Measurements Group

Lorraine Solomon

Tucked away in the northern end of Building 727 is the lab of the Magnetic Measurements Group of the NSLS. This group serves a broad range of functions relating to the magnets in use (and in planning) at the NSLS X-Ray ring, VUV ring, Accelerator Test Facility (ATF) and Source Development Laboratory (SDL) which can include any or all of the following: magnet specifications, magnet modeling and design, construction, testing, magnetic field measurement, and magnetic field shimming. The main focus of the group is magnetic field measurements, and to this end, various measurement techniques are available depending upon the magnet and the goal of the measurements.



Pictured (left to right), Mike Lehecka, Lorraine Solomon, Bill Graves, George Rakowsky (Group Leader), and Jeff Aspenleiter.

Almost all of the magnets in use throughout the rings at the NSLS, ATF and SDL, have been worked on and measured in the Magnetic Measurements Lab prior to their installation. These include dipole, quadrupole, sextupole and trim magnets, as well as insertion devices such as X1, X21, X25 and IVUN (in-vacuum undulator). The specific measurements vary depending upon the magnet and its application.

The test plan can include field homogeneity measurements, determination of the magnetic axis of a device, measurement of the magnetic multipoles, and correction of the measured field. The latter function can be accomplished through some redesign or re-engineering of the magnet, or through shimming of the magnet to subtly shape and modify the field. Presently, many of the magnets that we work on in the lab are novel devices, such as undulators or wigglers. These devices are typically either pure permanent magnet devices, or hybrid devices, which use permanent magnet material in conjunction with steel poles to shape the field. The minimum requirement for all of these magnets is that the device must be transparent to the general user, which means that both the angle and the position of the electron beam must be unchanged after the beam traverses the device. These parameters are directly related to the total first and the second integral of the magnetic field of the device. Generally, however, for optimal operation of the device, the details of the trajectory (i.e., the local first and second field integrals) through the device are crucial, and considerable work goes into tuning these in the lab.

There are three basic techniques used to measure magnets in the Magnetic Measurements Lab. One is point by point field measurements with a Hall probe. This data is directly integrated to yield angular and trajectory information. Another is moving taut wire measurements. The taut wire is one leg of a wire loop that is moved in the magnetic field to be measured, resulting in a change in the flux which crosses the loop, and an induced voltage signal which is read through an integrator. This signal does not yield detailed spatial information, but is quick, accurate and easily yields multipoles of the field. The third technique is a pulsed wire measurement in which a current pulse is imposed onto a wire strung through the magnet. This current pulse results in a distortion of the wire in the environment of the magnetic field in the device, causing a wave to propagate along the wire. The wave is detected photo-optically as it travels down the wire, producing an instant "snapshot" of either the electron trajectory or the electron angular deviation, depending upon whether the current pulse is a step function or an impulse. This technique is very useful for quickly assessing field quality, for fine tuning the trajectory in undulator magnets, and for locating the magnetic axis in quadrupole, sextupole, and solenoid magnets.

A lot of the Magnetic Measurement Lab work is associated with specific NSLS projects. For example, we reworked and re-measured the mini-undulator, which had been used in the X13 straight section for many years, for use as the modulator in the HGHG (High Gain Harmonic Generation) experiment at ATF. For the same experiment, we also designed and measured the dispersion magnet, and measured the Cornell undulator. More recently, considerable time and effort has gone into work on the VISA undulator magnet, which will be used in an FEL (free electron laser) experiment at the ATF. When it is moved to the SDL, two more sections will be added. This experiment is a joint collaboration between BNL, SLAC, LANL, UCLA, LBL, and LLNL. This magnet consists of four 99-cm long pure permanent magnet undulator sections that are mounted inside the vacuum chamber at the ATF. Tolerances for overlap between the electron beam and the photon beam impose tight magnetic and alignment tolerances of 50 microns. In order to meet these tolerances, pulsed wire measurements are made, the magnet sections are shimmed, and interferometer measurements are used to precisely measure the magnet alignment so that this alignment can be accurately and precisely reproduced at the ATF beam line. We are in the planning stages for measuring the NISUS magnet, to be used in the SDL facility. This magnet is 10 meters long, which poses unique measurement challenges. The Magnetic Measurements Group is also involved in planning for the future at the NSLS. In collaboration with many others at NSLS, we are exploring various possibilities for the next generation of insertion devices in the X-ray ring and for the fourth generation user facility, expected to be an x-ray free electron laser-based facility which will be built in ten or so years time when the Linac Coherent Light Source (LCLS) at SSRL R&D facility has delivered

its promise.

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July 1999 NSLS Newsletter

X-Ray Ring Update

Rich Biscardi

X-Ray Ring Manager

As most everyone knows, Roger Klaffky has left BNL. Roger had been at the Light Source for nearly 20 years. Recently, as X-Ray Ring Manager/User Liaison, Roger handled daily operational issues and user concerns while overseeing improvements to the X-Ray ring. As a friend and coworker, Roger will be missed. We wish him all the best in his new position at Los Alamos.

The jobs that Roger performed as X-Ray Ring Manager/User Liaison have been divided among three people. Rich Biscardi will serve as Ring Manager, dealing with the technical aspects of running and upgrading the X-Ray ring. Zhong Zhong is the interim User Liaison. Zhong will run the users' meetings and deal with user concerns. A third person will be hired in the upcoming months to deal with the physics issues which arise in operating and upgrading the X-Ray ring.

As reported in the previous NSLS Newsletter, the second rf cavity had to be returned to ACCEL of Germany for replacement of the defective center electrode. The old electrode has been successfully removed and the newly-forged billet has been received from the forging company. The rf cavity is now at KME, ACCEL's subcontractor, for rough machining. The projected delivery date is mid-to-late August.

The new X-17 wiggler is still at Oxford Instruments undergoing repairs. The leak between the liquid nitrogen and liquid helium reservoirs has been repaired, as has the electrical short in the power leads. After reassembling the wiggler, excessive heat loss in the cryostat was detected by Oxford technicians. This was followed up by a series of tests to isolate the sources of the heat leak. The problems have been isolated and Oxford is correcting them. We do not expect to receive the wiggler from Oxford until late spring or early summer, but we still plan to install it during the December 1999 shutdown as previously reported.

Progress continues on low emittance lattice operation at 2.8 GeV. A vendor has been chosen for the 700 Volt, 1000 Amp sextupole magnet supply needed for this upgrade. We expect the supply will be delivered by the end of September. Temperature measurements on a spare sextupole magnet are complete. Only moderate temperature rises were noted at 1000 amp operation. New high-current cables have been ordered and will be received sometime in June. The Mechanical Group is designing a new cable-magnet connection that is rated for the higher current operation.

Installation of the cables and retrofitting of the cable-magnet connectors will be started during normal maintenance periods by the electricians, electrical, and mechanical groups. It is expected that all work will be completed by the end of the December 1999 shutdown.

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July 1999 NSLS Newsletter

User Administration

Mary Anne Corwin

User Administrator

NSLS 1998 Activity Report

The 1998 NSLS Activity Report has been completed. The hardcopy version contains Sections One through Seven in printed form and all abstracts are presented on CD Rom attached to the back of the report. Copies are available at the NSLS User Administration office.

The electronic version, in PDF (Portable Document Format), is available on the web at: **http://www.nsls.bnl.gov/Pubs/ActivR/AR-98/Ar98toc.htm** . Acrobat Reader, which is required to view the documents, can be downloaded through the above URL.

Congratulations to Eva Rothman, Jerry Hastings, Linda Feierabend, Lydia Rogers, and Nancye Wright on a job well done in producing the Activity Report. It is proof of another year of hard work and dedication.

Guest/User Database

The new guest/user database, programmed in Oracle, is expected to be fully operational by the time this edition of the newsletter is distributed. The database was put online in April as a test, running parallel with the Informix database. Several critical problems were noted during that test and were corrected. As of the writing of this article, plans are to run a second parallel test from June 2 - 11. If all goes well, we will continue on the new system and archive Informix. The Oracle guest/user database was designed to be more user friendly and provide reports more easily.

Safety Approval Form

Plans have been implemented to update the safety approval form (SAF) database and system to make it more "user friendly" for users and administrators. In May, a consultant was hired to modify the database and the design of the online pages. Some of the more important improvements include:

(1) linking the SAF database to the new Guest/User database so that users who have BLOSA training on a particular beamline will be noted on the SAF;

(2) building in searches so that users will no longer be required to remember their serial number and administrators can locate SAFs more rapidly;

(3) extending the "time out" feature of the system; and

(4) adding a utility to archive expired SAFs. These are just some of the 80-odd enhancements planned for modification.

In May, User Administration received two requests for assistance concerning problems with the SAF approval form. Both problems, described below, were resolved as follows:

1. ***MAC Users****:* Experimenter could not get a new number assigned and could not get to the main page. The experimenter is using a Mac and his browser was Explorer. We have found that this combination can cause problems. Fortunately, the experimenter also had Netscape. After suggesting he try using Netscape as his browser for this form, he had no further problems.

2. ***Looping Back to Default Page****:* Experimenter could not get to the main page and kept looping back to the default home page. After going step by step through the process with her, the experimenter stated she was prompted with a pop up box requesting her to "Continue." The problem turned out to be incorrect Netscape preference selections. It was discovered that the experimenter's *cookies* were disabled. *Motto of the story: Accept your cookies!*

New Security Measures and Its Impact on Foreign Nationals

New security measures have been implemented by the Department of Energy affecting access to BNL (and many other Department of Energy facilities) by all foreign nationals. The new measures require background checks for foreign nationals from sensitive countries and pre-visit approval for foreign nationals from both sensitive and non-sensitive countries require that Form IA-473 be submitted to NSLS prior to arrival at BNL (in some cases, 90 days in advance).

The procedure requires the visitor's/assignee's submission of Form IA-473, "Request for Foreign National Unclassified Visit or Assignment" to NSLS. In turn, NSLS forwards a copy of the form to Safeguards & Security at BNL. If an indices check is required, Safeguards & Security then submits the form electronically to DOE. Under normal circumstances, this approval process can take up to eight weeks to complete. As BNL is not the only laboratory required to comply with these procedures, initially, there will be an influx of indices checks to be performed and there may be delays in receiving approval. Once approved, an indices check is effective for two years.

Form IA-473 must be approved by BNL prior to entering Brookhaven National Laboratory site. Therefore, we are asking that all foreign nationals who plan to work at the NSLS complete Form IA-473 and submit the form to the User Administration office as quickly as possible

The form and instructions can be found at our website, **http://www.nsls.bnl.gov/foreign/foreign.htm**. This requirement applies until further notice. Please revisit our home page or the above URL periodically since the possibility exists for some modification to this requirement for BNL users. If and when that occurs, the User Administration office will release the appropriate announcement immediately. Please communicate this information to your staff, employees, collaborators, and users. Thank you.

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VUV Status Update

Stephen Kramer

VUV Ring Manager

The VUV ring has been performing quite well since the Dec-Jan shutdown of the ring. The average beam availability during scheduled operations has been 99.9% for the last three months. The beam lifetime is still down slightly compared to the record values in November 1997.The cause of this reduced lifetime is still unclear.

Improvements in the ring performance have been in the area of the low frequency fluctuations of beam intensity, seen by the infrared beamlines. These frequencies range from around 50 Hz up to several KHz and are not seen by most beamlines, but limit the resolution level for experiments on the Fourier Transform Infrared beamlines. A task force has been setup under Jim Murphy to study this problem and to propose solutions. Considerable effort is being expended to understand the sources of the low frequency vibrations and how they couple into the user's signal. These sources are from motors, but the obvious sources of the high power water system pumps turned out not to be the dominant source of the vibrations as measured by accelerometers on the experimental floor. The largest sources in this frequency range appear to be motors on the experimental floor itself, including the large number of user vacuum pumps and chillers. However, improvements in the accelerometer measurements haven't always correlated with improvements in the beam signals. Understanding the source of this coupling has been quite difficult.

Studies have developed several new capabilities for operation of the ring. The short bunch lattice has been used several times for timing experiments on the ring. This lattice provides a bunch length of half the value for the normal achromatic lattice with only a small reduction in beam lifetime. This small decrease in lifetime results from the increase in the horizontal beam size for this new lattice. The RF Group has provided a new mode of operation where the harmonic RF system actually shortens the bunch length rather than stretching it for increased lifetime (normal operations). This capability will help keep the bunch length short as the current is increased. Operations with bunch shortening has been used up to 600mA where an instability causes a fast beam loss. While studying these new capabilities, the VUV ring succeeded in storing beam in two stable RF buckets using only one RF cavity. This was achieved by controlling a nonlinear focusing force to create the second stable potential well within the ring vacuum aperture. This possibility was predicted more than 40 years ago in proton accelerators but has never before been demonstrated so clearly in an accelerator. These two beam bunches, demonstrated in the synchrotron radiation light monitor image shown in **Figure 1**, have a small and controllable energy and time difference. The two beam spots are separated in space due to the energy difference between the two stable buckets and the energy dispersion of the storage ring lattice. This achievement was reported at the 1999 Particle Accelerator Conference at New York City in March. This result, in addition to providing a direct measurement of the nonlinear dynamics of the accelerator, could also provide a unique capability for synchrotron radiation users. The energy difference between the two bunches provides two colors of light from an undulator. This was measured for each bunch individually by Hyun-Jo Kim using the undulator on the U5 beamline, as shown in **Figure 2**. The electron bunch energy difference of 0.64% yielded a 1.28% photon energy difference, as measured at the 3rd harmonic energy of 85 eV. Future studies of these nonlinear beam bunches will be performed in order to better understand the nonlinear dynamics of the ring and to develop these unique beam properties for user operations.

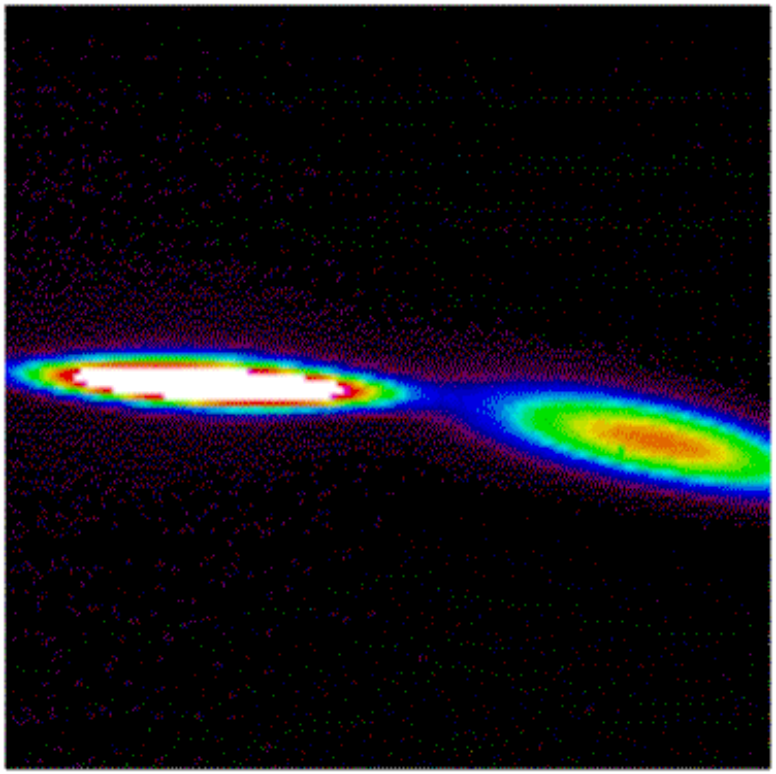

Figure 1

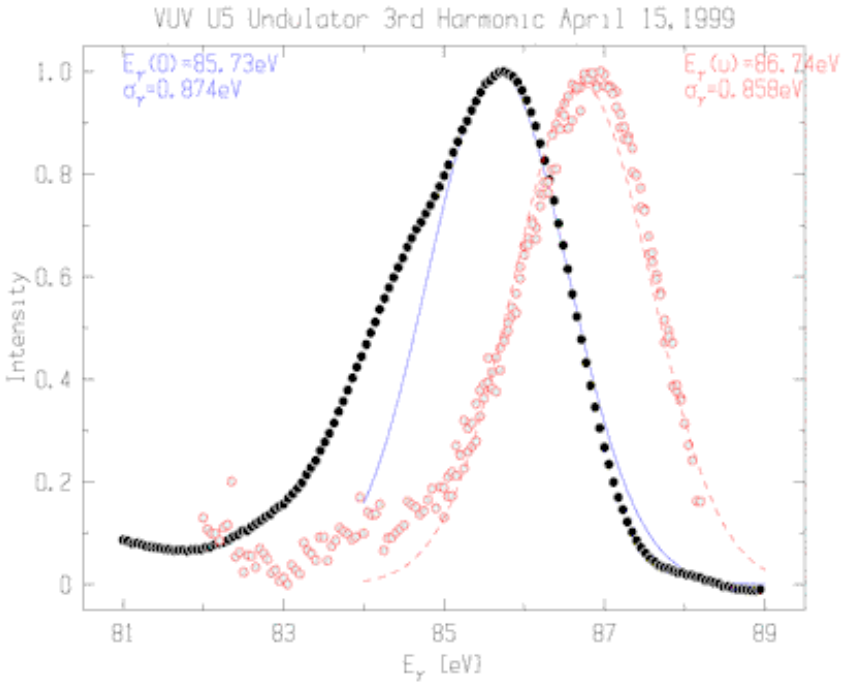

Figure 2

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Identification and Tagging of Equipment

The Department of Energy requires that all capital equipment 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 below).

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, during the inspection, untagged equipment is found, a tag will be applied.

If you need assistance or have any questions, please call Donna Buckley at X3599.

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CALL FOR GENERAL USER PROPOSALS

Deadline for proposals and requests for beam time on the NSLS X-Ray and VUV Rings is

Thursday, September 30, 1999 for scheduling Jan - Apr 2000

Prior to Submitting a Proposal

You must contact the beamline personnel responsible for the beamline(s) selected in order to verify technical feasibility on the beamline(s) and discuss any special arrangements for equipment. Your chance of getting beam time is improved by being able to use more than one beamline.

Preparing Your Proposal

The same form is used for both new proposals and beam time requests against existing proposals. Follow the instructions on the information sheet and complete and submit all the required sections. Type or print all information legibly. MAIL OR FAX **ONE COPY** of the proposal form and any attachments to the NSLS User Administration Office. Only **one copy** is required - *do not mail a hard copy if you have already faxed one to us.*

Special Notes for Macromolecular Crystallography (PX)

NEW PROPOSALS: The proposal represents a two-year program. Provide an overall plan for your research according to the instructions on the proposal form. If you can, estimate the number of crystals you plan to measure over the two years. If you require the use of an insertion device beamline like X25, be sure to indicate your need for the enhanced performance. New proposals must also include your plans for the upcoming cycle for which you are requesting time (below).

BEAM TIME REQUESTS: Be specific about what you plan to study in the upcoming cycle. Submit PX Forms only for the crystals you plan to study in that cycle. Answer all the questions, use the back of the form if you need more space. Be clear about what crystals you already have, which you expect to have, and how you would use the beam time you requested if you were unable to obtain the planned crystals in time (i.e., other crystals described in your program).

Proposal Deadline

The complete proposal package must be received by the User Administration Office on or before 5:00 pm Eastern Time Thursday, September 30 in order to be considered for the January - April cycle. **The fax machine is always extremely busy on the deadline date; please do not rely on faxing the proposal successfully on September 30**. We encourage submitting new proposals by mail or fax prior to the deadline. Beam time requests for active proposals will be accepted after the deadline, but will be allocated beam time only after requests received on time have been allocated. Late requests are not eligible for a rating upgrade if beam time could not be allocated to them.

Each proposal will receive a prompt preliminary review to verify that it is complete and legible. If there is a problem with the proposal, you will be contacted immediately. Submitting your proposal well in advance of the deadline date assures that the User Administration Office has time to reach you and that you will have enough time to correct any deficiencies.

Additional Information and Forms

Blank proposal forms and instructions are available on the World Wide Web at **http://www.nsls.bnl.gov/Proposal/wordfiles.html** . A guide to the NSLS beamlines and more information about the General User Program can be found through our homepage, **http://www.nsls.bnl.gov** or by contacting E. Pinkston or L. Rogers in the NSLS User Administration Office. Office hours are Monday through Friday, 8:00 a.m. to 5:00 p.m. Eastern Standard Time (EST). Contact information is on the back page of this Newsletter.

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Safety Approval Form

Reminder: Submit via the Web ***at least one week before*** your scheduled beam time. Do not send in a SAF with your proposal. ***Complete all fields*** on the SAF Web form - use "NA" or "not applicable" if you need to, but answer each question! The SAF can be found at: **http://130.199.76.30/safety/**

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Form IA-473 for Foreign Nationals

Submit by fax or mail prior to arrival at BNL (in some cases, 90 days in advance). ***Complete all fields*** as indicated in the instructions. Form IA-473 and instructions can be found at **http://www.nsls.bnl.gov/foreign/foreign.htm**

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Stop-Work Procedures at BNL

Reminder...The Laboratory recently established new procedures for issuing "Stop-Work" orders for situations involving "Imminent Danger" or violations of Radiological Requirements. All BNL employees are being trained on these procedures, as well as all guests who hold appointments to work in our facilities.

Any users who receive the NSLS Safety Orientation or request radiation dosimeters in User Administration will receive this brief training. The training involves reading two short pamphlets and answering 5 multiple-choice questions.

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CALL FOR EXPERIMENTAL SUMMARIES (ABSTRACTS) SYSTEM

FOR THE 1999 NSLS ACTIVITY REPORT

http://www.nsls.bnl.gov under Experimental (Abstract) Submission System

E-Mail and Disk Submissions Accepted Until September 25, 1999:

Anyone who decides not to use the ESS (Electronic Submission System) this year, for whatever reason, can still submit a via E-mail or on disk (WordPerfect, Mac or PC Word file, or ASCII text). The catch is that your submission must be post-marked no later than September 25, 1998. We would like to accommodate those who prefer submitting without the ESS, but because it introduces extra processing on our end and an extra round of editing on your side, we must limit thesubmissions to this time period.

ESS Submissions Between September 7 and October 29:

* NEW submissions accepted until 5 pm Friday October 15.

* EDITS to existing submissions allowed until 5 pm Friday October 29.

Detailed information and instructions can be found on the Web on the ESS pages (http://www.nsls.bnl.gov, under **Experimental (Abstract) Submission System**. Note that many people are going to submit at the last minute and the system will be extremely busy and slow near the deadlines. There is not much we can do to change this system limitation - please save yourself anxiety and try to submit earlier!

After October 29, 1999:

We must enforce this strict deadline in order to produce the Activity Report according to schedule and in time for the Users' Meeting. October 29 is a hard deadline. Although the system will remain open, any submissions entered after October 29 will be automatically rolled over into the 2000 Activity Report. The November Newsletter will have information about when the abstracts will be available for viewing on the Web.

CALL FOR PUBLICATION REFERENCES

for the period

September 30, 1998 through December 31, 1999

Our publication references are an important element in how funding agencies judge the productivity of the NSLS. All NSLS users are obligated to send the NSLS a complete publication reference for any paper based in whole or in part on research done at the NSLS. Each beamline PRT submits a list of their own papers as part of their Annual Beamline Progress Report, but the NSLS must also collect references by General Users and other collaborators in order to present the complete picture of the work being done here.

Please take a few moments to e-mail or fax us your publication references. If you are not sure whether you had already sent us your list, send it in anyway - we always check on this end to avoid double-listings. Send your references to Nancye Wright at wright1@bnl.gov or FAX 516-344-7206.

DO YOUR PART TO SUPPORT THE NSLS!

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Important Upcoming Dates

Sept. 2, 1999	Deadline for submissions, November Newsletter
Sept. 30, 1999	Deadline for General User Proposals (Jan. - Apr. 2000)
Oct. 15, 1999	Deadline for Experimental Summaries (Abstracts)

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