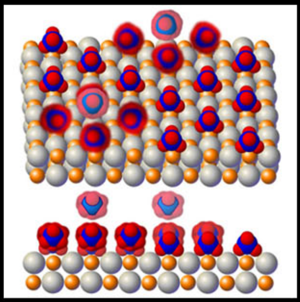

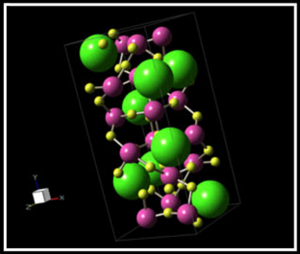

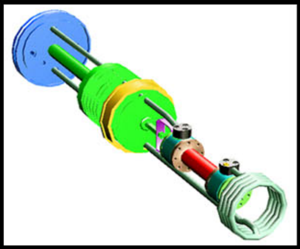

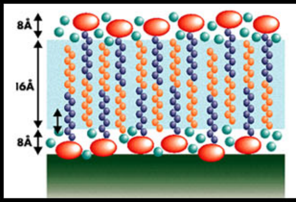

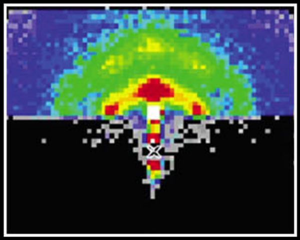

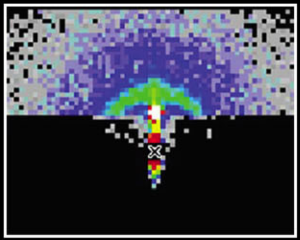

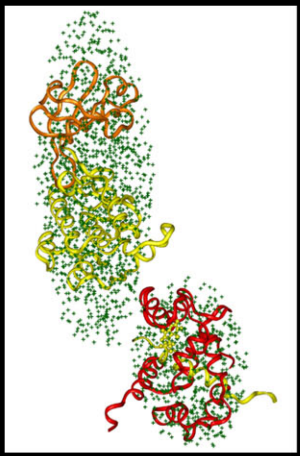

NSFChemBio

Neutron Scattering for Chemistry and the Chemistry/Biology Interface

Workshop Report

Joint Institute for Neutron Sciences Workshop Series
Florida State University, Tallahassee, Florida
September 23-25, 2003

Sponsors

National Science Foundation
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EXECUTIVE SUMMARY

The U.S. neutron users' community is eagerly anticipating the commissioning and operation of beam lines starting in 2006 at the Spallation Neutron Source (SNS) (for more information, see www.sns.gov), currently under construction at Oak Ridge National Laboratory (ORNL). In addition, ORNL's upgrade of the High Flux Isotope Reactor, and associated new and upgraded instruments there, is eagerly awaited. Opportunities exist for wonderful scientific advances with neutron scattering and spectroscopic investigations by chemists and by biologists working at the chemistry-biology interface. However, the number of active neutron users in the United States among these communities is currently small, and the communities as a whole had little opportunity to communicate their interests and needs before the workshop on Neutron Scattering for Chemistry and the Chemistry-Biology Interface (NSFChemBio) and the overlapping workshop on Sample Environments for Neutron Scattering Experiments (SENSE). Most of these needs will be of interest to the other U.S. scattering facilities as well: the Center for Neutron Research at the National Institute of Standards and Technology, the Intense Pulsed Neutron Source at Argonne National Laboratory, and the Lujan Center for Neutron Scattering at Los Alamos National Laboratory. The Joint Institute for Neutron Sciences (JINS), an intellectual center for the neutron sciences, played a leading role in the organization of both the NSFChemBio and the SENSE workshops. Florida State University (FSU) partnered with JINS in the organization of the NSFChemBio workshop, and FSU and ORNL's Center for Nanophase Materials Science (CNMS) were partners in the SENSE workshop. Oak Ridge Associated Universities also provided financial support.

The two overlapping workshops were held at Florida State on September 23-26, 2003. The goals of these two workshops fit nicely within the National Science Foundation's themes of people, ideas, and tools. The goals accomplished the following:

- Informed the chemistry and chem-bio communities of opportunities—instrumentation and supporting facilities—currently planned for SNS and CNMS, which will be adjacent to and connected to the SNS Central Laboratory Office Building:

    - Tutorial-style presentations covered the fundamentals of neutron scattering and spectroscopy, a "Neutrons 101" approach that assumed little familiarity with the subject.
    - Presentations on important scientific questions and the role that neutrons can play in answering them were drawn from areas such as catalysis; materials for energy production and storage; electrochemistry; magnetism; structure and dynamics in liquids, glasses, complex fluids, synthetics, and biopolymers; molecular behavior under confinement and near interfaces; and the role of weak interactions and molecular architecture in self-assembly on multiple length scales.

- Solicited the community's ideas on the needs for:

    - Instrumentation, including research and development themes such as detector development.
    - Sample environment development (addressed by the overlapping workshop, SENSE).
    - Time-resolved neutron scattering.
    - Preparation of deuterated or other isotopically substituted compounds, of both low and high molecular weight: is a centralized or distributed research resource needed?
    - Data analysis suites that integrate modeling and simulation.
    - Education in designing, executing, and analyzing scattering experiments; remote access and service-mode.

- Identified tools needed and outlined a path to realization via the formation of concept teams to develop science cases and funding proposals for:
  - Instrumentation.
  - Sample environments.
  - Supporting lab facilities and instrumentation (e.g., deuteration, X-ray diffraction (XRD), nuclear magnetic resonance (NMR), microscopies).
  - Best practices for education of new users—short courses, summer schools, curriculum development at the graduate and undergraduate levels.
  - Fellowship and research participation programs.

This report focuses on outcomes from the NSFChemBio workshop. A companion report[1] focuses on the SENSE workshop. The combined attendance at the workshops was 160 participants, with many attendees who are not currently users of neutron-scattering techniques. Participation from abroad was significant. U.S. academic institutions represented ranged from primarily undergraduate institutions through research-intensive universities and included universities that serve underrepresented minorities. There were participants—and exhibits—from industry, as well as from government laboratories. Details of the workshop structure and agenda are presented in Appendix A.

In addition to tutorial-style talks on the basics of neutron beam production and delivery to instruments optimized for a range of neutron-scattering techniques, the participants heard talks on the exciting science being accomplished in the chemical sciences and at the chemistry/biology interface using neutron scattering. In four breakout sessions, opportunities and needs of current and prospective neutron users were discussed and a series of findings and recommendations were provided concerning the following: (1) support facilities needs for soft matter; (2) needs for hard matter: support facilities, sample environments, and isotopic labeling; (3) deuterium labeling; and (4) education and outreach. Five additional breakout sessions, organized by the SENSE Workshop, focused on needs in the area of sample environments.

Attendees expressed strong support for community-initiated efforts to incite proposals in each of these areas. The need for facilities – perhaps located at multiple sites - for isotopic labeling, especially hydrogen/deuterium substitution, is especially urgent. Prompt action is also needed to provide space, equipment and staff for laboratories that support users' needs for sample preparation, manipulation, and environmental control, and for the use of auxiliary characterization techniques. The initiative to seek funding for both labeling facilities and supporting laboratories must come from the community. In addition, both the hard and soft condensed matter communities are excited by the prospect of the neutron vibrational spectrometer (VISION) that is proposed for SNS becoming reality.

[1] The report of the SENSE Workshop can be found at www.sns.gov/jins/jins.htm or www.sns.gov/users/users.htm.

1. FINDINGS AND RECOMMENDATIONS FROM THE BREAKOUT SESSIONS

These recommendations focus on the community's needs in general at neutron-scattering facilities. A few are specific to the Spallation Neutron Source (SNS).

1.1 SUPPORT FACILITIES NEEDS FOR SOFT MATTER

Recommendations:

- Provide dedicated laboratory space for soft matter work; sample preparation and manipulation of specialized equipment for sample environments should be located near the experimental station. In this regard, it is believed that SNS does not have adequate space currently planned and should as soon as possible add a room off of the Target Building wall behind the instruments for small-angle neutron scattering and reflectometry to house such laboratory space.
- Provide laboratory-based X-ray equipment, both a small-angle X-ray scattering instrument and an instrument for X-ray reflectivity measurements. On-site access to this equipment is critical for a successful soft matter neutron-scattering program, particularly in biology.
- Provide the supporting services necessary to maintain sample environmental conditions *at* the instrument site on the floors. These services would include, for example, gas supply and vacuum lines; chilled water; ventilation of the sample area (e.g., via elephant trunk); and temperature, humidity, and pressure control for samples/sample environments before mounting and during measurements on the sample needs.
- Maintain ample clear space around the sample area to accommodate the growing number of specialized experiments that bring a large amount of ancillary equipment connected to the sample area.
- Provide 24-hour-per-day/7-day (24/7) service for transporting large objects to the sample areas.

1.2 NEEDS FOR HARD MATTER: SUPPORT FACILITIES AND SAMPLE ENVIRONMENTS; ISOTOPIC LABELING

Recommendations:

- Create a general deuteration/isotope materials facility for the production of deuterated and novel isotopically substituted materials that extends beyond biological and soft matter.[2]
- Create reasonably sized, dedicated technical/sample environment (SE) staff who are available 24/7 during neutron operations
- Build and locate SE preparation areas at SNS near instruments and amply equip them with appropriate devices such as glove boxes, hoods, and SE equipment, as detailed in the SE list.
- Develop SE technician training programs with local technical colleges in partnership with SNS and other institutions.
- Provide an adequate and trained environment, safety, and health (ES&H) and technical staff.
- Develop and sustain a centralized, 24/7 SE preparation and technical assistance center.

[2] Additional recommendations on facilities for deuterium labeling are found in the session report that follows this one.

1.3 DEUTERIUM LABELING (H/D SUBSTITUTION) AND ISOTOPIC SUBSTITUTION

The major requirements and recommendations for H/D-isotopic substitution are as follows:

- Provide facilities for in vivo and in vitro production of H/D-labeled proteins and other macromolecules.
- Develop methods for the synthesis of labeled polymers, peptides, phospholipids, surfactants, and other complex biomolecules and other small molecules of interest.
- Explore possibilities to coordinate development of labeling techniques and of additional facilities for soft matter science.
- Develop, maintain, and distribute improved systems and technologies for production and analysis of isotopically labeled molecules through creation of a virtual support network.
- Train research staff and students in the aforementioned techniques.
- Explore overlapping and complementary needs in the NMR and other communities.

1.4 EDUCATION AND OUTREACH

- Act as Science Scouts: the neutron-scattering community could grow if facilities would have members attend national meetings, give talks on neutron scattering, and in general act as advocates for neutron scattering by hooking up interesting science and scientists with neutron tools.
- Encourage instrument scientists and neutron scatterers to present their research in general sessions, not specific neutron sessions.
- Target funding for neutron experiments to both encourage people on the edge and bring visibility and facilitate the use of sources.
- Actively follow-up to neutron schools and events to make the most of interactions with students and to provided encouragement to the students.
- Consider ways to facilitate integrating neutron scattering into graduate and undergraduate curricula.
- Simplify data analysis and visualization so that neutrons can be a tool and entry to neutron science but that does not require a commitment to being a neutron scientist.
- Have focused schools offered often so that in any year a student can find a school on nearly every major topic (magnetism, hard matter, soft matter, structure, and dynamics).
- Facilitate training of graduate students by paying for six months to one year at a facility for work as technical staff to an instrument scientist while conducing his or her doing their own scientific studies.

2. WORKSHOP BACKGROUND AND CONTEXT

The following points show why neutrons are such useful probes of structure and dynamics in matter and why neutron scattering and spectroscopy provide such a powerful complement to other scattering and spectroscopic techniques.

- Neutron cross sections exhibit no regular dependence on atomic number and are similar in magnitude across the periodic table, giving rise to sensitivity to light elements in the presence of heavier ones.
- Certain large differences in isotopic scattering cross sections (e.g., H/D) make neutrons especially useful for study of light atoms in materials.
- The range of momentum transfer available allows probing of a broad range of length scales (0.1 to 10^5 Å), which is important in many different materials and applications.
- Thermal and cold (longer wavelength) neutrons cover a range of energies sufficient to probe a wide range of lattice or magnetic excitations, molecular vibrations, rotations, and “slow” dynamical processes such as polymer chain reptation.
- Neutrons have magnetic moments and are thus uniquely sensitive probes of magnetic interactions.
- Neutrons can be polarized, allowing the cross sections (magnetic and nonmagnetic) to be separated.
- The simplicity of the magnetic and nuclear interactions make interpretation of results straightforward.
- Neutrons are electrically neutral leading to penetration depths of centimeters, thereby enabling in situ studies and the study of samples in extreme environments.

Figures 1 and 2 show the scientific landscape that can be explored using neutron scattering and spectroscopy. The landscape is presented in terms of capabilities: momentum transfer (Q), energy transfer (ω), and resolution. Coverage is included for selected instruments at SNS. The web sites for other neutron-scattering facilities in the United States and elsewhere can be consulted for details of their instruments’ capabilities.

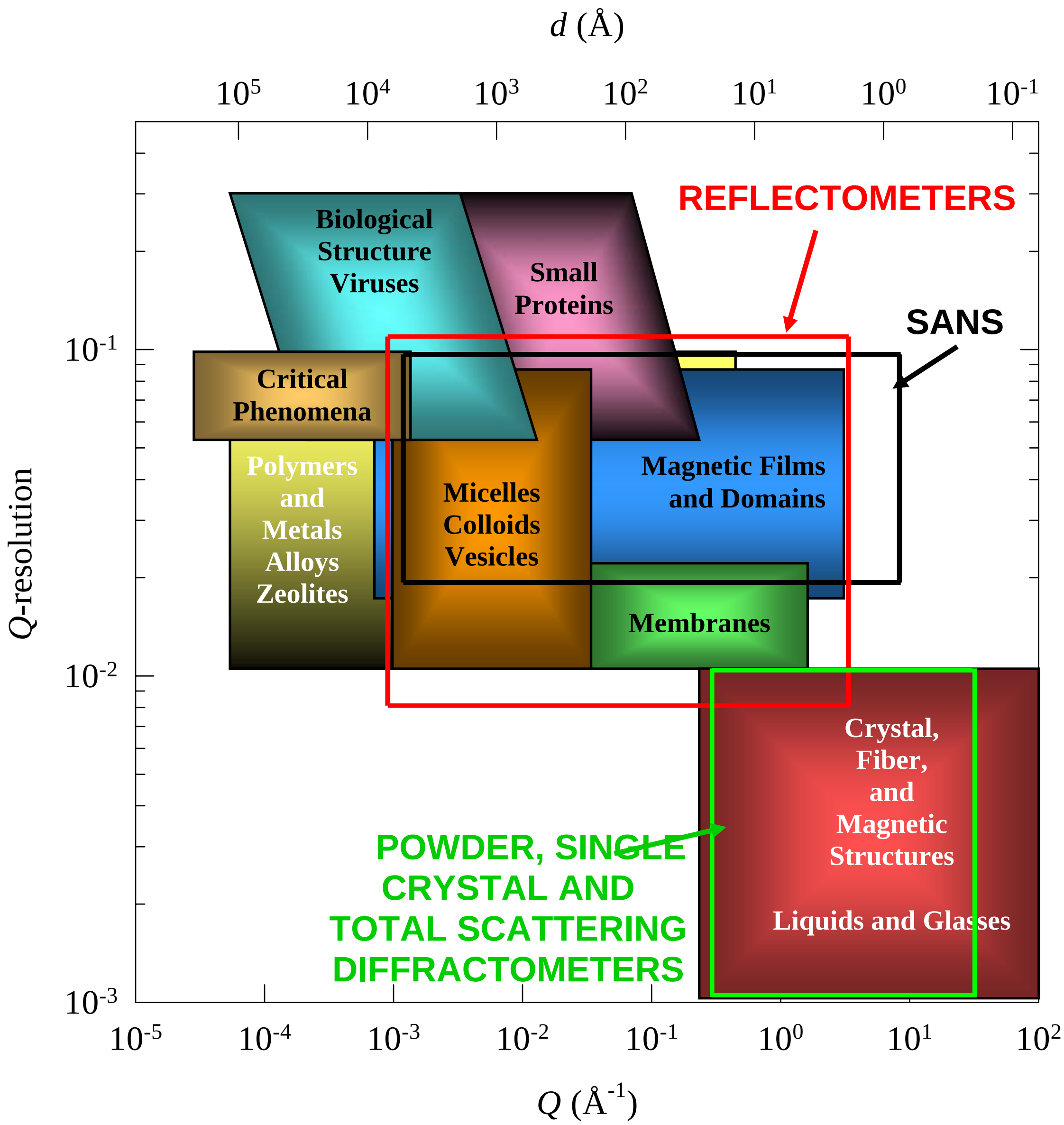

Fig. 1. Elastic neutron scattering: the study of structure.

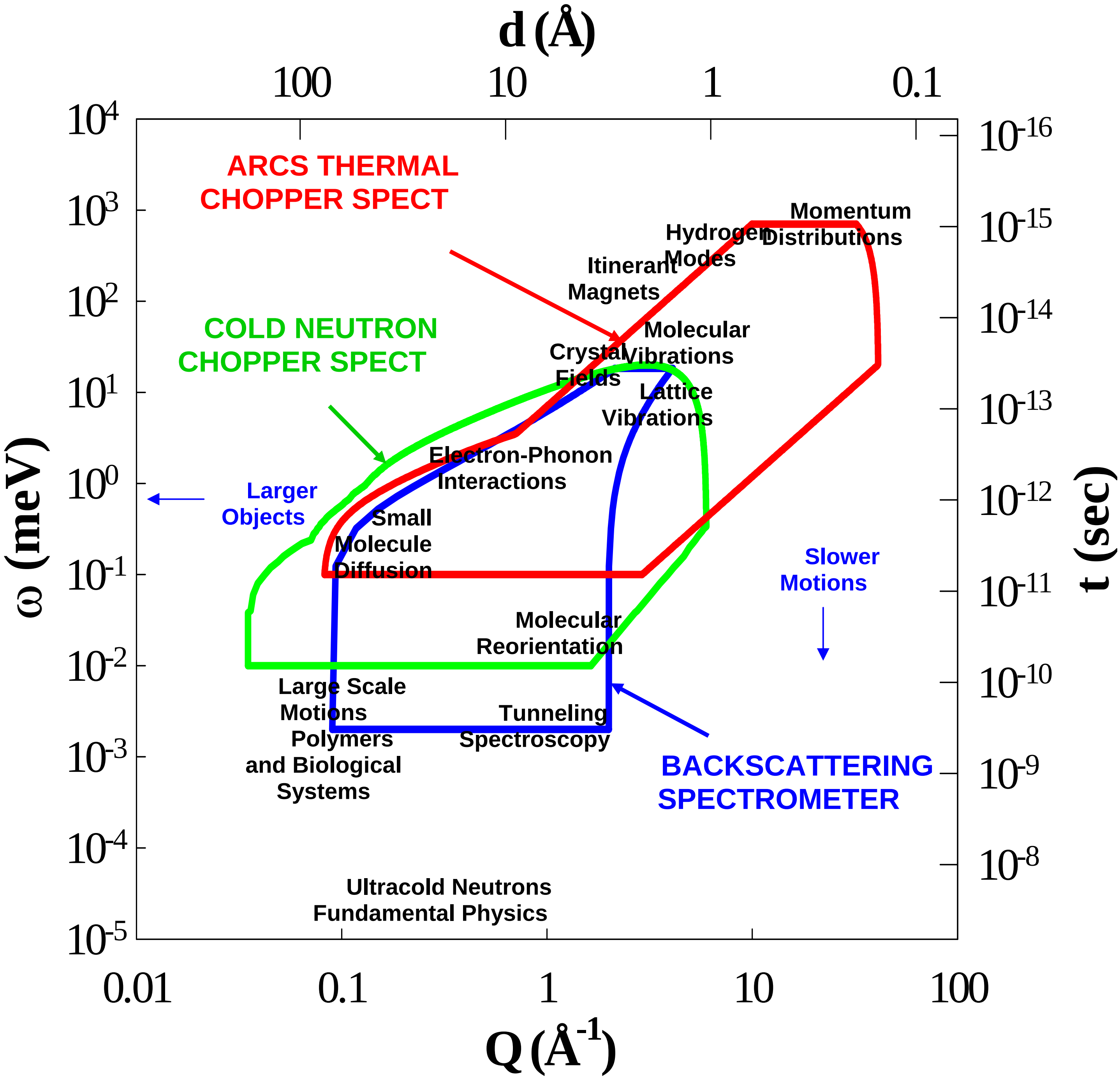

Fig. 2. Elastic neutron scattering: the study of dynamics. Adapted from Neutron Scattering Instrumentation for a High-Powered Spallation Source, R. Hjelm et al., LA0-UR 97-1272.

The number of beam lines that remain uncommitted on the first SNS target station is now only 8 of 24. The sixteen approved instruments are shown on the map in Fig. 3. All of the instruments used for structural determinations—diffractometers, the small-angle neutron scattering spectrometer, and the reflectometers—are of significant interest in the chemical and biological sciences. Many of the spectrometers used to study dynamics are of interest as well; the two for which funding is not yet identified are in that category: (1) VISION is of great interest for neutron vibrational spectroscopy applied to chemical and biological problems, as is (2) a neutron spin-echo spectrometer for studying dynamics from the picosecond to the microsecond timescale over a broad range of momentum transfer vectors. A spectrometer for macromolecular crystallography using neutrons—not yet on the map—has an SNS instrument development team (IDT) working on its full science case and desired performance characteristics. A second, long-wavelength target station, which may begin operation at SNS in 2013, is a priority in the 20-year facilities plan of the Department of Energy Office of Science. This second target will be instrumented with up to 22 additional spectrometers, many of them ideally suited to studying the large characteristic dimensions and low-frequency motions that are of interest in soft condensed matter.

Chemists and chemical biologists need to carefully assess whether the instruments being designed and constructed for the SNS first target are optimized to provide answers for the grand scientific questions of most interest to them and whether instrumentation is currently missing that is a high priority to the community. The NSFChemBio Workshop provided a venue for champions of such instruments to explore joining existing SNS IDTs or instrument advisory teams (see www.sns.gov) for instruments already on the map. The workshop also provided opportunities to consider building the science case for instruments not yet funded and/or still in the conceptual stage that are of interest in the chemical and biological sciences. The midscale instrumentation initiative in the president's FY 2004 budget request for the National Science Foundation (NSF) could provide the means to acquire instrument funding. The following quote from the National Science Board's report on "Science and Engineering Infrastructure for the 21st Century" (NSB-02-190) is relevant:

> "Examples of infrastructure needs that have long been identified as very high priorities but have not been realized include . . . beam line instrumentation for neutron science . . . In many cases the midsize instruments [the millions to tens of millions of dollars range] that are needed to advance an important scientific project are research projects in their own right, projects that advance the state-of-the-art or that invent completely new instruments."

The Office of Science and Technology Policy Interagency Working Group on Neutron Science was charged to investigate the issues facing (current and prospective) users of U.S. neutron facilities and what can be done to maximize the scientific impact of these facilities. In their June 2002 report, they listed as two of four priorities: "seek(ing) ways to fully exploit the best present neutron source facilities (including the SNS)" and "broadening access to these facilities for the benefit of the broadest possible scientific community." Strategies to maximize the scientific impact of neutron-scattering facilities in the chemical and biological sciences and broaden access to them were addressed by workshop attendees in four breakout sessions:

- Support Facilities Needs for Soft Matter
- Support Facilities Needs for Hard Matter
- Deuterium (and other isotopic) Labeling
- Education and Outreach

Some of the NSFChemBio breakout groups also addressed standard and specialized sample environments. In addition, the five breakout sessions in the companion SENSE Workshop focused on sample environment needs shared by all scientific disciplines that use neutrons to probe materials.

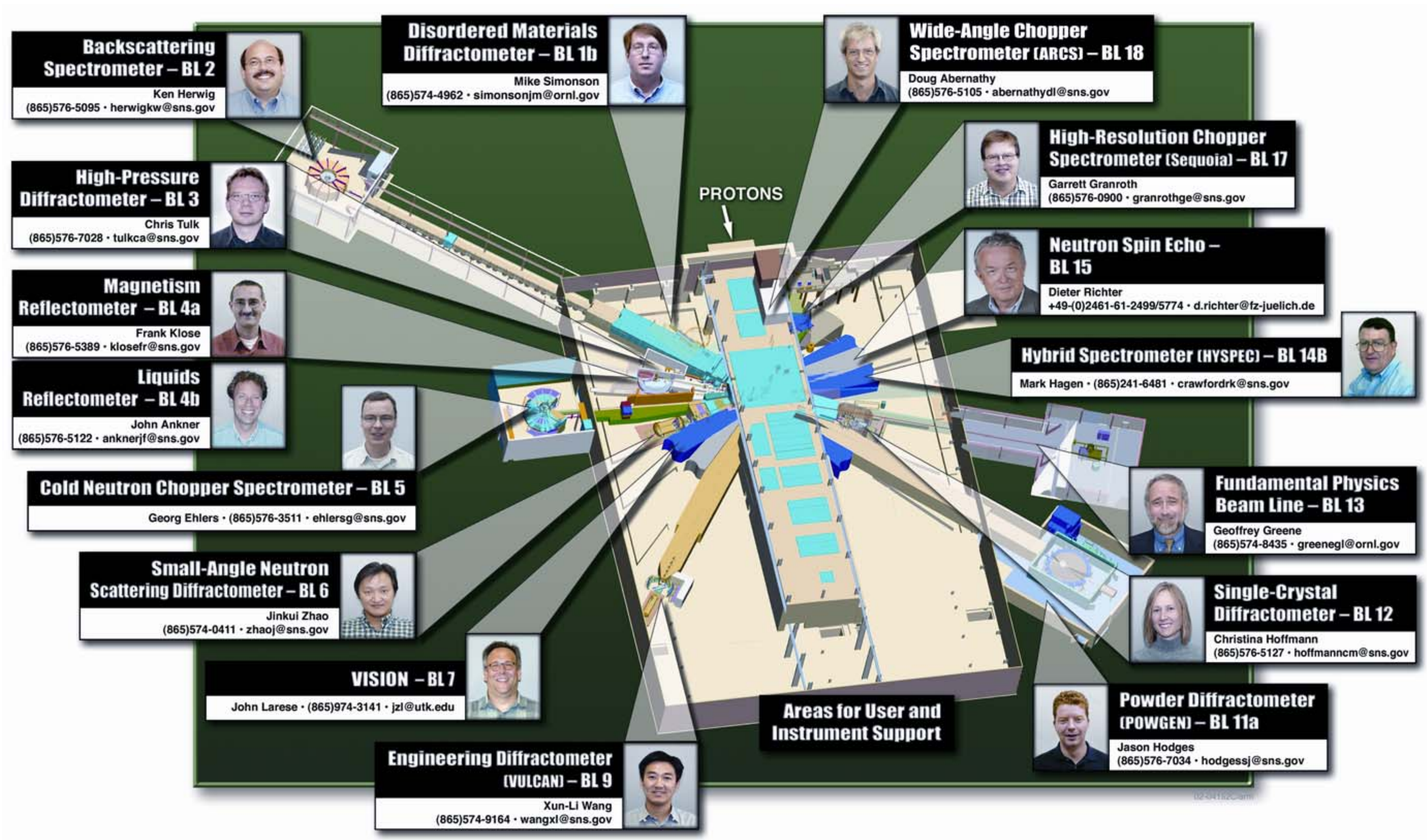

Fig. 3. Instrument scientists and instrument layout of 16 approved instruments at SNS.

3. BREAKOUT SESSION ON SUPPORT FACILITIES NEEDS FOR SOFT MATTER

Facilitators:[3] Joanna Krueger, University of North Carolina-Charlotte, jkkruege@email.uncc.edu
Paul Butler, Neutron Scattering Section, Oak Ridge National Laboratory, butlerpd@ornl.gov

Both the prospective and the experienced users of neutron beams from the soft matter (polymer and biological) sciences were given the opportunity to advise scientific leaders on the community's needs for on-site support facilities, such as sample preparation labs, characterization techniques, and computational support needs. The primary instruments that were discussed by this particular group were the small-angle neutron-scattering (SANS) and neutron reflectometry (NR) instruments. Specifically excluded from the discussion was on-line sample environment needs, as that was to be addressed in another session.[4] Two primary issues emerged: (1) laboratory–based, X-ray scattering/reflectivity instrumentation on site at the neutron facility is essential and (2) significant laboratory space on the floor in proximity to the instrument is critical, as is substantial access space around the sample area. In this vane, it was noted that the floor space geometry at SNS around the SANS and NR instruments is extremely constrained and it was the strong consensus of the group that no space currently available would be adequate. Thus, the recommendation is made to SNS that they plan as soon as possible to add a room off the wall behind the instrument to house such a facility. The following represents a fairly broad consensus on issues developed by those present.

I. There was a strong consensus that access to laboratory-based x-ray equipment is essential for users in the neutron-scattering community. Such access could potentially make the difference between a successful and a nonsuccessful experiment and most certainly would decrease the uncertainty of the neutron data refinement. It was also noted that new users might not have access to small-angle x-ray scattering (SAXS) or x-ray reflectivity (XR) instrumentation. Thus, access to such support facilities would not only allow all users to collect an additional set of contrast data at the same time (and with the same sample) as they collect the neutron-derived contrast data but could also be useful to new users in testing their systems before using expensive neutron beam time. In summary, the primary purpose of providing on-site x-ray facilities would be to ensure the quality of the specimens (for both reflectivity and diffraction), the isomorphism of fully protonated/specifically deuterated pairs of specimens (reflectivity and diffraction), the quality of inorganic multilayers to be used as reference structures in interferometric phasing of neutron reflectivity, and to provide an independent data set for the joint refinement of x-ray and neutron data (reflectivity and diffraction). Two independent instruments were recommended—one with reflectometer geometry (theta-theta diffractometer) that included a sealed tube source, Goebel mirrors, and a scintillation detector and one for small-angle x-ray scattering, possibly with a rotating anode and position-sensitive detector. Due to the complexity of these instruments, it is vital that they be under the care of a primary instrument responsible or technician.

II. It was deemed essential that a dedicated lab for soft matter be at the experiment level and adjacent to (or very near) the instrument. Besides sample preparation needs, the need for space to clean and store the variety of specialized sample environments was mentioned. Experiences from ISIS, the Institut Laue-Langevin (ILL), the Los Alamos Neutron Science Center (LANSCE), and the National Institute of Standards and Technology (NIST) were all cited. In this regard, it was deemed that SNS does not currently have adequate floor space, particularly given requirement III that follows. Thus, the recommendation is made to SNS in particular that they plan as soon as possible to add a room off the wall

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[3] David Worcester, Univ. of Missouri-Columbia and Jarek Majewski, Los Alamos National Laboratory, also contributed to the written report.

[4] The companion workshop, Sample Environment Needs for Scattering Experiments (SENSE), held break-out sessions that focused on sample environments. See that workshop report for more details www.sns.gov/jins/jins.htm or www.sns.gov/users/users.htm.

behind the instrument to house such a facility. It was noted that this is not really any different than putting building extensions for instruments onto the experiment hall. Types of equipment essential at this work station include Milli-Q water plant, a hood, Langmuir troughs (one to study neutron or X-ray reflectometry on the X-ray and neutron spectrometers and second to perform off-line isotherms measurements and Langmuir-Blodgett depositions) and setup needs, a dedicated UV spectrometer, Brewster angle microscope, an ellipsometer, a (90 degree) dynamic light scattering (DLS) particle sizer, a spin coater, sputtering, chromatography pump, as well as standard equipment like balances, centrifuges, water baths, refrigerator, ozone cleaner, ovens, glove boxes, etc. The lab should also be stocked with standard laboratory items such as pipettes, glassware, gloves, etc. The recommendation was made that Susan Krueger at NIST, who has recently completed a list for stocking a new biology lab, Zoey Bowden at ISIS, Jarek Majewski at LANSCE, and Yamali Hernandez, who stocks the regular labs at NIST, could be contacted for a list of what standard equipment and supplies might be needed rather than attempting to enumerate them in this discussion. With increasing complexity of experiments, there is a growing need for a more complete nearby laboratory for preparation of samples that do not transport well or have short shelf lives and offer access to a variety of equipment such as liquid chromatography, freeze dryers, rotovaps, etc. This laboratory could be somewhat further removed (e.g., an adjacent building) or in the case of the SNS be located in the Central Laboratory and Office Building (CLO). The stocking of this laboratory would probably best be done gradually, based on the needs of the in-house staff and in collaboration with the users that have particular needs. It may be appropriate to have two such facilities, one for biology and another for the rest of soft matter as the needs can be quite different. Key to the success of a good support lab with useful support equipment is an appropriate level of technical staffing and a strong personal interest from in-house scientific staff.

III. The needs of the variety of experiments carried out on SANS in particular dictate that a substantial amount of floor space be available around the sample area. Supporting services required include gas supply (helium, N_2, compressed air, etc.), vacuum line, chilled water, three-phase power, a ventilation hood over the sample area (elephant trunk) for ventilation of volatile samples, and noise-reduction curtains. Ways of dealing with maintaining sample environmental conditions (particularly temperature and humidity) during transport to the beam position (off-line sample environment) were considered important as well. The idea was also raised of having a liquid nitrogen line near the SANS instrument, leading from a large supply tank outside the hall. This could reduce the need for filling and lifting large Dewars. Of course, the supply tank must be accessible by a liquid nitrogen delivery truck.

IV. It was deemed very important that the ability to transport large objects (e.g., Dewars) to the sample area be available 24/7. Along those lines, again, it is noted that the only access for the SNS SANS would be by overhead crane and the question was raised as to what accommodations are to be made for such access (e.g., crane operators on duty 24/7, users trained in crane operation).

V. Time precluded lengthy discussions on software support needs. It was pointed out that another workshop a few weeks away was being held to address this very issue. One theme that did surface was the need for real-time visualization coupled with modeling.

3.1 SESSION PARTICIPANTS

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4. BREAKOUT SESSION ON NEEDS FOR HARD MATTER: SUPPORT FACILITIES AND SAMPLE ENVIRONMENTS; ISOTOPIC LABELING

Facilitators: John Larese, University of Tennessee/Oak Ridge National Laboratory, jzl@utk.edu
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The main areas discussed during the hard matter breakout session were (1) assistance for new users by trained technical staff with sample environment/preparation tasks, (2) development of well-equipped sample environment areas in the vicinity of neutron instruments, (3) creation of a deuteration/separated isotope preparation sample facility, and (4) development of schools or training programs to develop technical staff for sample environment assistance. Several recommendations and a non-exhaustive list of sample environment equipment emerged from the discussion outlined in detail below.

Recommendations:

- Create a general deuteration/isotope materials facility for the production of deuterated and novel isotopically substituted materials that extends beyond biological and soft matter.5
- Create reasonably sized, dedicated technical/SE staff who are available 24/7 during neutron operations.
- Build and locate SE preparation areas at SNS near instruments and amply equipped them with appropriate devices like glove boxes, hoods, and SE equipment, as detailed in the SE list.
- Develop SE technician training programs with local technical colleges in partnership with SNS and other institutions.
- Provide an adequate and trained ES&H and technical staff.
- Develop and sustain a centralized, 24/7 SE preparation and technical assistance center.

4.1 DISCUSSION SUMMARY

The audience of the hard matter session was composed of many experienced neutron users with a smaller number of newer users (mostly students and junior faculty). The less experienced neutron users indicated that they clearly need an area for sample preparation and experienced guidance during all aspects of their use of neutrons (including data collection and postexperiment data reduction). There was a consensus among all session attendees that laboratory preparation space be located near the instruments (i.e., as close as possible to the instrumental floor) and that the SE areas should be well-equipped (see attached list of typical experimental sample environment equipment) and accessible 24/7 during run cycles. Furthermore, it was recognized that an ample number of well-trained, SE technical staff was a requirement for the successful operation of SNS or any neutron user facility. Finally, everyone agreed that all of the interfaces to instrument control, sample environments, and data reduction must be user friendly and well documented.

Some time was spent discussing the need to expand the idea of a deuteration lab to include more that just biological/soft matter samples. In fact, the hard matter attendees felt that a more diverse center incorporating general isotope substitution and deuteration was needed for SNS. It was felt that there was a need to hire resident expertise to perform the required chemistry. The staff at this facility would likely be chemists and material scientists who would perform deuteration or partial deuterations of organics, biological samples, and other hard matter samples. It was suggested that this resource was one that could be extended to include a multisite, multicountry effort with pooling of resources and perhaps a central repository of materials/knowledge. This would help crystallography, inorganic, and polymer areas of science.

[5] Additional recommendations on facilities for deuterium labeling are found in the session report that follows this one.

The rest of the discussion focused on the need for qualified personnel to assist in and run the SE and ES&H areas. It was recognized that good technical support staff are the backbone of the neutron facility and are often responsible for a major portion of the success of an experiment. Attendees thought that some type of SE technical training course and interfacility apprenticeships or mentoring might be a useful way to train individuals for these SE jobs. The use of community colleges and technical centers was also suggested. Some users indicated that they would like access to certain technical areas (i.e., users with proper training and certification) like machine shops, glass shops, and chemistry labs for out-of-hours operations. Finally, it was uniformly recognized that the SE people need to be employed in sufficient numbers (average of about two per instrument) and reside in a general pool, including electronics/computing, vacuum/gas handling, low–temperature, and mechanical experts. It was hoped that a true 5.5 full-time equivalent (FTE)/instrument with 3 instrument scientists per instrument would be possible.

4.2 SAMPLE PREPARATION

The following is an itemized listing of the major components of the sample environment requirements:

- Accurate balances (1-kg and 100-g range)
- Inert gas glove box handling systems (with appropriate antechamber access)
- Environmental/chemical hoods with appropriate external venting and air vacuum and gas supply lines
- Complete set of hand, simple machine tool, and electrical devices (meters) and ample work bench space
- Vacuum and conventional furnaces
- Complete high-vacuum systems including high-volume, corrosive atmosphere, and oil-free systems (turbo pump based)
- Computer-controlled and conventional gas-handling manifolds (both in lab and on instrument systems)
- Sample holders, annular, flat plate and cylindrical geometry (aluminum, vanadium, zero-scattering alloy) with gas entry capillary access
- Sample assembly enclosures (Plexiglas box system for assembling samples in a helium atmosphere)
- Sample storage vessels including bell jars and desiccators for retention of air-and moisture-sensitive samples
- Complete set of hand tools and ancillary fasteners and sealants
- Helium leak detector
- Cryostat-compatible sample inserts with gas-handling or remote sample introduction capabilities, also with rapid temperature change/quick quench, remote gas sampling, or gas flow capabilities
- Cryostats-cryo-furnaces with various features covering the temperature range from <300 mK to 1000°C (including but not limited to recirculating helium 3 fridge, simple dilution fridge, rapid change displex, and ILL orange cryostats)
- Gas storage cabinets and regulators for handling a wide range of inert and corrosive gases
- Distilled water source
- Separate sample prep area

4.2.1 Sample Characterization On/Off line (*Adapted for On-Line or In Situ Use)

- GC-mass spectrometer*
- Differential scanning calorimeter*
- IR–Raman spectrometer
- Ellipsometer

- X-ray powder diffractometer
- Vapor pressure adsorption apparatus capable of BET analysis and variable temperature*
- Residual gas analyzer*
- Flow reaction chambers and cells for catalysis experiments*
- Hydrothermal cells* (zeolite to clathrates)
- Cells for examining liquids in porous media
- Medium/moderate pressure vessels (< 20 kbar) (e.g., to study hydrogen storage)

4.3 SAMPLE STORAGE/DISPOSAL

Environment-regulated storage containers for postexperiment analysis and medium-term (six- month) storage.

4.4 SESSION PARTICIPANTS

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5. BREAKOUT SESSION ON DEUTERIUM LABELING

Facilitators: Dean Myles, Center for Structural Molecular Biology, Oak Ridge National Laboratory, mylesda@ornl.gov
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5.1 SUMMARY

This session aimed at identifying the needs and requirements for H/D-isotopic labeling in the soft matter (polymer) and biological-scattering communities. Neutron scattering provides a unique nondestructive tool that is able to probe these complex macromolecules over a wide range of length and time scales and provides key insights into their structure and dynamics. The power of the neutron-scattering technique lies in the ability to exploit—and manipulate—the isotopic H/D content of the target molecule and of its environment in order to selectively highlight and analyze selected parts of complex systems in situ. Together with improvements in instrumentation, isotope labeling not only improves signal/noise ratios and reduces the size limitations that have complicated such applications in the past, but, in many cases, provides new, more sophisticated, more powerful approaches to complex problems in biology and soft matter science.

The need for a concerted and coordinated approach to meet the H/D-labeling requirements for chemistry, biology, and soft matter science was emphasized repeatedly during the meeting. Providing users with the tools and facilities needed to produce deuterated material would enhance both the quality and quantity of neutron experiments that can be done at SNS and other neutron research centers and would, in many cases, make feasible new and more sophisticated experiments than can currently be performed. The requirements for H/D-labeled materials in these fields are so fundamental that this group concluded that SNS should lead efforts to coordinate these activities and should explore the provision of facilities and infrastructure for the design and production of H-D labeled materials. Such efforts were flagged as a priority for SNS.

The major requirements and recommendations for H/D-isotopic substitution are as follows:

- Provide facilities for in vivo and in vitro production of H/D-labeled proteins and other macromolecules.
- Develop methods for the synthesis of labeled polymers, peptides, phospholipids, surfactants, and other complex biomolecules and other small molecules of interest.
- Explore possibilities to coordinate development of labeling techniques and of additional facilities for soft matter science (e.g., the Center for Nanophase Materials Science).
- Develop, maintain, and distribute improved systems and technologies for production and analysis of isotopically labeled molecules through creation of a virtual support network.
- Train research staff and students in these techniques.
- Explore overlapping and complementary needs in the NMR and other communities.

5.2 DEUTERATION FOR THE LIFE SCIENCES

Structural and dynamic studies of biological systems using neutron scattering are greatly enhanced by uniform, random, and selective H/D isotopic-labeling of complex biomolecules such as proteins, nucleic acids, lipids, and sugars. The techniques, facilities, and expertise required are often nontrivial, and the provision of central support facilities and infrastructure for in vivo H/D labeling was considered to be of key and critical importance for the future development of neutron-scattering research in the life sciences. In addition to enabling innovative approaches to be developed for complex problems in biology, H/D-labeling would (1) increase throughput and efficiency, (2) improve signal/noise and reduce size limitations (e.g., by >10 in diffraction), (3) enable analysis of H/D-labeled multiprotein complexes by

SANS, (4) allow analysis of membrane-associated or integral proteins by reflectometry, and (5) enable site specific and segmental H/D-labeling of individual parts of macromolecular complexes for structural and dynamical studies (inelastic neutron scattering (INS)/neutron spin-echo (NSE), etc.). As the focus in biology shifts towards understanding how molecules combine and interact to form complex biological systems, the need and demand for H/D labeling of other important labeled biological molecules, such as nucleic acids, lipids, and sugars, will significantly increase. Efficient methods are therefore required either for the direct chemical synthesis of these materials or for their recovery from the of H/D-labeled cellular biomass.

The expertise and facilities required to produce these labeled materials typically resides in small specialized groups working independently (and in isolation) on individual problems and is often inaccessible to new or casual users. This presents a significant potential barrier for new, occasional, or latent users wishing to use neutron-scattering techniques who lack the time, resources, and/or expertise to produce labeled macromolecules. There is therefore significant need and support for a coordinated approach to the provision of labeled material. The meeting strongly endorsed the creation of a pilot phase *deuteration laboratory* to be developed at Oak Ridge National Laboratory (ORNL) that would provide user access to the expertise and facilities necessary to prepare specifically H/D-labeled biological macromolecules for neutron-scattering applications. Needs and requirements identified would be to accomplish the following:

- Provide scaleup and support facilities for in vivo and in vitro production of perdeuterated proteins/nucleic acids and other labeled biomolecules.
- Optimize labeling strategies for readily expressed proteins and lead process development for the more difficult ones (e.g., labeled membrane proteins).
- Serve as a repository, maintainer, and distributor of the expertise/systems and materials produced by the community.
- Train researchers in application of these techniques.

This would provide expert neutron users access to H/D-labeled materials that are precisely designed and tailored for more demanding and innovative applications. Critically, such a facility would create a "point of access" for the wider (latent) community of structural biologists who are nonexpert neutron scatterers and would introduce and train a new generation of users in application of these powerful techniques.

5.3 DEUTERATED ORGANIC MOLECULES AND POLYMERS

The ability to produce H/D-labeled materials is generally a prerequisite for nonbiological polymer and soft matter applications, especially in the areas of reflectometry and SANS. Problems requiring further research and development relate mainly to matters of cost, process efficiency, and the expertise needed to produce labeled materials. Specific problems are that the necessary perdeuterated raw materials are either unavailable (and difficult to make) or are too expensive for the further synthesis to be feasible, especially if the synthesis is an inefficient one. In many instances, the chemical processes involved may be regarded as too difficult for the individual user to pursue and requires expert synthesis.

This is often simply a matter of scale that could be overcome by coordinated purchasing and by enabling commissioned groups to develop larger-scale syntheses and supply of specific materials.

At the Chem-Bio interface, the added complexity and inefficiency of the syntheses again demands both the lowering of the costs of raw materials, process development, and more specialist synthetic skills, skills that are often centred within small specialist groups that do not have the resources to support the wider community. There is therefore a strong interest in the development of an additional isotope labeling facility to meet the specific requirements of this community. This could be coordinated with development of support facilities at the Center for Neutron Material Sciences (CNMS), which will be collocated on the SNS site in Oak Ridge. One suggested approach is to commission specialist groups to (over)produce specifically labeled H/D materials and precursors that could be locally stored and distributed to the wider community through a networked or virtual laboratory.

Areas identified for further work included development of methods for (1) synthesis of deuterated organic monomers and precursors for complex syntheses, (2) synthesis of deuterated polymers with controlled stereochemistry, (3) synthesis and purification of deuterated surfactants, (4) production of labeled phospholipids, and (5) selective labeling of synthetic polymers.

5.4 CONCLUSION

Significant interest exists for developing coordinated activities in these fields and in having some facility(ies) to specialize in synthesis of labeled peptides/polymers/lipids and other compounds of chemical/biological interest. Provision of labeled materials would have dramatic effect upon many neutron-scattering studies of fundamental and industrial interest. Direct applications in biology include analysis of interactions of H/D–labeled, membrane-bound proteins/peptides and other biomolecules in H/D-labeled model membranes using neutron reflectometry and solid-state NMR, peptide/ligand/lipid-binding interactions in protein complexes studied by neutron crystallography, SANS, inelastic scattering and NMR, and kinetic/dynamic studies of these interactions. In chemistry and polymer physics, the availability of high-purity deuterated precursors, such as olefins and dienes, for example, would enable new labeled polymers to be produced with defined structural architecture like branched and tactic polymers for analysis by SANS and NSE. Provision of D-labeled surfactants would enable neutron scattering to yield selective information on multicomponent oil/water/surfactant systems, providing better understanding of their function in a range of technologically and industrially important processes, such as emulsification and detergency.

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6. BREAKOUT SESSION ON EDUCATION AND OUTREACH

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The four broad themes discussed were (1) training; (2) facilities and neutron schools; (3) development of new instruments, experiments, and ideas; and (4) outreach to the chemistry/biology community. A few common threads emerged, and they are summarized here with the broader context discussed in the following section on educational requirements and opportunities.

- Act as science scouts: the neutron-scattering community would be grown if facilities would have members attend national meetings and give talks on and in general act as advocates for neutron scattering by hooking up interesting science and scientists with neutron tools.
- Encourage instrument scientists and neutron scatterers to present their research in general sessions, not specific neutron sessions.
- Target funding for neutron experiments to encourage people on the edge, bring visibility, and facilitate use of sources.
- Actively follow up to neutron schools and events to leverage already occurring interactions to encourage students.
- Consider ways to facilitate integrating neutron scattering into graduate and undergraduate curricula.
- Simplify data analysis and visualization so that neutrons can be a tool and entry to neutron science and that does not require a commitment to being a neutron scientist.
- Offer focused schools often so that in any year a student could find one on nearly every major topic (magnetism, hard matter, soft matter, structure, and dynamics).
- Facilitate training of graduate students by paying for six months to one year at a facility acting as technical staff to an instrument scientist, while conducting his or her own science.

6.1 EDUCATIONAL REQUIREMENTS AND OPPORTUNITIES

6.1.1 Introduction

The techniques of neutron science, largely developed throughout the physics community, provide remarkable understanding of the nature of matter. The chemical and biological communities, by contrast, have traditionally focused on the manipulation of matter and organization of matter in organisms and new materials. The latter disciplines tend to be “users” of the information gained from a variety of measurement techniques. As users, the chemistry and biology communities would like a sample-in/data-out, near black-box technique, creating a culture vastly different from that which understands the physics behind the measurement and the needed processing of the data to transform acquired data into scientifically usable data. Nevertheless, unprecedented science can be achieved when these disciplines work together, with advances in neutron science emerging from developing methods to understand ever more complex materials and advances in chemistry and biology from neutrons as a unique probe of matter.

Like any cross-cultural endeavor, education of the current and future generations of participants must be the foundation of any interdisciplinary effort. A breakout session at the NSFChemBio Workshop gathered chemists, biologists, and neutron scientists ranging from students to well-seasoned users, to articulate educational requirements and opportunities that will foster a greater use of neutron science in the chemistry and biology communities, thus optimizing our national investment in large user facilities. Increased involvement by chemists and biologists in the neutron sciences will require a diverse set of initiatives that reach out to and educate principle investigators (PIs), graduate students and postdocs, undergraduate students, and students at technical schools. These initiatives call for support of personnel, infrastructure, and curricula and describe possibilities for creative outreach. In this summary, these initiatives are discussed from the perspective of the respective people group to which the initiative is targeted.

6.1.2 PI Initiatives

PIs, as well as classroom professors, are often the controlling interface between students and new research opportunities. In the classroom, professors provide exposure to varied research possibilities (see the subsequent section on undergraduate initiatives). In the research laboratory, it is unlikely that a student will to do an internship or run a complex set of experiments, let alone give up time or people to attend a workshop in an area outside of the PI's realm of expertise, unless the PI is convinced of the scientific value. Therefore, significant resources are needed for education, training, and support.

Science Scouts. More often than not users of neutron science became users only after an expert in the field became aware of their research and suggested that a technique of neutron science could provide valuable insight into their research problem. That "expert" neutron scientist normally will invite the "new user" to apply for beam time, and a collaboration is forged to begin the investigation. Successful collaborations generally result in graduate students and postdocs trained in the specific neutron science technique and a PI willing to invest in student training and neutron science education. To enhance the process of generating more chemist and biologist neutron science users, we recommend that the national user facilities, in conjunction with national funding agencies, add a partial FTE to each instrument at a user facility with responsibilities for scouting the literature and scientific conferences for interesting science. A University National Lab Partnership (UNLP) initiative, modeled after the GOALI-type program of the NSF, could provide funding for travel costs for PI and students, the purchase of isotopes, instrument sample environment modifications, as well as the partial support of the instrument scientist.

Students Are Always New Users. Currently, programs exist at several of the national user facilities to provide full or partial support for new users to conduct experiments. These are excellent programs that should continue to receive support, but participants raised some concern that these opportunities are not well advertised. Nevertheless, to create a culture that regularly thinks of utilizing these national user facilities, it is necessary to recognize that students are always new users. It has been said that university research labs are the only institution where you hire incompetence and fire competence. To address this, it is necessary to create a series of initiatives that support not just new research groups but rather each new user PI, student, or postdoc who is trained to use the facility.

- We recommend that national funding agencies and user facilities work toward the European model, where funds for travel and lodging are made available when a proposal for beam time is approved. (By way of comparison, currently the Intense Pulsed Neutron Source (IPNS) has $15K for user travel support, whereas ISIS has $250K.)
- The residence time in a research group for graduate students (three to five years) and postdocs (one to two years) requires the continual training of new personnel. In addition, most chemistry and biology users will primarily be focused on the chemical/biological questions of their project and will not have the time or background to become experts in neutron science and instrumentation. This requires personnel, associated with the national user facility instruments (related to the aforementioned science scout/UNLP program), to provide student training and continual support of great science by nonexpert users.

Neutron Science Schools. Courses related to the variety of techniques accessible with neutron science are invaluable for training students to become users of neutron science to enhance their graduate research. From the PI's perspective, it is helpful to have a range of course offerings from the more general, such as the Argonne X-Ray and Neutron Scattering School, to more specific courses focusing on one specific topic, such as the 2004 LANSCE Neutron Scattering School on Magnetism. However, it would be useful to have a greater organization and consistency of course offerings. The Neutron Scattering Society of America (NSSA) could, for example, support a "virtual neutron science university," which would coordinate a series of regular course offerings, with a Neutrons 101, followed by advanced topics in small-angle scattering, neutron reflectivity, amorphous scattering, magnetism, inelastic scattering, etc. Because of the continual turnover in personnel, it is necessary that Neutrons 101 be offered annually and advanced topics courses be offered on an annual or semiannual schedule.

6.2 GRADUATE/POSTDOCTORAL INITIATIVES

Graduate students and postdoctoral scientists are generally the hands that will form the actual bridge between the chemistry/biology PI/research group and the national user facility. While education of the PI is necessary to initiate a collaboration, training of graduate students and postdocs is necessary for experiments to actually be conducted. As a result, graduate/postdoc initiatives parallel many of the PI initiatives but require an additional perspective.

Neutron Science Schools. Neutron science schools provide a useful introduction to the possibilities of neutron science. As discussed previously, a regular scheduling of courses, both general and advanced, is important to facilitate student training and progress toward a degree. Several students present in the breakout session had participated on the Argonne X-Ray and Neutron Scattering School. All affirmed its usefulness, but they also expressed the general sentiment that it would be more efficient to have a one-week introductory course that gave a general introduction to neutron science but did not consist of as many different experiments. In their research, most students will only do one or two types of neutron experiments. The experience gained from this course provides cursory exposure to techniques, but it is insufficient training for becoming a true a user of any of the techniques. Also, there is a need for advanced courses, offered on an annual or semiannual basis, that are specifically focused on one technique, where students can gain proficiency with experiments that will augment their research. Such focused courses will fill an important niche to train students whose research will strongly benefit from neutron science but are unlikely to pursue a career as neutron scientists.

Personnel Support. A near vertical learning curve is a common experience of new graduate student and postdoctorial users of beam line instrumentation. This is particularly true for students with backgrounds in chemistry and biology, where the math and physics of neutron science are a bit like a foreign language. By contrast, traditional beam line/instrument support personnel have primary responsibility for instrument design, construction, and maintenance. There is a serious lack of personnel to support people. This experience gap is best conquered when a scientific collaboration is established between a PI and a beam line scientist. The aforementioned science scout/UNLP program could serve to facilitate the hiring of support staff.

User Friendly Software. In addition to personnel support, the vertical learning curve for new users is exacerbated by a maze of software required to collect an analyze data. While the creation of a user service where a sample is sent to the national lab and figures with a one paragraph summary of the pertinent science is returned is unrealistic and arguably not the best practice of science, there is a serious need for greater attention and investment in the software and sample interface side of facilities. Much instrument and data analysis software is not well documented, and certain data analysis parameters appear to be randomly applied fudge factors. Again, there is a cultural barrier between code-writing instrument scientists and chemists and biologists who generally understand the experiment but struggle to get the measured data into a useable form for understanding the science of the experiment. Thus, as equally or more important than funding initiatives for "mid-scale instrumentation projects" is the need to fund the creation of and support for data analysis interfaces to the neutron experiments.

Internships. Graduate internships and postdoctoral fellowships at national user facilities provide a more advanced level of training in the neutron sciences. Investment in such internships is necessary to train the scientists who will become the "expert" neutron scientists who will fill the much-needed roles as the scientific scouts and personnel support to interdisciplinary scientists.

6.3 UNDERGRADUATE INITIATIVES

The undergraduate curriculum catalyzes the process by which students choose to become scientists. Unfortunately, neutron science is virtually without exposure in biology and chemistry curricula. As such, few students are even aware of the opportunities that exist to use “big science” (national user facilities). Initiatives in outreach and curriculum development will provide the supply of students who will become tomorrow’s leaders in the pursuit of interdisciplinary science.

Expert Seminar Series. To supplement the resources available in individual departments, it is recommended that an organization such as the NSSA develop and promote a list of persons who utilize neutron science to address questions in chemistry and/or biology and would be willing to give seminars geared toward undergraduate and beginning undergraduate students. A similar list of consultant experts should be made available for professors to contact and ask questions as they incorporate the following curricular suggestions into their courses and labs.

Curriculum Development. An introduction to the fundamentals of X-ray and neutron scattering can be logically included into a general chemistry curriculum. Much time is spent describing atomic and molecular structure. Diffraction experiments provide a relatively straightforward observable of atom-atom distances. Materials such as the “Optical Transform Kit,” published by the Institute for Chemical Education, can readily be incorporated into course and laboratory curricula. In addition, it should be possible to create web-based interactive laboratory projects whereby advanced undergraduate laboratory students could interface with data repositories at national laboratories, not unlike the National Aeronautics and Space Administration’s (NASA’s) interface between schools and the Hubble Space Telescope or the National Oceanic and Atmospheric Administration’s (NOAA’s) interfaces to national climate data. Neutron scientists are encouraged to write and disseminate such curricular materials in publications such as the *Journal of Chemical Education* in order to connect to a broader science audience.

It is further encouraged that efforts be made to incorporate an “introduction to big science” (i.e., science that can be done at large national lab user facilities) into the American Chemical Society’s (ACS’s) accredited curriculum. Similar efforts may also be possible through organizations such as the Federation of American Societies of Experimental Biology (FASEB).

Research Internships. Undergraduate summer research internships, such as those already in place at several national labs, are recognized as a vital contribution to training future generations of neutron users. These should continue to receive strong support from user facilities and funding agencies. However, a centralized user-friendly interface to all such undergraduate research opportunities would be very useful. A multidisciplinary organization such as NSF would be a logical clearinghouse for such research opportunities, with links to the site in a prominent location from all divisional professional societies such as the ACS, American Physical Society, FASEB, etc.

LENS. The initiative to create the university-based spallation neutron source at Indiana University promises to be an outstanding facility for the training of a generation of neutron users, as well as a center for the development of neutron science–related curricula. Programs such as this should receive strong support at the national funding level.

6.4 TECHNICAL SCHOOL INITIATIVES

It is too easy for Ph.D.s involved in the design of instrumentation and the pursuit of science to forget that a large and qualified technical staff is required to build and maintain large user facilities such as neutron sources. One technical staff representative to the NSFChemBio Workshop raised the important question “Where do we go to hire technical staff who can build and maintain the facility and its instrumentation?” Much of the training of technical staff is on-the-job training. However, it seems there is an untapped opportunity for national labs to partner with vocational/technical high schools and two-year colleges in the vicinity of the national user facility to train personnel for specialized X-ray and neutron technical support.

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APPENDIX A. WORKSHOP STRUCTURE AND COMPLETE AGENDA

The NSFChemBio and SENSE workshops had a significant number of attendees who were new to neutron beams as a characterization tool. This proved to be an effective means for stimulating discussion after talks and during the breakout sessions because several of the speakers in this category presented science talks and then speculated on how they would like to add neutron scattering and spectroscopy to their research programs' toolkit. The give and take with experienced neutron scatterers offering advice was very useful.

For the morning session on day 1, NSChemBio opened with two tutorial-style presentations on the basics, nominally, Neutrons 101a and 101b. Neither assumed any prior experience. The first dealt with the fundamentals of neutron scattering and spectroscopy: what can be measured, what makes neutrons unique as a probe of matter's structure and dynamics. Neutrons 101b introduced instrumentation for both neutron scattering and spectroscopy, with an emphasis on instrumentation at SNS. Additional details on types of instrumentation and the capabilities at other neutron-scattering facilities, including those at NIST, Los Alamos, Argonne, and ORNL's High Flux Isotope Reactor, were included in the workshop's poster session. The poster session also offered an opportunity for the workshop attendees, especially students and postdoctoral associates, to present their research work and interact with the neutron-scattering experts in attendance.

On the morning of day 1, attendees also heard two talks on supporting facilities, including complementary characterization techniques. One talk covered the European D-lab network,[6] which is a distributed laboratory resource for the preparation of (mostly high molecular weight) deuterated materials of interest to biologists. The second presented an overview of facilities for chemistry and biology that will be available to the user community at CNMS, which is presently under construction adjacent to the SNS CLO.

The science talks that followed in parallel sessions on days 1 and 2 provided the meat of the science case in chemistry and at the chemistry/biology interface for using neutrons. On the afternoon of day 2, both the NSFCHEMBIO and SENSE workshops met together for the fifth science session, with a focus on sample environments for neutron experiments. Uses of deuteration and other isotopic substitution, modeling and simulation, a range of sample environments, and complementary characterization techniques were included in these presentations.

The NSFChemBio breakout sessions occurred on the afternoon of day 1 and the morning of day 3.

Day 3 also provided an opportunity to hear from representatives of three funding agencies: the NSF, the Department of Energy, and the National Institutes of Health. Also on day 3, the five breakout sessions on sample environments occurred (see the SENSE Workshop report for details). Many of the speakers at the two workshops gave permission for their talks to be converted to pdf files and posted on the Joint Institute for Neutron Sciences (JINS) JINS web site (www.sns.gov/jins/tallahassee_workshops_2003/CombinedAgendaSep19A.pdf).

[6] Information about the European Deuteration Network: http://www.embl-grenoble.fr/d_lab/ .

NSFChemBio and SENSE Workshops
September 23-26, 2003
Turnbull Conference Center, Florida State University
Tallahassee, Florida

Sponsors

National Science Foundation
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Florida State University
Oak Ridge National Laboratory / Spallation Neutron Source
Oak Ridge National Laboratory / Center for Nanophase Materials Sciences
Oak Ridge Associated Universities

Tuesday, September 23, 2003

7:00 am	Registration opens Vendor Exhibit Set-up
8:15	**Session N-I - Opening Session** Room 122
8:15	**Welcoming Remarks** Lee Magid, JINS Acting Director Jack Crow, NHMFL Director Joan Frye, NSF Chemistry Division, Instrumentation Program
8:30	**Neutrons 101a: What Can Be Measured Using Neutrons,** John Root, NRC Canada
9:45	**Break, Fireside Lounge**
10:00	**Neutrons 101b: Instrumentation for Elastic and Inelastic Scattering Studies**, Kent Crawford, ORNL
11:00	**The European D-Lab Network**, Dean Myles, ORNL
11:30	**CNMS Facilities for Chemistry and Biology**, Mike Simonson, ORNL
12:30	**Lunch, Room 121**

Tuesday, September 23, 2003 afternoon

1:30 pm	**Session N-2: Condensed Phases** **Room 123a** Chair: J. Martin	**Session N-3: Thin Films/Confinement** **Room 122,** Chair: J. Lal
1:30	**Water and Ice**, Alan Soper, ISIS	**Studying surfactant adsorption at interfaces by neutron reflectivity: the current 'state of the art' and future prospects**, Jeff Penfold, ISIS
2:00	**Unraveling Polymer Dynamics**, Michael Monkenbusch, Juelich	**Confined Complex Fluids**, Tonya Kuhl, University of California-Davis
2:30	**New Opportunities In Neutron Scattering: Local Sources and Novel Instrumentation**, David Baxter, Indiana	**Nanoporous Thin Films**, Shenda Baker, Harvey Mudd College
3:00	**Break, Fireside Lounge**	**Break, Fireside Lounge**
3:30	**Novel In-situ Studies**, TBD	**The Dynamics of Confined Quantum Tops**, Dan Neumann, NIST
4:00	**Dynamics of Materials**, Franz Trouw, Los Alamos	**Surface Adsorbed Films**, John Larese, University of Tennessee/Oak Ridge
4:30	**Discussion/Break**	**Discussion/Break**
5:00	**Session N-4, room 123a**	**Session N-5, Room 122**
5:00	**Advanced Isotopic Labeling Center and Facilities** (Dean Myles, Jeff Penfold)	**Educational Requirements and Opportunities** (Shenda Baker, Jim Martin, Joe Zwanziger)
6:30	**Session ends** **Buses Depart for Hotel**	**Session ends** **Buses Depart for Hotel**

Wednesday, September 24, 2003

7:00 am	Registration opens		
8:05	Vendor Exhibit Opens		**Session S-1 - Room 122** **Science Drivers for Neutron Scattering: Impact of Enhanced Sample Environment** **Welcoming Remarks** Lee Magid, JINS Acting Director J. E. Crow, NHMFL Director
8:15			**Purpose and Goals of the SENSE Workshop**, Ian Anderson, Oak Ridge
8:30	**Session N-6, Room 123b** **Biological/Polymer Topics** Chair: J. Martin **3D Structure and Composites**, Ulrich Wiesner, Cornell	**Session N-7, Room 123a** **Catalysis/Vibrational Spectroscopy** Chair: J. Turner **Catalysis Studies Using TOSCA**, John Tomkinson, ISIS	**Nanomagnetism and Neutron Scattering**, Ivan Schuller, University of California – San Diego
9:00			**Quantum Liquids and Solids**, Paul Sokol, Penn State
9:15	**SANS Bio-polymer Studies**, Joanna Krueger, UNC-Charlotte	**The application of inelastic neutron scattering spectroscopy to advance the development of reaction mechanisms in heterogeneous catalysis**, David Lennon, Glasgow	
9:30			**Frontiers in High Pressure Science**, Russ Hemley, Carnegie Institution of Washington
10:00	**Break, Fireside Lounge**	**Break, Fireside Lounge**	**Break, Fireside Lounge**
10:30	**Organic-Inorganic Composites**, Josef Zwanziger, Dalhousie	**Novel Studies Using FANS**, Craig Brown, NIST	**Highly Correlated Electron Systems**, Zach Fisk, Florida State
11:00	**Polymer Patterned Surfaces**, Jan Genzer, NCSU	**Novel Studies Using FDS**, Luc Daemen, Los Alamos	**Materials Science and Engineering Studies Using Neutron Diffraction**, D. W. Brown, Los Alamos
11:30	**Neutron Spectroscopy and Molecular Dynamics Simulation Studies of Protein Dynamics**, Doug Tobias, UC-Irvine	**Nanocomposites for electronic and biomedical applications,** Chris Durning, Columbia	**Three-Dimensional Neutron Microscopy for Structural Dynamics Investigations**, Ben Larson, Oak Ridge

Wednesday, September 24, 2003 Afternoon

	Combined Session of NSFChemBio and SENSE
1:00 pm	**Sessions N-8 and S-2, Room 122** **Beyond Traditional Neutron Science** **Biological and Chemical Science Opportunities at the Center for Nanophase Materials Science**, Mike Simonson, Oak Ridge
1:30	**Dynamic Structure of Membranes: The Concerted Use of Bi-layer Diffraction and Molecular Dynamics Simulations**, Stephen H. White, University of California - Irvine
2:00	**Synchrotron X-ray Studies of Liquid Surfaces**, Peter Pershan, Harvard
2:30	**Extreme Environments for Catalysis**, John Turner, University of Tennessee
3:00	**Break, Fireside Lounge**
3:30	**Environments for Biological Studies**, John Katsaras, Chalk River
4:00	**Polymers and Macromolecules**, Tom Russell, University of Massachusetts
4:30	**Investigation of Liquid Surfaces**, Jarek Majewski, Los Alamos
5:00	**Chemical Reaction Dynamics of Aerosols**, Barbara Wyslouzil, Worcester Polytechnic Institute
5:45	**Depart for National High Magnetic Field Laboratory** **Poster Session and Tour** **Buses Provided from Turnbull to the NHMFL and return to Turnbull and the hotels**
8:30	**Poster session and tour over** **Buses return to Turnbull and Hotels**

Thursday, September 25, 2003

7:00 am	**Registration open**		
8:30	**Session N-9, Room 123a** **Future Opportunities and Needs:** **Support Facilities Needs for Soft Matter** (Paul Butler, Joanna Krueger)	**Session N-10, Room 123b** **Future Opportunities and Needs:** **Support Facilities Needs For Hard Matter** (John Larese, John Turner)	**Session S-3, Room 122** **Present Status of Neutron Sample Environments at High Magnetic Fields and Low Temperatures**, Michael Meissner, HMI, Berlin
9:00			**Research Capabilities at High Pressure**, Chris Tulk, Oak Ridge
9:30			**High Temperature Capabilities**, Trudy Kriven, University of Illinois, Urbana-Champaign
10:00	**Break, Fireside Lounge**	**Break, Fireside Lounge**	**Break, Fireside Lounge**

Combined NSFChemBio and SENSE Sessions

10:30 am	**Sessions N-11 and S-4, Room 122** **Funding Opportunities and New Program Initiatives**
10:30	**New Funding Programs for Mid-Scale Projects and International Cooperation**, Tom Weber, National Science Foundation
11:00	**What's New in DOE's Neutron Scattering Program** Helen Kerch, DOE
11:30	**Funding Opportunities at National Institutes of Health**, Michael Marron, NIH
Noon	**Lunch: Registration closes**
1:00 pm	**Sessions N-12 and S-5, Room 122** **Current Opportunities for Interfaces to Neutron Scattering Research and Education** **National Science Foundation International Materials Institutes (IMI) Program, Advanced Neutron Scattering netWork for Education and Research: with a Focus on Mechanical Behavior of Materials**, P. K. Liaw, University of Tennessee
1:30	**Sessions N-13 and S-6, Room 122** **Current Neutron Scattering and Sample Environment Capabilities**
1:30	**Enabling 21st Century Science**, Zoe Bowden, ISIS
2:00	**Panel Discussion: Thoughts on Current Sample Environment Capabilities and Future Needs at U.S. Facilities** **High Flux Isotope Reactor**, Oak Ridge, Greg Smith **Intense Pulsed Neutron Source**, Argonne, Ray Teller **Los Alamos Neutron Scattering Center**, Los Alamos, Alan Hurd **NIST Center for Neutron Research**, NIST, Jeff Lynn **Spallation Neutron Source**, Oak Ridge, Thom Mason
3:00	**Break, Fireside Lounge**

Thursday, September 25, 2003

Combined NSFChemBio and SENSE Sessions N-14 and S-7

Parallel Sessions: Establishing Sample Environment Priorities

Short presentations will be followed by group discussions; summaries of these discussions will be presented Friday morning in session S-8.

3:30 pm	**Panel I, Room 123a: Sample Environment Priorities in Nano-Magnetism and Nano-sciences.** Chair: Frank Klose, Oak Ridge	**Panel 2, Room 115: Sample Environment Priorities in Biological and Life Sciences.** Chair: David L. Worcester, University of Missouri - Columbia	**Panel 3, Room 123b: Sample Environment Priorities for Quantum Liquids and Solids and Other Highly Correlated Electron Systems.** Chair, Jeff Lynn, NIST	**Panel 4, Room 110: Sample Environment Priorities for Polymers and Macro-molecules**. Chair, Thomas Russell, University of Massachusetts	**Panel 5, Room 122: Sample Environment Priorities for Materials Evaluation and Systematic Studies of Pressure, Temperature, Stress, Etc.** Chair, Thomas Proffen, Los Alamos
	In-situ X-ray Scattering Studies of Epitaxial Crystal Growth, Paul F. Miceli, University of Missouri - Columbia	John Katsaras, Chalk River	Alex Lacerda, NHMFL, Los Alamos	Greg Smith, HFIR, Oak Ridge	**Assembling and Studying Metastable Materials Using Containerless Techniques**, Richard Weber, Containerless Research
	Self-assembly of Epitaxial Magnetic Nanostructures, Donqi Li, Argonne	Stephen H. White, UC-Irvine	Michel Kenzelmann, NIST and Johns Hopkins	Lee Magid, University of Tennessee/JINS	**Conventional Sample Environment Challenges**, Takeshi Egami, University of Tennessee/ORNL
	Studies of Magnetic Nanostructures Using Polarized Neutrons – Current Status and Future In-situ Studies, Hal Lee, ORNL	Jim Torbit, University of Pennsylvania	Meigan Aronson, University of Michigan		Chris Benmore, Argonne
	Opportunities for Magnetic Field Sample Environments for Neutron Scattering, Mark Bird, National High Magnetic Field Lab				

Thursday, September 25, 2003 Afternoon

	Pushing Science Frontiers with state-of-the-Art Sample Environment, Rongying Jin, Oak Ridge				
5:00 pm	Session ends Bus departs for Hotel	Session ends Bus departs for Hotel	Session ends Bus departs for Hotel	Session ends Bus departs for Hotel	Session ends Bus departs for Hotel

Friday, September 26, 2003 Morning

7:00 am	**Turnbull Conference Center Opens**
8:30	**Session S-8, Room 122** **Establishing Sample Environment Priorities: Discussion of Recommendations from Breakout Sessions**, Chair, Jack Crow, Florida State University
8:30	**Panel 1 Report: Sample Environment Priorities in Nano-Magnetism and Nano-Sciences**, Frank Klose, ORNL
9:00	**Panel 2 Report: Sample Environment Priorities in Biological and Life Sciences**, D. Worcester, University of Missouri
9:30	**Panel 3 Report: Sample Environment Priorities for Quantum Liquids and Solids and other Highly Correlated Electron Systems**, Jeff Lynn, NIST
10:00	**Break, Fireside Lounge**
10:30	**Panel 4 Report: Sample Environment Priorities for Polymers and Macro-molecules**, Thomas Russell, University of Massachusetts
11:00	**Panel 5 Report: Sample Environment Priorities for Materials Evaluation and Systematic Studies**, T. Proffen, LANL
11:30	**Wrap-Up and Summary of Panel Recommendations**
12:30	**Adjourn and Box Lunch Provided**

12:45	**Begin with Working Lunch** **Session S-9, Room 122** **Technical Workshop, Follow-up to the April 2001 Workshop at PSI** Chair, Ken Volin, Argonne
1:30	**Sample Encapsulation Considerations in Designing High Temperature Neutron Diffraction Experiments**, Ken Volin, Argonne
	Do we still need helium-flow cryostats? Frederic Thomas, Institut Laue Langevin
	Twin solution dilution refrigerators, Ton Konter, Paul Scherrer Institute, Switzerland

APPENDIX B. NSFCHEMBIO ORGANIZING COMMITTEES

SCIENTIFIC ORGANIZING COMMITTEE:

Cochairs:

- Prof. Shenda Baker, Dept. of Chemistry, Harvey Mudd College
- Prof. John Larese, Dept. of Chemistry, University of Tennessee and Chemical Sciences Division, Oak Ridge National Laboratory

Members:

- Dr. Paul Butler, Neutron Scattering Section, Condensed Matter Sciences Division, Oak Ridge National Laboratory
- Prof. Wayne Goodman, Dept. of Chemistry, Texas A&M University
- Prof. Martha Greenblatt, Dept. of Chemistry, Rutgers University
- Dr. Jyotsana Lal, Intense Pulsed Neutron Source, Argonne National Laboratory
- Prof. Joanna Krueger, Dept. of Chemistry, University of North Carolina at Charlotte
- Prof. Lee Magid, Dept. of Chemistry, University of Tennessee and UT/ORNL Joint Institute for Neutron Sciences
- Prof. James Martin, Dept. of Chemistry, North Carolina State University
- Dr. Dean Myles, Director, Center for Structural Molecular Biology, Oak Ridge National Laboratory; formerly head of the European D-Lab network.
- Prof. Doug Tobias, Dept. of Chemistry, University of California at Irvine
- Prof. John Turner, Dept. of Chemistry, University of Tennessee

Local Arrangements:

- Dr. Al Ekkebus, User Coordinator, Spallation Neutron Source, ORNL
- Ms. Janet Patten, National High Magnetic Field Laboratory, Florida State University
- Ms. Maria Fawver, Joint Institute for Neutron Sciences, University of Tennessee
- Ms. Annetta Hendricks, Joint Institute for Neutron Sciences, SNS/ORNL