

Sample Environment Plans and Progress

October 2005

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SNS Sample Environment Team Leader

Talk Outline



- Sample environment support staff
- Standards & infrastructure
- Equipment inventory

Talk Outline



- Sample environment support staff
- Standards & infrastructure
- Equipment inventory

discussed in light of user needs



SENSE Workshop

Sample Environments for Neutron-Scattering Experiments

Workshop Report

Joint Institute for Neutron Sciences Workshop Series
Florida State University, Tallahassee, Florida
September 24-26, 2003

October 2005

User-Driven Priorities

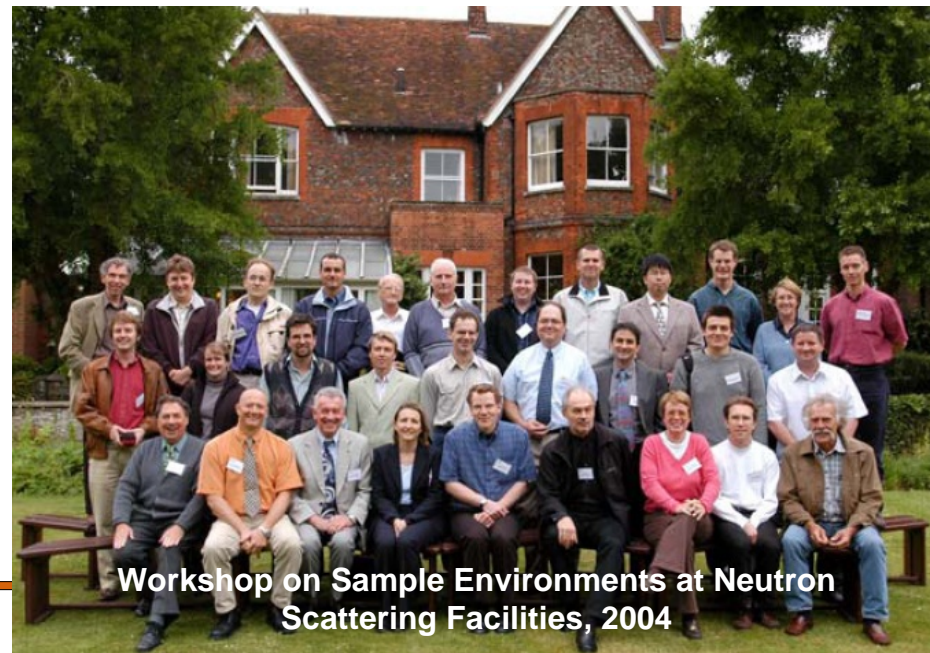


- Ample support staff
- Laboratory infrastructure
- Versatile standard equipment inventory
- Special environments developed with user community input



SENSE Workshop Participants

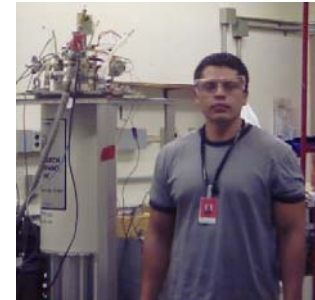
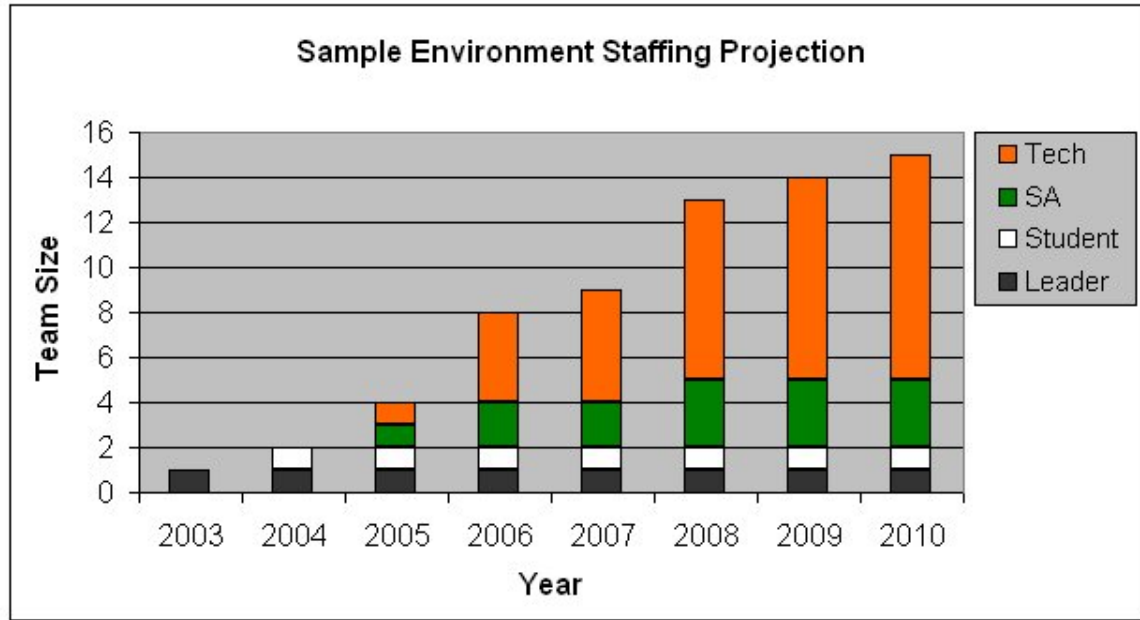
Sep 2003



Workshop on Sample Environments at Neutron Scattering Facilities, 2004

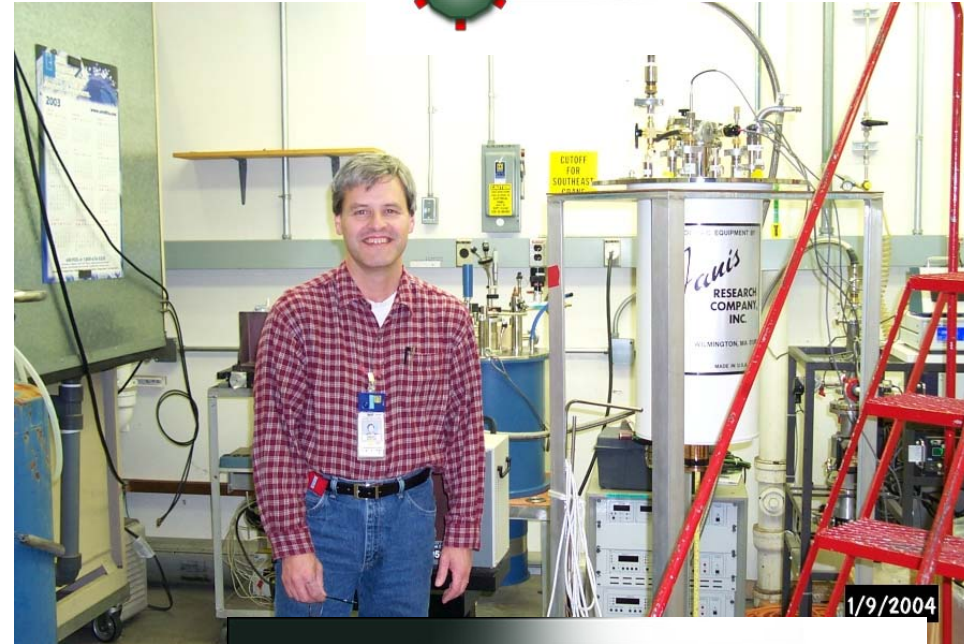
SNS Sample Environment Team

- Forming large dedicated team →
- Gearing up for full user program in 2008
- Working closely with Instrument teams
- Foundational work has been underway for years



Increasing Support at the HFIR

- Existing technician pool includes six with sample environment expertise
 - Chris Redmon, Brent Taylor, Scott Moore, Brian Larkins, Daryl Valentine, Doug Jones
- New scientific associates (SA) and technicians are coming on board
- Lee Robertson new technical group leader
- Instrument scientists play active role in user support



Standards & Infrastructure

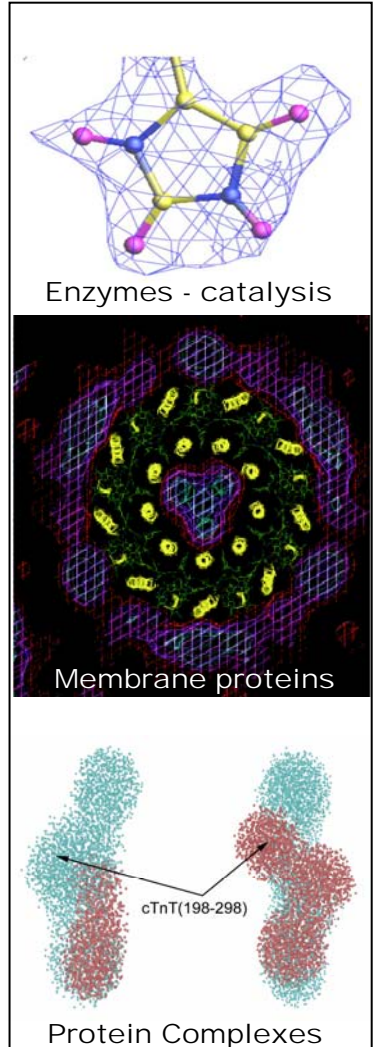
- One of the major concerns raised at



- ORNL plan
 - Sample environment team maintains basic support areas
 - Equipment setup
 - Sample loading
 - Common tools and supplies
 - Beamline-adjacent sample prep areas (space permitting)
 - ORNL centers such as ...

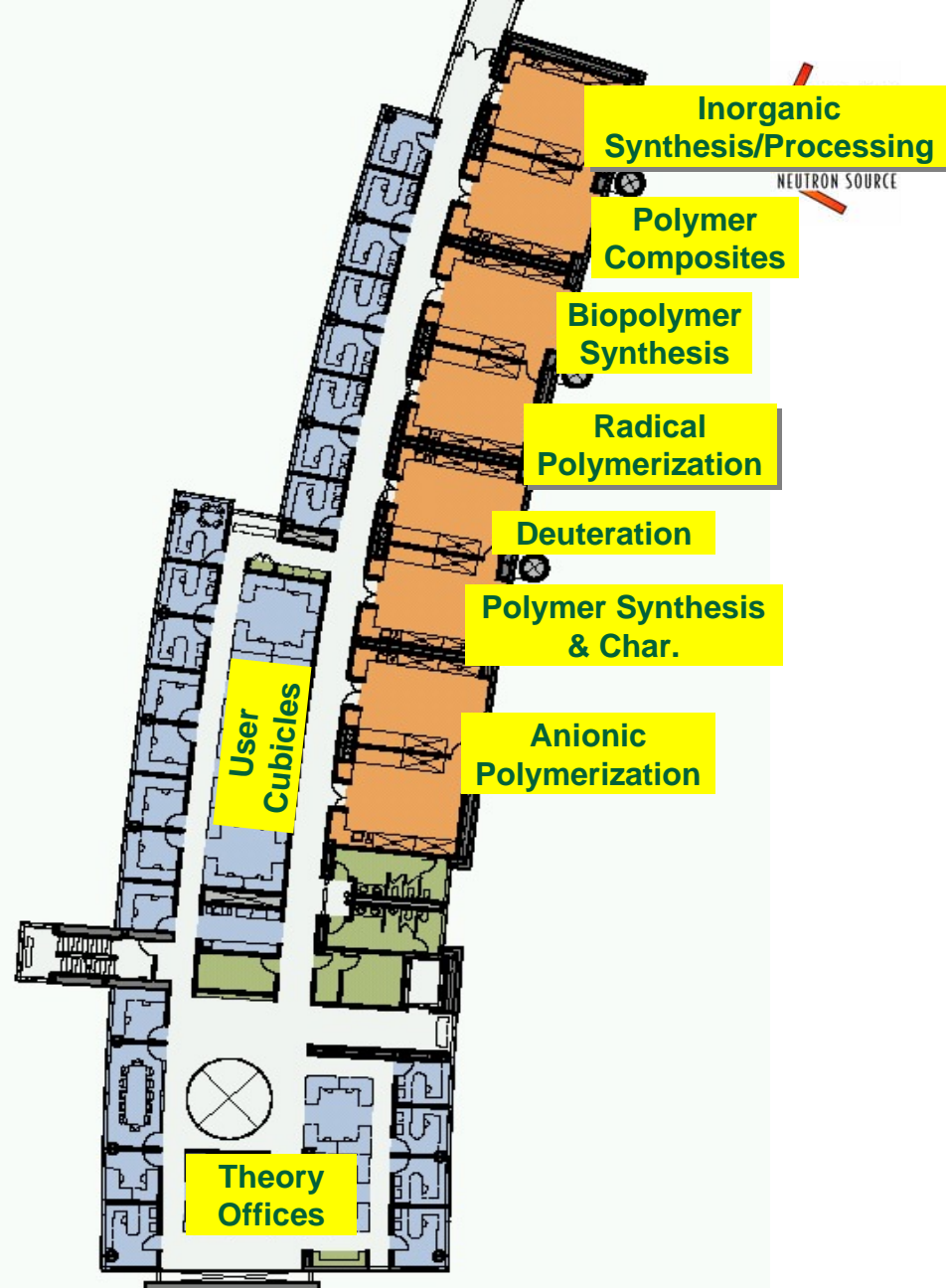
A Central facility and user program for *in vivo* H-D labeling of macromolecules

- **Develop a Central Deuteration Laboratory** dedicated to specific H/D labeling of cells, proteins, nucleic acids and other bio-molecules.
- **Develop better and faster systems and methods** to produce deuterium labeled biological macromolecules for the biology community
- **Improving downstream technologies** to exploit these reagents (including data collection and interpretation for neutron scattering)
- **Train research students and staff** in application of these techniques





- Seven synthetic labs →
- Most labs have three hoods:
 - 8' benchtop
 - 8' walk-in
 - 10' walk-in
- GPC characterization
- Glass annealing oven
- Refrigerators/freezers
- Glove box
- Three characterization labs
- Instrumentation used in many research themes



Standards for Samples and Sample Holders

- Ken Herwig will outline sample policy in later talk
- SE team will stock standard aluminum and vanadium cans



- Special cells will be developed through collaborative work among beamline teams and users
 - Transfer relevant knowledge & technology to SE team

Beamline Utilities and Setup Space

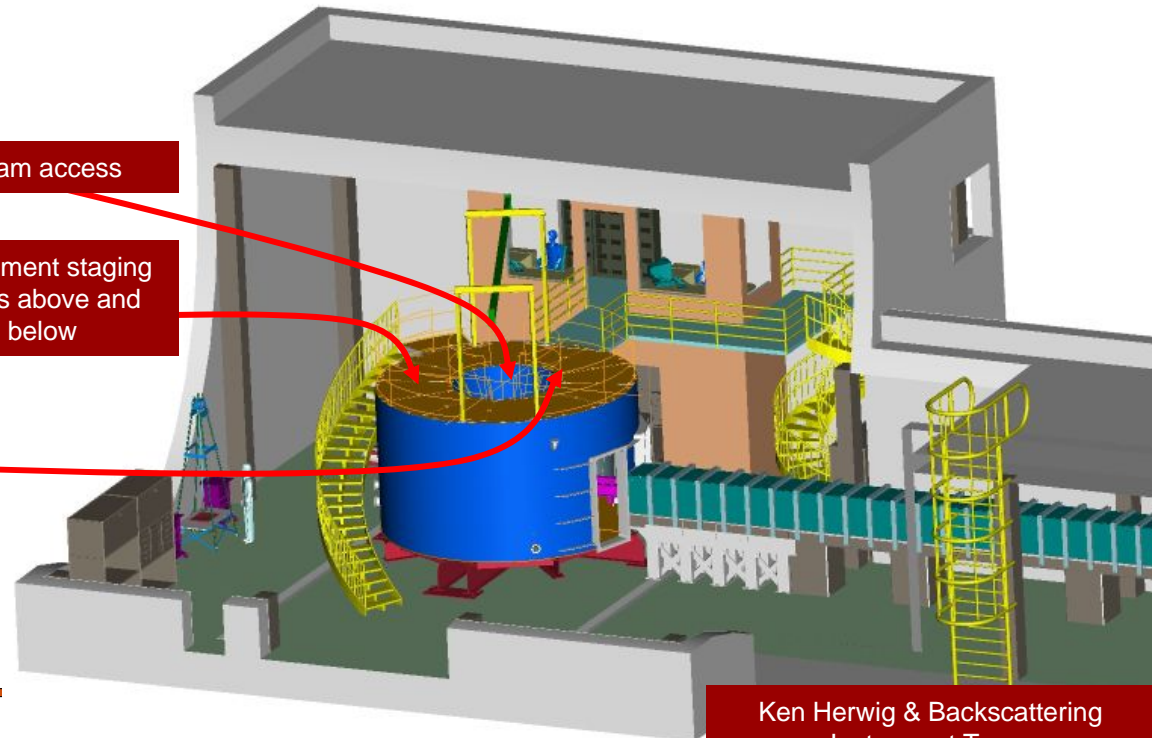
- Numerous electrical outlets
- Cooling water, compressed gases
- Equipment staging areas
 - Shared space planned on instrument floor
 - Instrument specific areas

Utility service panels (electrical, water, compressed gas) mounted around inner and outer perimeter railings

Double-sided electrical service panel shown below

Beam access

Equipment staging areas above and below



Standard Equipment Mounting Scheme

- Off-the-shelf systems are easily equipped with ORNL standard flanges
 - Body flange
 - Tank interface
 - Tail flange
 - Goniometer interface
- SNS and HFIR compatible cryofurnace already commissioned for user experiments



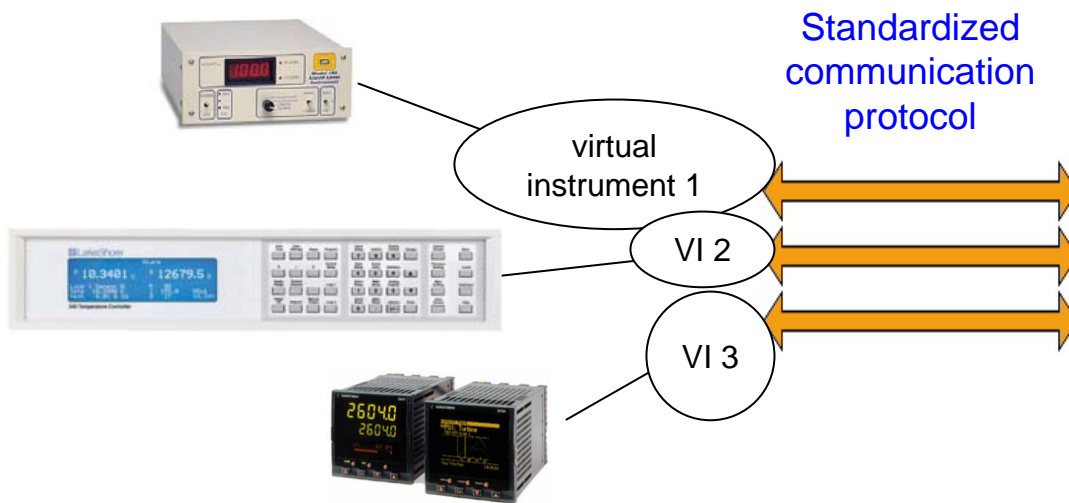
Commissioned for user experiments at the HFIR

Data Acquisition System

Designed with sample environment issues in mind

- Multiple devices & software modules
- Routinely changed
- Accommodate user-supplied hardware & software

- Integrated control package
- Consistent look-and-feel
- User-friendly



Equipment Inventory

- 2 K to 600 K range readily available
 - Maximum versatility, reliability, and ease-of-use sought
- Must combine with pressure, gas environment, etc.
 - Suite of specialty probes and sample cells needed
 - Standard and special equipment are interrelated
- Add beamline-specific options such as remote sample manipulation, automatic sample changing, etc.

Worldwide Trend in Standard Cryogenics ...

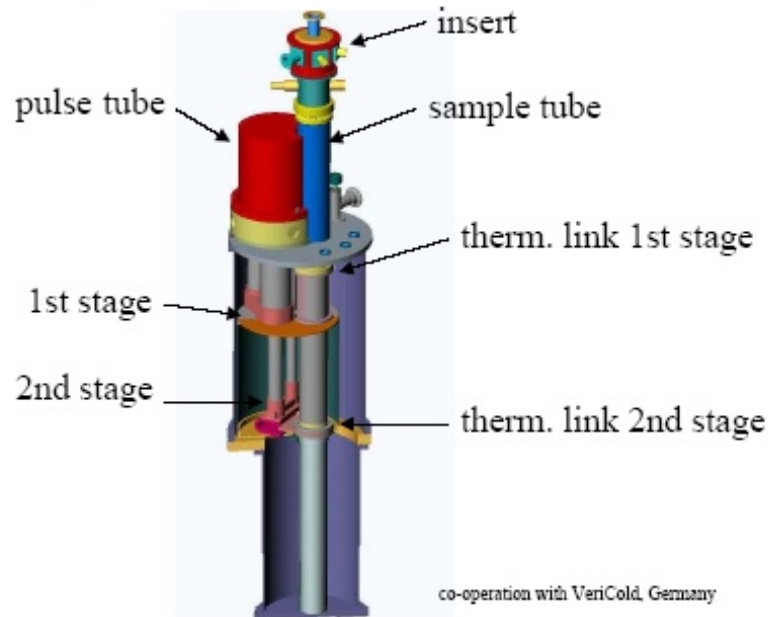
increasingly involves top-loading closed cycle refrigerators



The new neutron source FRM-II



Closed cycle refrigerator CCR2



Workshop on Sample Environment at Neutron Scattering Facilities, June 23-25 2004, Cosener's House, Abingdon, UK



CCR Joule-Thomson 3rd Stage for neutron scattering

work performed with J.-P. Gonzales, O. Losserand & X. Tonon



Top-loading system

Ø49 mm sample

1.5 K < T < 320 K

1l He/mn TPN @ 1.5 K

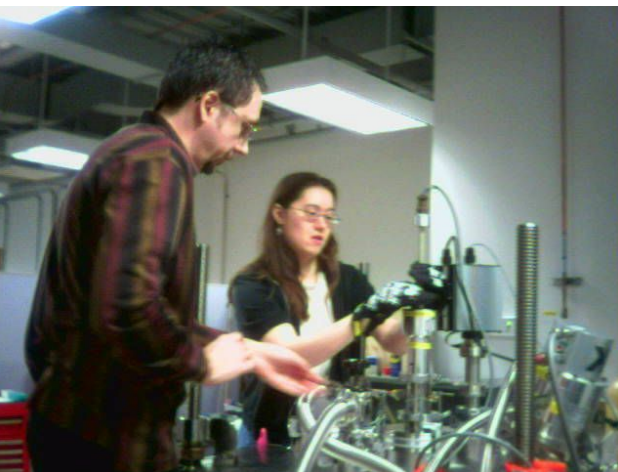
1-week autonomy for 50l@200bar



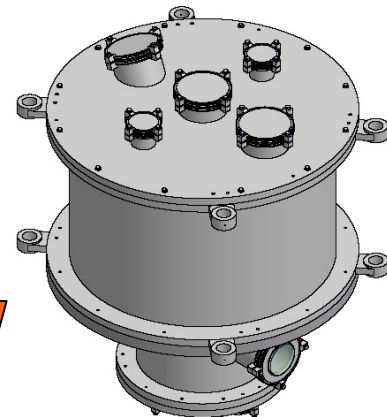
New Cryogenic Environments at ORNL



- HFIR now commissioning “Omniplex” system & high-temperature “Displex”
- Custom R&D platform in use at SNS
 - Developed in collaboration with Omniplex vendor
 - Completely re-configurable
 - Serving as test platform for special components



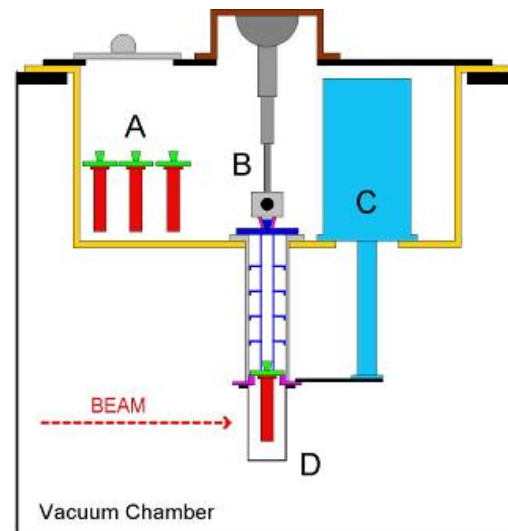
SMASH Rig



SE Team Collaborating with Several Groups to Develop Special Components for CCR Rigs



- Rapid cooling, control and sample changing modules
 - POWGEN3 diffractometer team, ANL/IPNS & small business
- Sample manipulation
 - TOPAZ single crystal team & small business
 - ARCS spectrometer team, LANL/LANSCE & consultant



Sample Changer Schematic

- A: sample storage carousel
- B: pick and place device
- C: CCR cold head
- D: sample tube

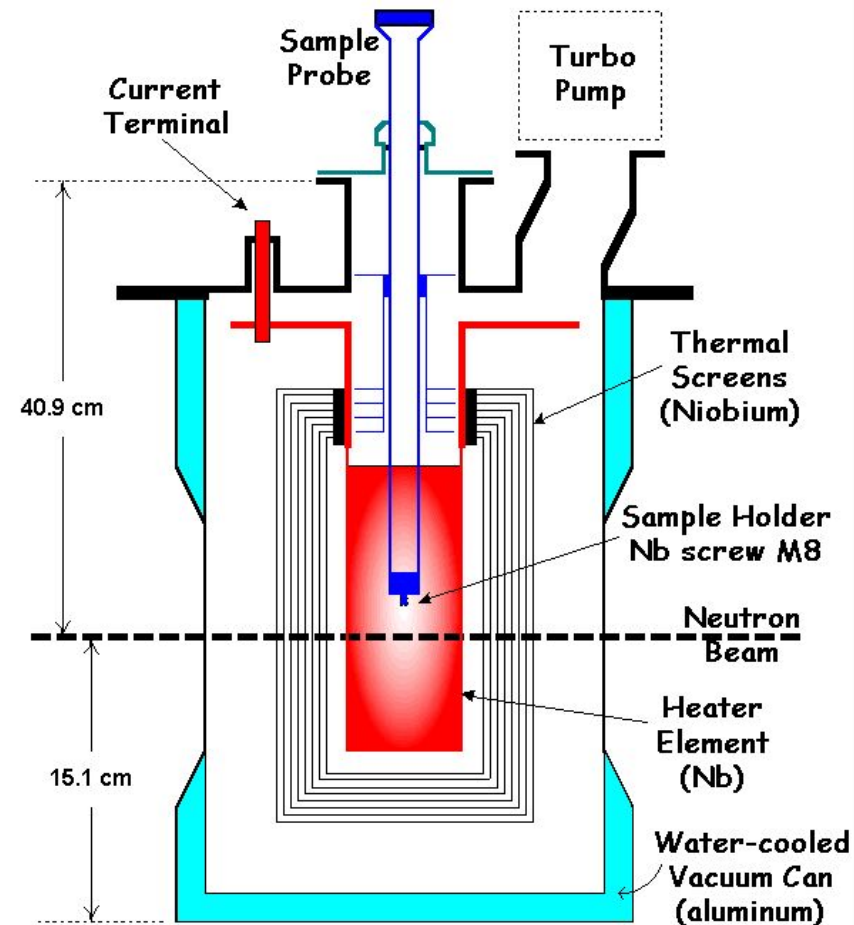
Standard Cryo-Inventory Plan



- Implement a system of rigorous testing, preventative maintenance, and continuous upgrades
- At least one CCR rig per instrument
 - Beamline-specific options such as sample changer
- Additional liquid helium systems (high cooling capacity)
- Extensive suite of specialty probes and cells

High Temperature Environments

- Maximum versatility and modularity sought
- SNS preparing first furnace purchase
 - Top-loading 1800 C system
 - Delivered as vacuum furnace
 - Add controlled atmosphere capability (special probe)
 - Collaboration with POWGEN3 team



Furnace sketch (courtesy of NIST)

High Temperature Environments



- HFIR has 3 furnaces in service
- New controlled atmosphere system under development
 - Collaboration with ORNL High Temperature Materials Laboratory
- Alternate technologies under evaluation
 - Image furnaces
 - Custom systems developed at other neutron facilities

SNS High Pressure Plan



- The *SNAP* team will play a leading role in developing and identifying world-class technology for SNS
 - Instrument-dedicated large anvil
 - Portable clamp and gas pressure cells
 - Training

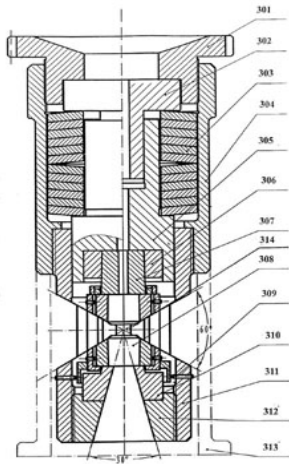
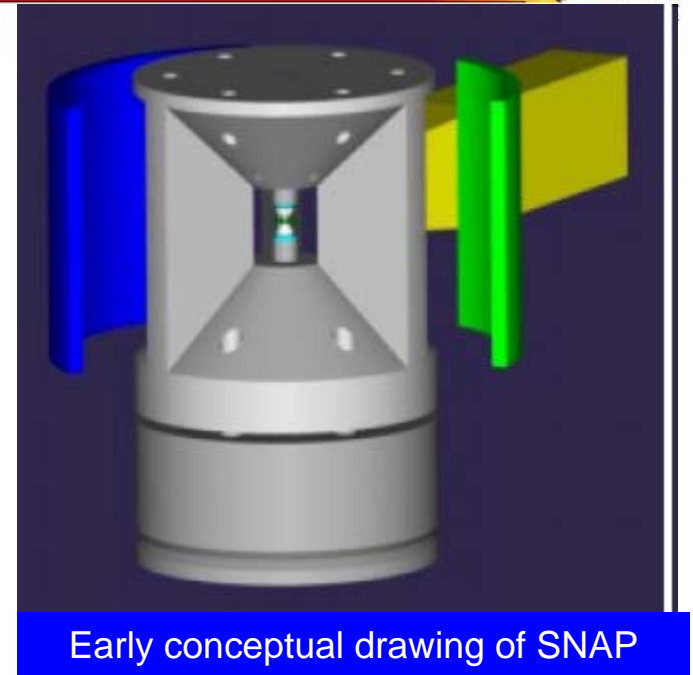


Fig. 3

Suite of small, portable cells under development by SNAP team

Chris Tulk
SNAP



Panoramic anvil cells

Magnet Systems



- High fields are on the way to Oak Ridge
 - 15 Tesla is present world standard for neutrons
 - 16 Tesla system now under development for SNS
 - PSI/SNS collaboration (deliver system in 2007)
 - Dedicated 40T instrument in planning
- Addressing stray field and spin transport challenges
 - SNS first facility in the world to implement policy and technology for stray field compensation and spin-transport optimization
- Medium field (7 – 10 T) also needed
 - Larger bores, combination environments, easier operation
 - SNS-vendor collaboration underway



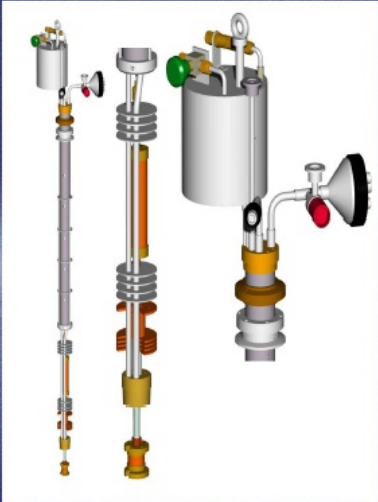
Intensive Cryogenics (below 1.4 K)

- Traditionally involves liquid helium cryostat plus a special insert and gas handling system
 - Hence “intensive”
 - Off-the-shelf equipment
 - *e.g.* →
- New trend
 - CCR compatible inserts

OXFORD INSTRUMENTS Superconductivity Sc

Heliox 3He Inserts

- **Heliox VT** to operate in static VTI
- Base temperature <280mK
- Hold time >24 hours (VT)
- 50 micro Watts cooling power at 350mK
- Simple operation
- No external pumping lines



SNS Intensive Cryogenics Plan



- Purchase the best off-the-shelf technology
- Helium-3 inserts (0.3 K)
 - Compatible with standard CCR-rigs and cryostats
 - One on day-one of SNS operations
 - Three by 2008
- Dilution inserts (0.03 K)
 - One for each magnet system
 - Interchangeable if possible
 - One shared insert for standard cryo-system by 2008

Mechanical Behavior Studies

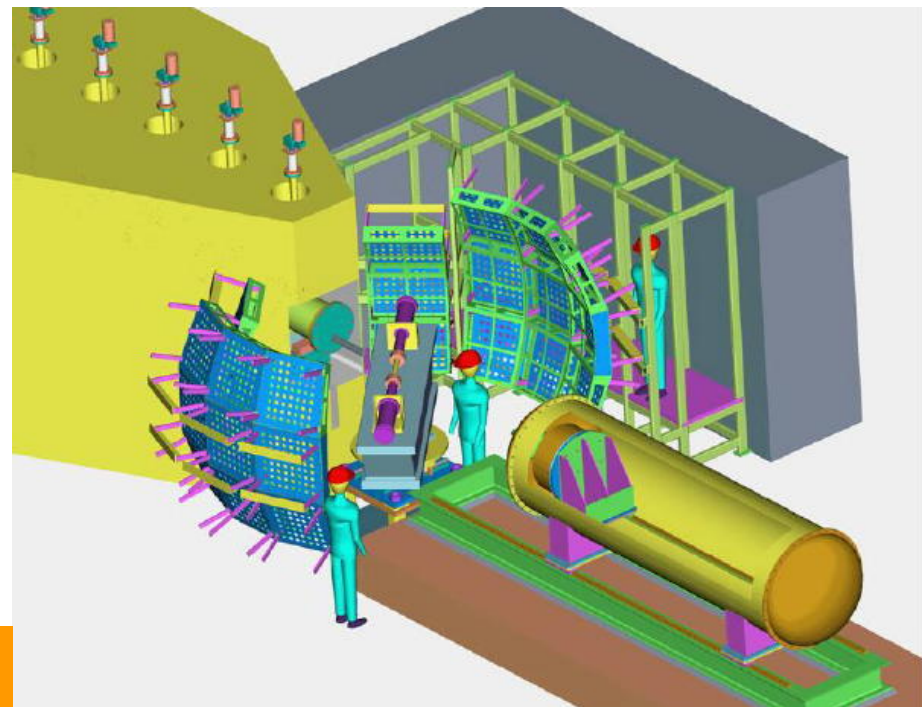
- Vulcan team awarded NSF Major Research Instrumentation (MRI) Proposal

"Development of an In-Situ Neutron-Scattering Facility for Research and Education in the Mechanical Behavior of Materials"

P. K. Liaw, H. Choo, R. A. Buchanan (U. Tennessee)

C. R. Hubbard and Xun-Li Wang (ORNL)

\$2M USD



Environments for Liquids



- Liquids Reflectometer and SANS teams will play leading role in identifying and acquiring dedicated equipment
- Will work with SE team to identify general-use items and provide training
 - Cells and ancillary equipment for liquids
 - Humidity control
- SE team will provide ...
 - Maintenance and custom fabrication support
 - Budget for general-use cells and instrumentation

SNS Inventory Projection



Shared Inventory

year-by-year >>	2005	2006	2007	2008
Beamline instruments	0	3	7	10
Closed Cycle Refrigerator Rig	1	4	8	11
Liquid Helium Cryostat	2	2	3	4
Furnace	0	1	3	4
Mid Range Magnet (5 to 10 T)	0	1	2	3
Gas Atmosphere Control System	0	2	5	10
Gas Pressure Rig, Probe & Cell	0	1	3	6
Chem/Bio Compatible Probes	0	1	3	5
Intensive Cryogenics (3He & 3/4He)	1	2	4	6
High-Field Magnet (> 10T)	0	0	1	2
Standard sample cans	4	500	1500	2500
Vacuum system	1	4	6	8
Leak detector	2	3	4	5

Summary



- Staffing projection
 - Users have been heard
 - Large dedicated sample environment team forming
- Comprehensive equipment suite by 2008
 - You will be the judge
- Sample environment team does not work in isolation
 - Close interaction with Instrument Scientists and SA's
 - Working with vendors
 - Input and collaboration with the user community