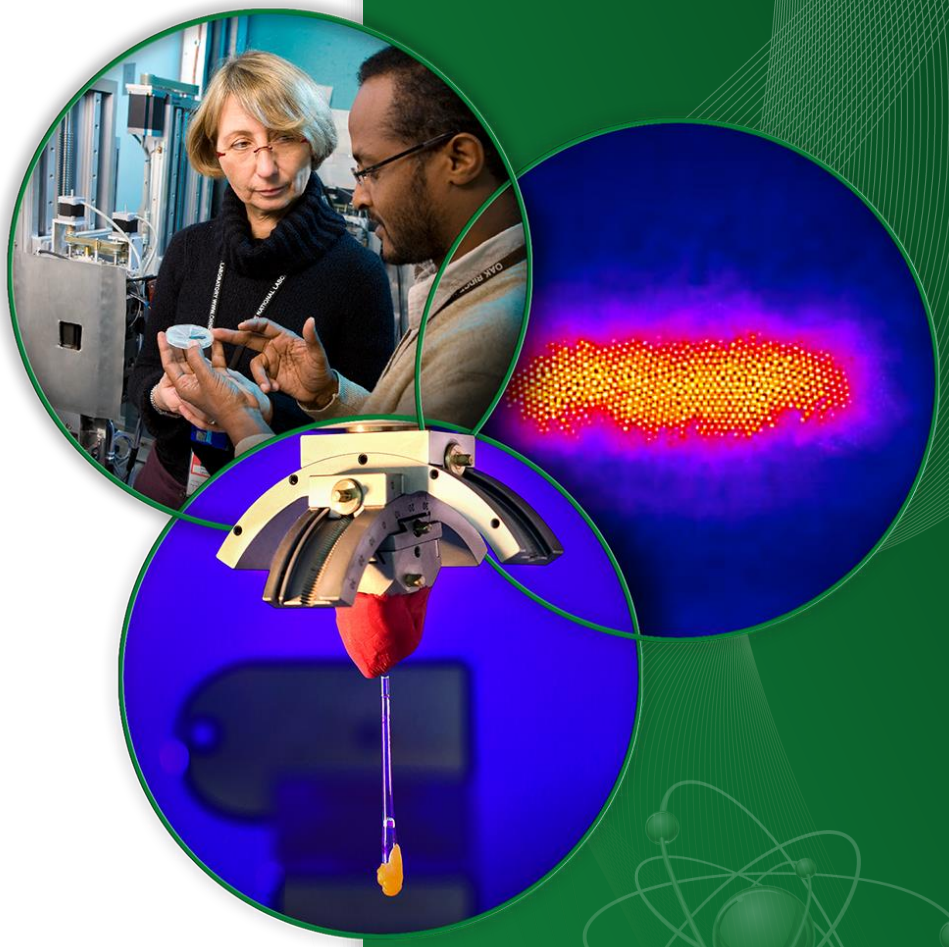
Sample Environment Update



Presented at the

Neutron Day Seminar Series

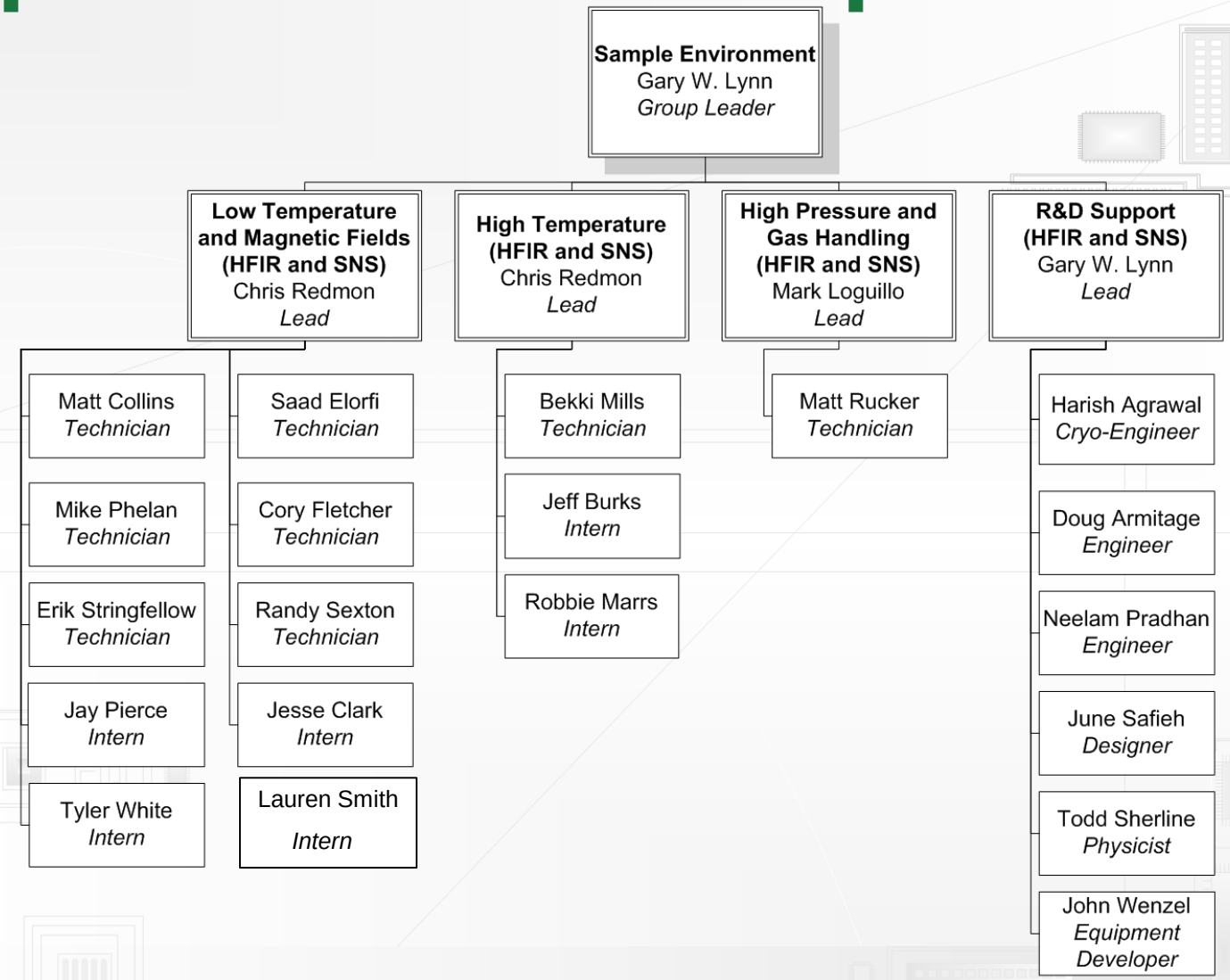
Gary W. Lynn, Group Leader
Sample Environment
Instrument and Source Division

August 22, 2017

Outline

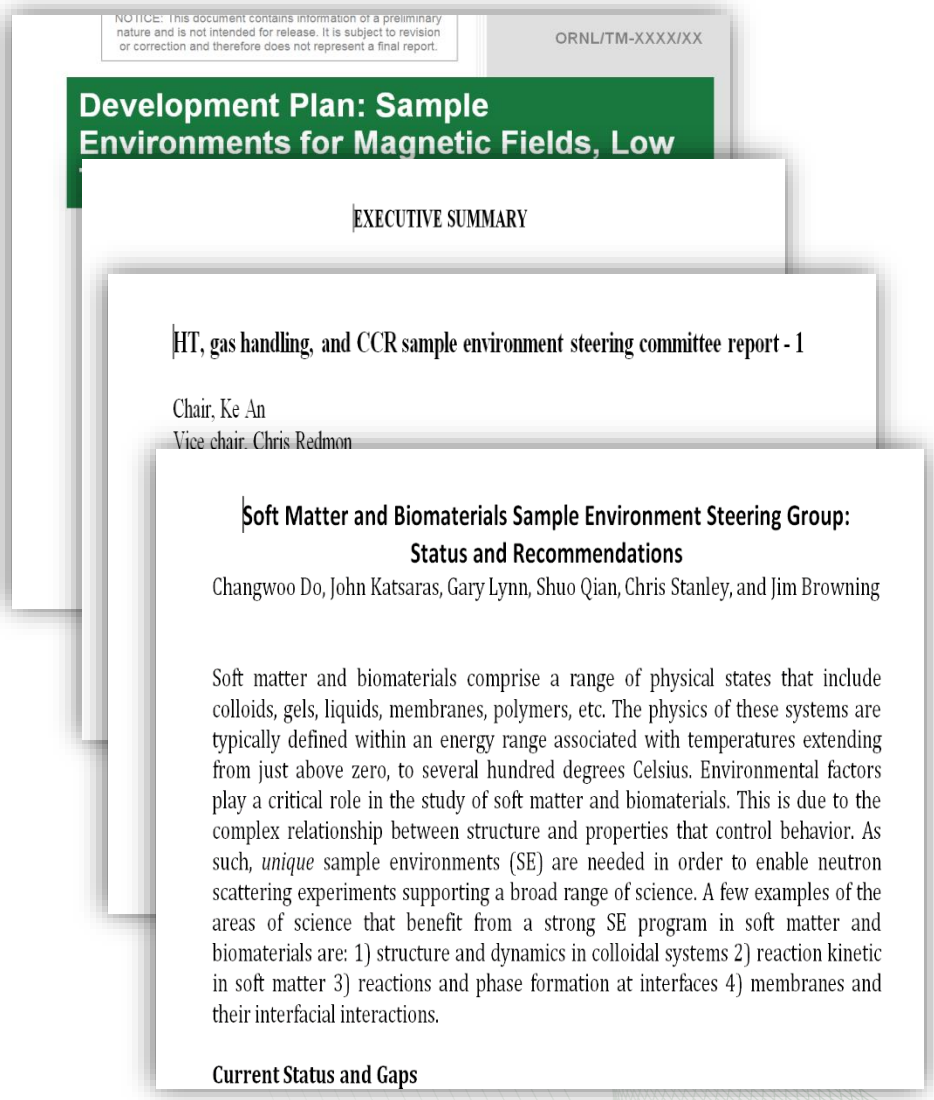
- Sample Environment Group http://neutrons.ornl.gov/sample
  - Organization and expertise
  - Integration with the Science Divisions
    - Sample Environment Steering Committees
    - Sample Environment Strategic and Management Plans
- Highlights of new capabilities
- Contact Information
- *Today's activities*
  - *Sample environment tours (north side of target building) meet in lobby at 10:25 a.m.*
  - *Sample environment forum (C156) 12:30-1:45 p.m. and 2:00-3:15 p.m.*

Sample Environment Group At HFIR and SNS



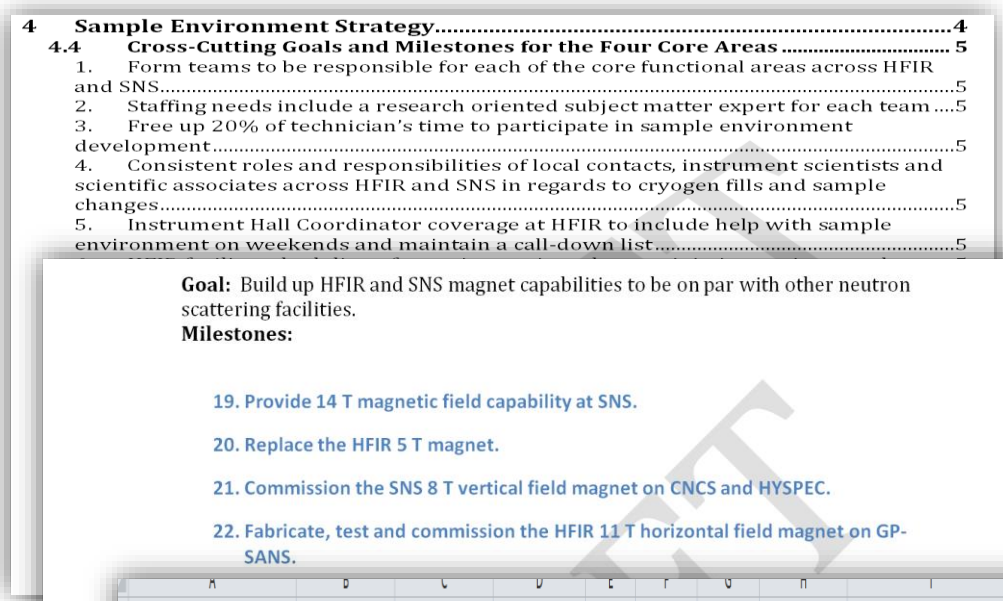
Sample Environment Steering Committees

- Magnetic Fields and Low Temperature (Barry Winn)
- Pressure and Gas Handling (Bianca Haberl)
- High Temperature (Simon Kimber)
- Soft Matter and Biological Materials (Jim Browning)



Sample Environment Strategic Plan

- Roadmap for what we want to accomplish in the next three years
- Aligns with NScD Science Plan
- Action recommendations for the NScD Science Productivity Steering Committee (K. Herwig)
  - 14 T magnet for SNS
  - 5 T magnet replacement for HFIR
- Goals and milestones for improving capabilities, infrastructure and skills
  - Standardize hardware for clamp cells
  - User friendly neutron diamond anvil cell



4 Sample Environment Strategy 4
4.4 Cross-Cutting Goals and Milestones for the Four Core Areas 5
1. Form teams to be responsible for each of the core functional areas across HFIR and SNS 5
2. Staffing needs include a research oriented subject matter expert for each team 5
3. Free up 20% of technician's time to participate in sample environment development 5
4. Consistent roles and responsibilities of local contacts, instrument scientists and scientific associates across HFIR and SNS in regards to cryogen fills and sample changes 5
5. Instrument Hall Coordinator coverage at HFIR to include help with sample environment on weekends and maintain a call-down list 5

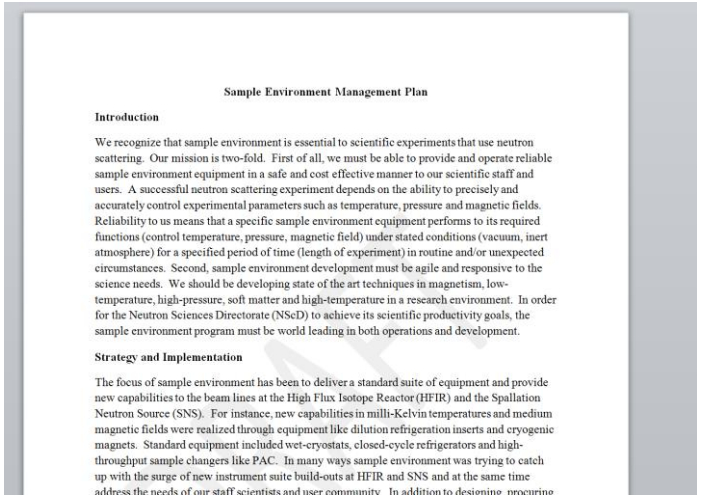
Goal: Build up HFIR and SNS magnet capabilities to be on par with other neutron scattering facilities.
Milestones:

19. Provide 14 T magnetic field capability at SNS.
20. Replace the HFIR 5 T magnet.
21. Commission the SNS 8 T vertical field magnet on CNCS and HYSPEC.
22. Fabricate, test and commission the HFIR 11 T horizontal field magnet on GP-SANS.

Task Name	Primary (Secondary) Org/Area	Facility	Primary (Secondary) Owner	Duration	Start	Finish	Budget (fully burdened)	Status	Origination of task
magnetic fields force gauge	Magnetic Fields, Low Temperature and Wet Cryostats	HFIR and SNS	Todd Sherline	6 months	Nov. 2015	Apr. 2016	$5,993	purchased, parts ready for assembly	SE Group
total of 5 pulsed magnet inserts	Magnetic Fields, Low Temperature and Wet Cryostats	SNS	John Wenzel (Matt Rucker)	5 months	Nov. 2015	Mar. 2016	$37,874	purchased, in fabrication	SE Plan (27.)
cold trap for 8T magnet	Magnetic Fields, Low Temperature and Wet Cryostats	SNS	Chris Redmon	3 months			$5,993	not started	SE Group
commission 8T magnet at CNCS and HYSPEC	Magnetic Fields, Low Temperature and Wet Cryostats	SNS	Todd Sherline	4 months	Sept. 2015	Jan. 2016	NA	completed	SE Plan (21.)
5T magnet (MAG-8) replacement	Magnetic Fields, Low Temperature and Wet Cryostats	HFIR	Harish Agrawal (Daniel Pajerowski)	14 months	Sept. 2016	Nov. 2017	$419,514	concept design finished, ready for budget allocation	SE Plan (20.)
	Magnetic Fields, Low								

Sample Environment Management Plan

- Outlines how we are going to implement our strategic plan
- Sets expectations for sample environment operations, research and development
  - Block scheduling of equipment
  - Use of clamp cells in the user program



Sample Environment Management Plan

Introduction

We recognize that sample environment is essential to scientific experiments that use neutron scattering. Our mission is two-fold. First of all, we must be able to provide and operate reliable sample environment equipment in a safe and cost effective manner to our scientific staff and users. A successful neutron scattering experiment depends on the ability to precisely and accurately control experimental parameters such as temperature, pressure and magnetic fields. Reliability to us means that a specific sample environment equipment performs to its required functions (control temperature, pressure, magnetic field) under stated conditions (vacuum, inert atmosphere) for a specified period of time (length of experiment) in routine and/or unexpected circumstances. Second, sample environment development must be agile and responsive to the science needs. We should be developing state of the art techniques in magnetism, low-temperature, high-pressure, soft matter and high-temperature in a research environment. In order for the Neutron Sciences Directorate (NScD) to achieve its scientific productivity goals, the sample environment program must be world leading in both operations and development.

Strategy and Implementation

The focus of sample environment has been to deliver a standard suite of equipment and provide new capabilities to the beam lines at the High Flux Isotope Reactor (HFIR) and the Spallation Neutron Source (SNS). For instance, new capabilities in milli-Kelvin temperatures and medium magnetic fields were realized through equipment like dilution refrigeration inserts and cryogenic magnets. Standard equipment included wet-cryostats, closed-cycle refrigerators and high-throughput sample changers like PAC. In many ways sample environment was trying to catch up with the surge of new instrument suite build-outs at HFIR and SNS and at the same time address the needs of our staff scientists and user community. In addition to designing, procuring

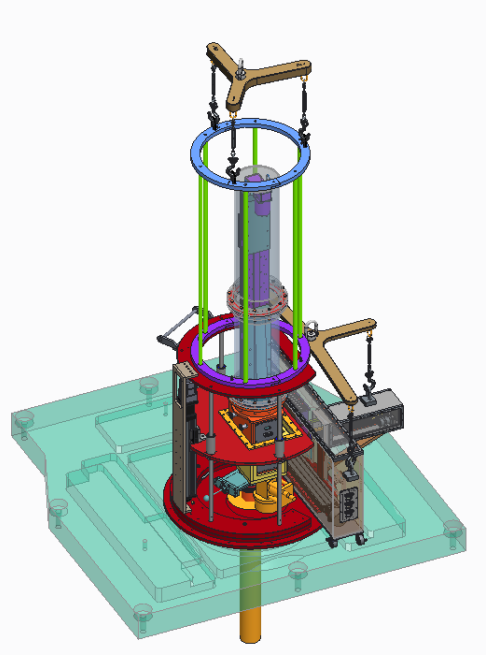
- Large-scale developments (>$100k)
  - Science Productivity Process; budget from the directorate ($10 million)
- Medium-scale developments (between $5k and $100k)
  - Vetted through the SE Steering Committees; budget from the Science Roundtable ($1.4 million)
- Small-scale developments (<$5k and <5 days of technician or engineer labor)
  - Worked through SE Group; budget from instrument budgets

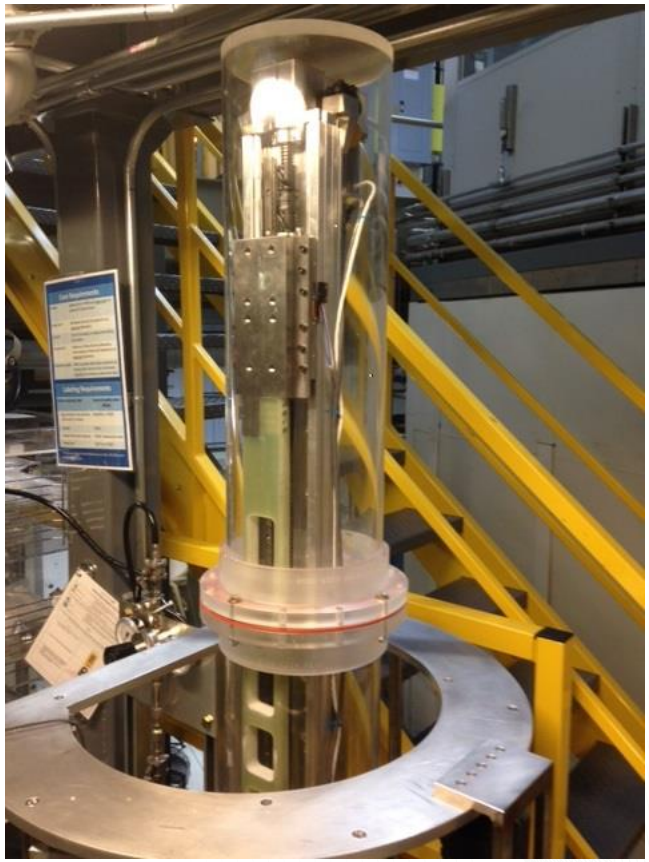
Establishes an Assessment Program and Communication Plan

- User surveys
- Sample Environment Review 2018?
- Website, User Newsletter

High-Throughput Sample Changer for VISION

- Complete initial cool-down in 4 hours
- Sample temperature of 15 K
- Sample changes every 25 minutes
- 50 sample load capacity
- Up to 10 samples pre-cooled
- Contact: Neelam Pradhan

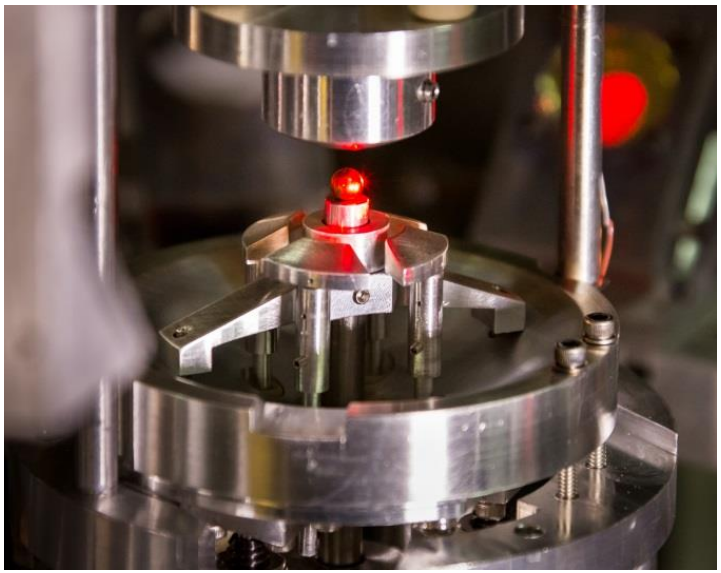


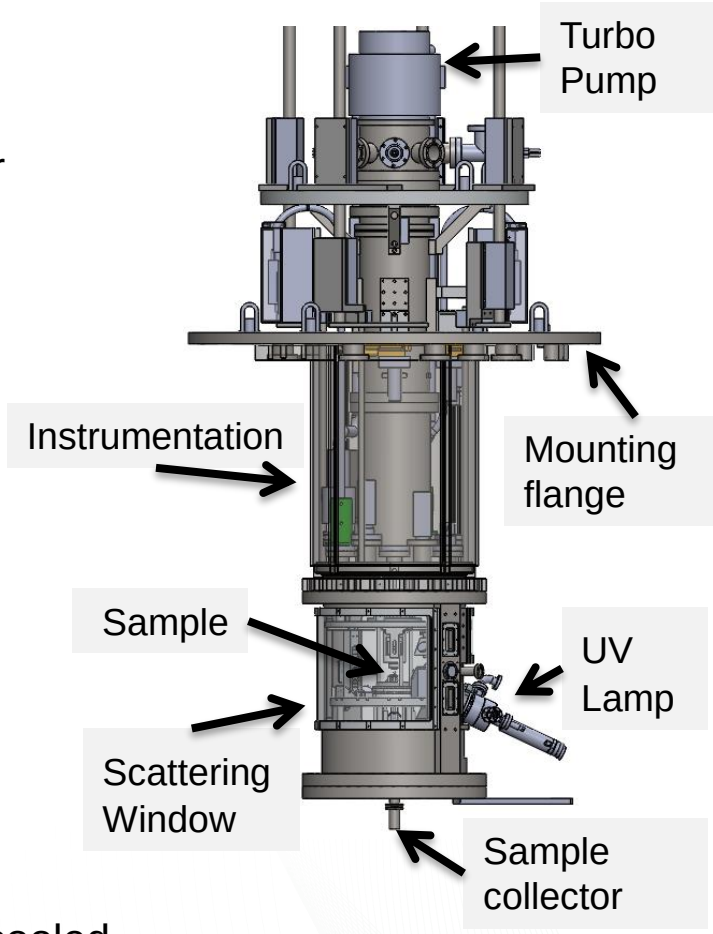


New High Temperature Capabilities in the User Program

Neutron Electrostatic Levitator

- Container-less sample environment: no obstruction or reactivity from container
- High temperature (500 – 2000 °C), high vacuum (10^{-7} torr), high voltage (30 kV, 14 mm)
- Sample carousel holds up to 29 samples: spherical shape preferred, diameter 3-5 mm, mass 100-400 mg
- Successful commissioning and experiments on NOMAD and ARCS





- Access to super-cooled liquid regime
- No container to induce nucleation
- Contact: Dante Quirinale

New Magnet Capabilities in the User Program

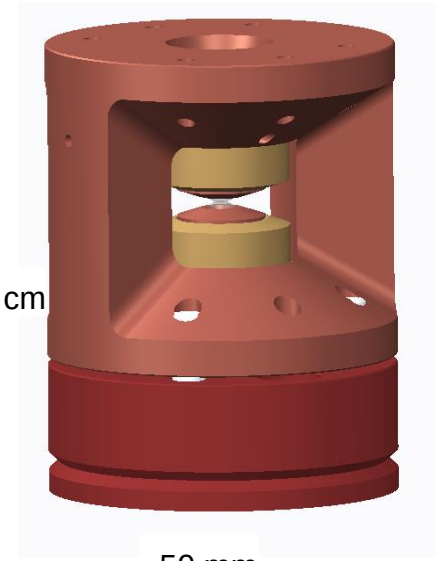
5 T Horizontal Field Magnet for GP-SANS at HFIR and Magnetism Reflectometer at SNS

- Cryogen-free with a warm bore (no VTI)
- Used in neutron polarization experiments on the GP-SANS at HFIR
- Contact: Todd Sherline

11 T Horizontal Field Magnet for GP-SANS at HFIR

- Silicon Windows for SANS
- 50 mm bore diameter
- Compatible with our current 3He and dilution refrigeration inserts
- Optimized for vortex lattice investigations: field inhomogeneity < 1%
- Achieved a base temperature of 1.4 K at 11 T in the lab and on the beam line
- With dilution insert and test sample: 65 milli-K at 11 T on the beam line
- Contact: Erik Stringfellow

New Developments in Pressure



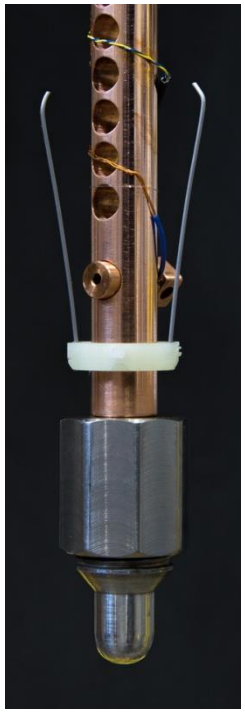
40 GPa User Friendly nDAC

- Increase reliability for use in the user program
- Sample volume of 0.4 mm^3 (1st gen cell 0.06 mm^3), 1.3 mm diameter and 0.3 mm gasket thickness
- Uses polycrystalline diamond
- New Design for CORELLI
- Contact: Reinhard Boehler

10 GPa at 4 K for VISION

- New design Neutron Diamond Anvil Cell
- Uses polycrystalline diamond
- 2 mm culet size
- Sample volume of 3 mm^3
- Tested on VISION and NOMAD with
- Contact: Reinhard Boehler

New Developments in Pressure



2 GPa at 70 milli-K and 7 T

- NiCrAl clamp cells
- Compatible with our dilution refrigeration insert and 8 T magnet at SNS
- Contact: Andrey Podlesnyak

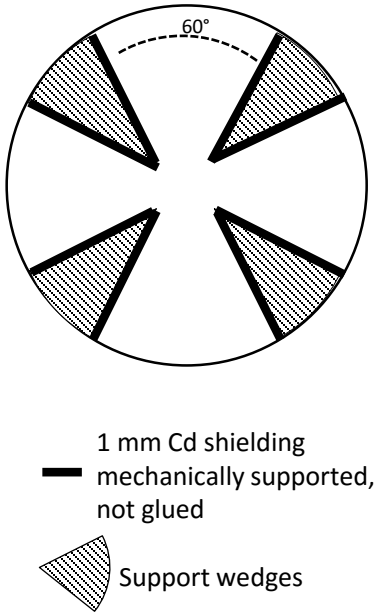
3 kbar Gas Loading System for nDAC

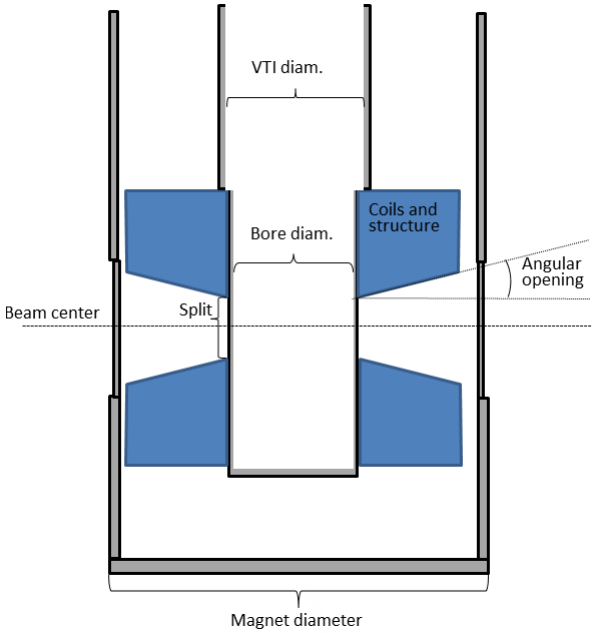
- Loading gaseous samples
- Allows for hydrostatic sample conditions
- Hydrogen compatible
- Successfully run pressurized hydrogen experiments on VISION
- Contact: Mark Loguillo

Current Procurements and Future Considerations

14 T Vertical Field Magnet for SNS

- 14 T symmetric and 12 T asymmetric
- Designed for CNCS, HYSPEC, CORELLI, ARCS and SEQUOIA
- ± 10° out of plane covers all of HYSPEC, ~ 60% of CNCS detectors and middle banks of ARCS and SEQUIOA detectors
- Total path-length of Aluminum < 6.5 mm
  - 16 T FatSam magnet path-length of Aluminum: 12.25 mm in direct neutron beam and 79.25 mm in scattered neutron beam
- Contract awarded
- Expected delivery in Fall of 2018
- Contact: Matt Stone





- VTI diameter – 50 mm above heat exchanger
- Bore diameter – 40 mm
- Split – 40 mm
- Sample volume – 20 mm diameter X 20 mm high
- Angular opening +/- 10 deg.
- Magnet diameter at beam – 695 mm

Contact Information

http://neutrons.ornl.gov/sample HFIR and SNS sample environment information:

http://sampleenvironment.org The International Society for Sample Environment: a platform to promote scientific and technical developments of sample environment at scattering facilities

Low Temperature and Magnetic Fields:	**Barry Winn (new developments)**
	Harish Agrawal, Chris Redmon and Todd Sherline (technical expertise)
Pressure and Gas Handling:	**Bianca Haberl (new developments)**
	Mark Loguillo (technical expertise)
High Temperature:	**Simon Kimber (new developments)**
	Bekki Mills (technical expertise)
Soft Matter and Biological Materials:	**Jim Browning (new developments)**