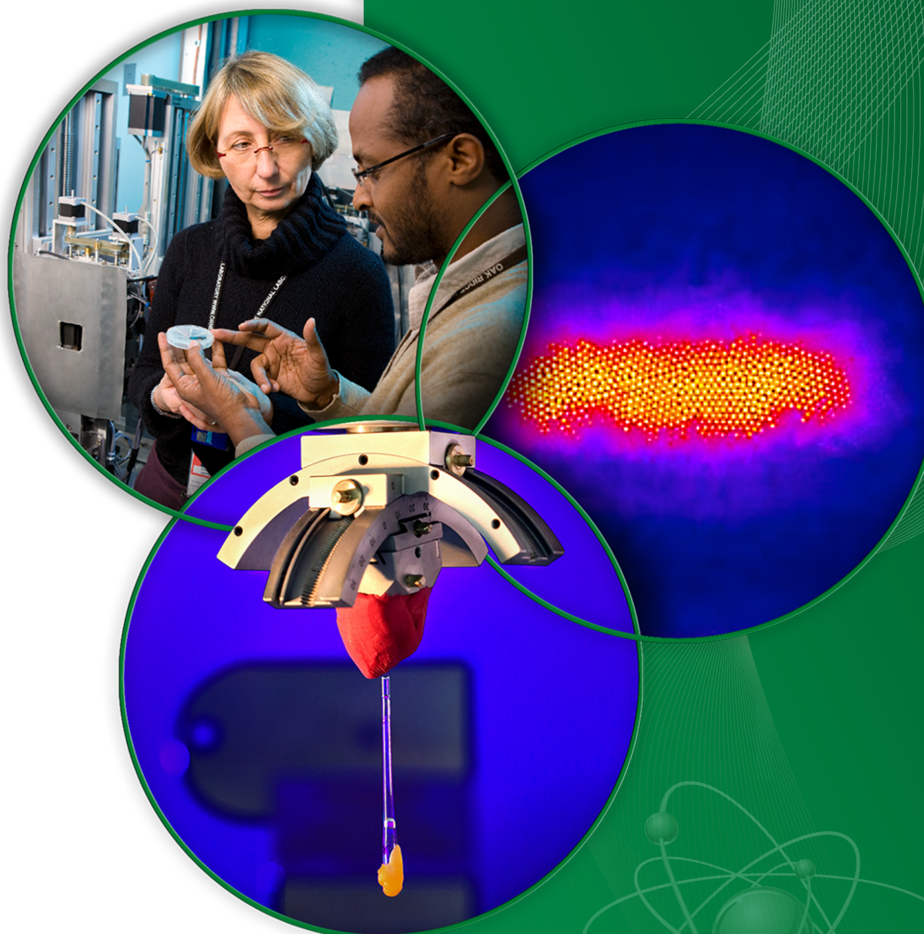
Sample Environment



Presented at the

XIV International School of Neutron Science and Instrumentation

Gary W. Lynn
Group Leader for Sample Environment

April 1-9, 2016

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Sample Environment

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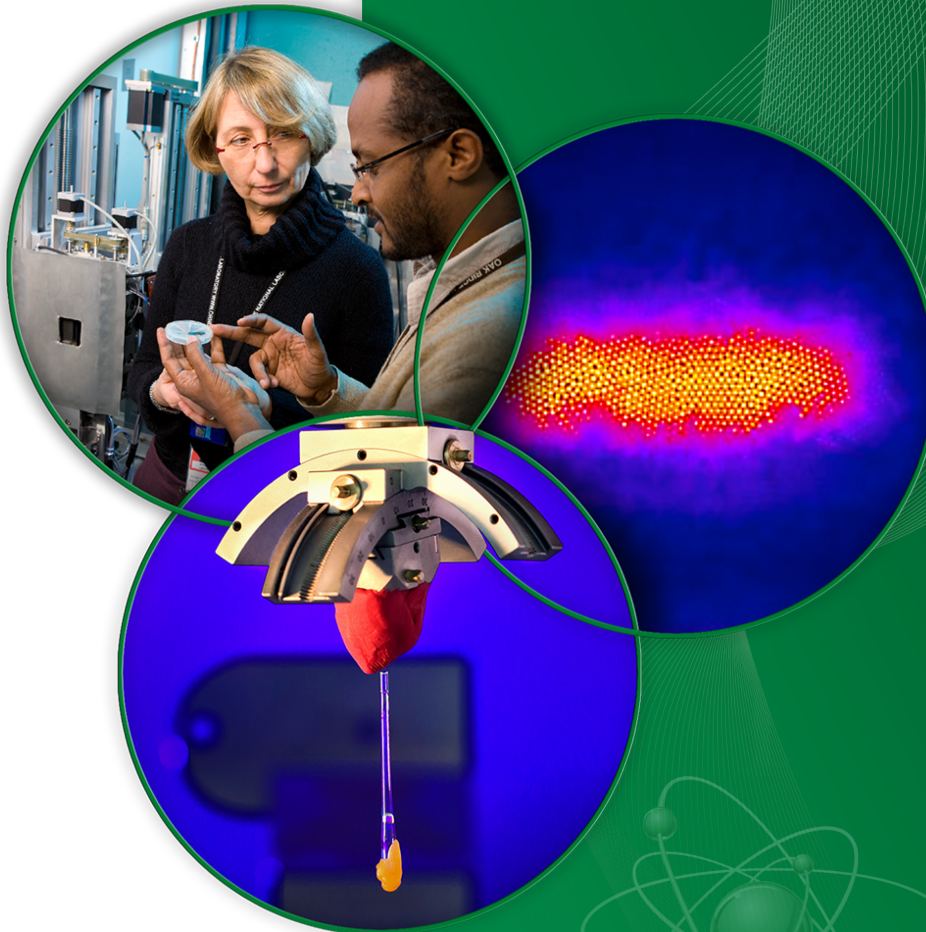

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Group Leader for Sample Environment

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with a few additions by Ken Andersen, including some slides from Eddy Lelièvre-Berna

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Ken's additions

EUROPEAN SPALLATION SOURCE

- In-Beam Materials
  - Al for transparency
  - V or TiZr to avoid Bragg peaks
  - single crystal or glassy quartz/sapphire to avoid grain structure
  - absorbers: BN, B_4C, Cd (except in furnaces!)
- Think about operating the instruments
  - decide on Top or Side Access
  - moving & lifting equipment into place
  - standardised mounts or flanges
  - utilities
  - minimise waiting time
  - minimise change-over time
- Flexibility
  - accommodate unknown future needs

Sample Environment Design Considerations

- Avoid background issues: Minimize amount of material in the incident and detected neutron beam paths
- There are always trade-offs and compromises: Yield strength of material allows for high pressures but has high neutron absorption cross section
- It is important to define requirements up front
- Project management: budget, scope and time

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Sample Environment Design Considerations

- Build in as much flexibility as possible in the sample area: Experiments are continuously more complex
- Don't skimp on the utilities: Electrical power, crane coverage, compressed air, chilled water, Helium recovery
- Remember your neighbors: Stray magnetic fields, neutron background

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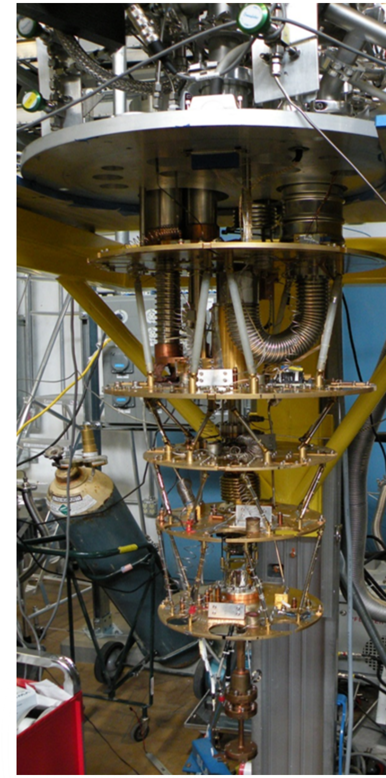
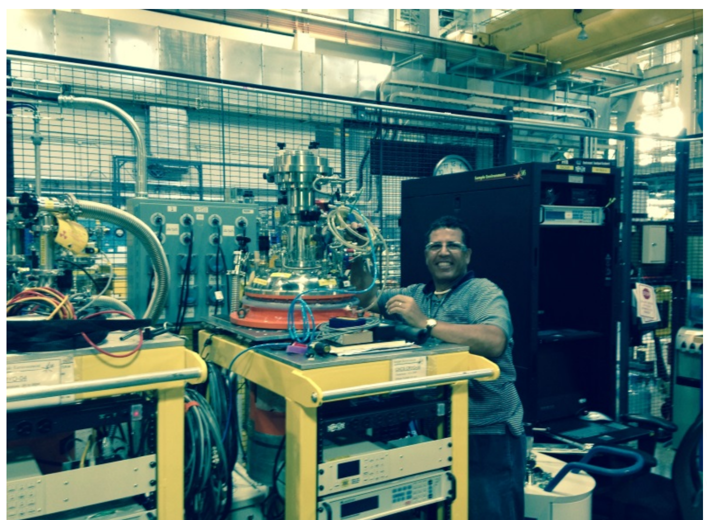
Low Temperature

Low Temperature Equipment:

- Closed cycle refrigerators 4 K - 300 K
- Liquid Helium Cryostats 1.5 K - 300 K
- 3He inserts 0.3 K - 300 K
- Dilution refrigeration inserts 0.03 K - 300 K





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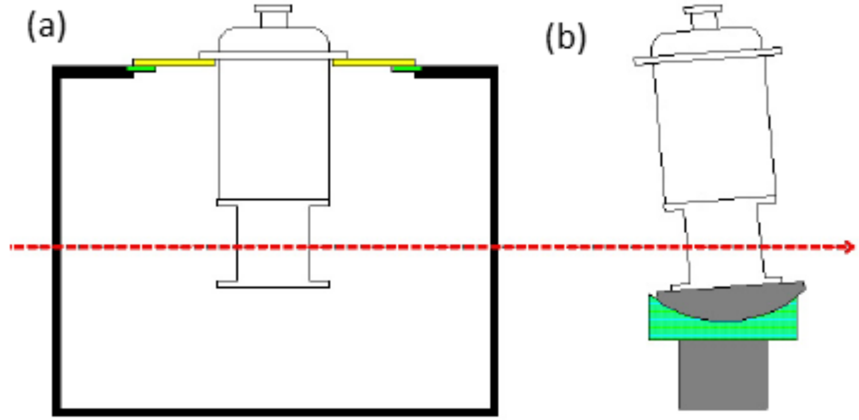
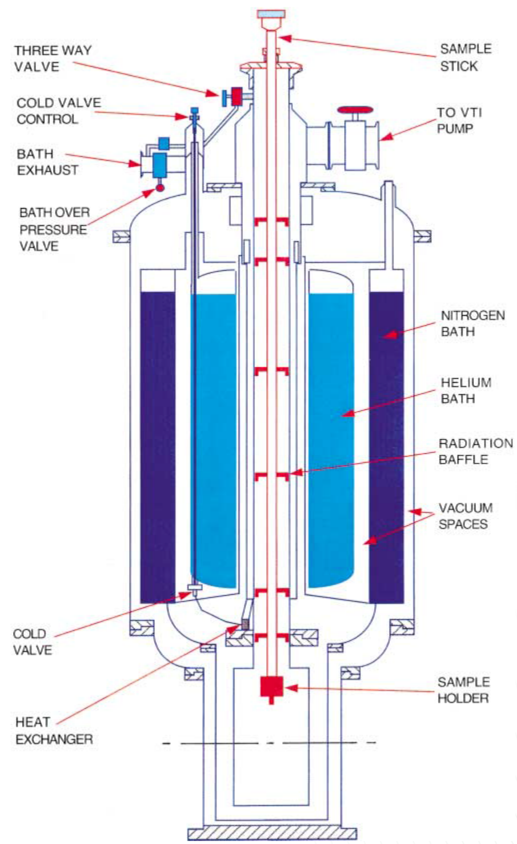
Low Temperature



Liquid Helium Cryostats

- Temperature range 0.3 K - 300 K
- Flange mount or tail mount
- Flange diameter defines maximum diameter allowed (700 mm typical)
- Define flange: bolt holes, vacuum boundary, etc.
- Tail mount: distance from beam center to bottom of tail (50 mm typical)
- Outer Vacuum Chamber (tails) diameter 350 mm
- Distance from stick flange to beam center 950 mm
- 50 mm bore to accommodate 3He and dilution refrigeration inserts
- Sample space diameter 43 mm



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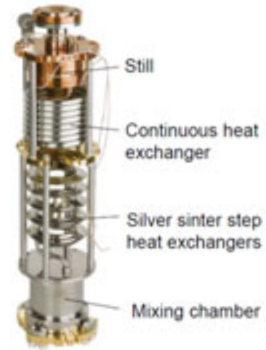
Low Temperature

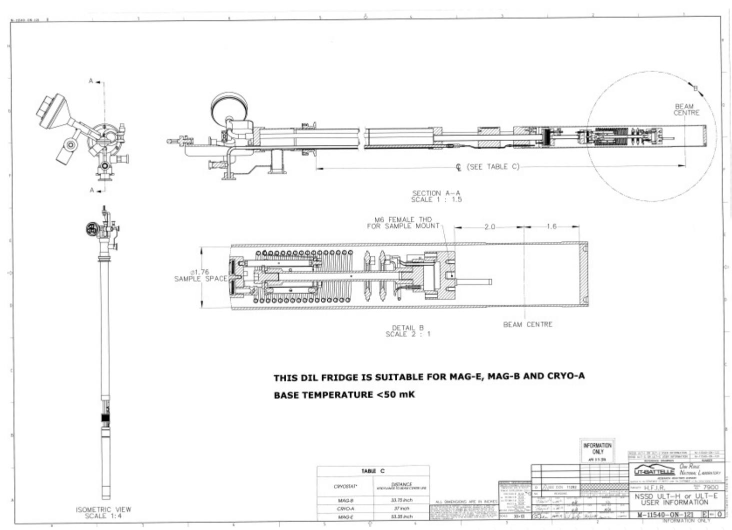
3He Insert

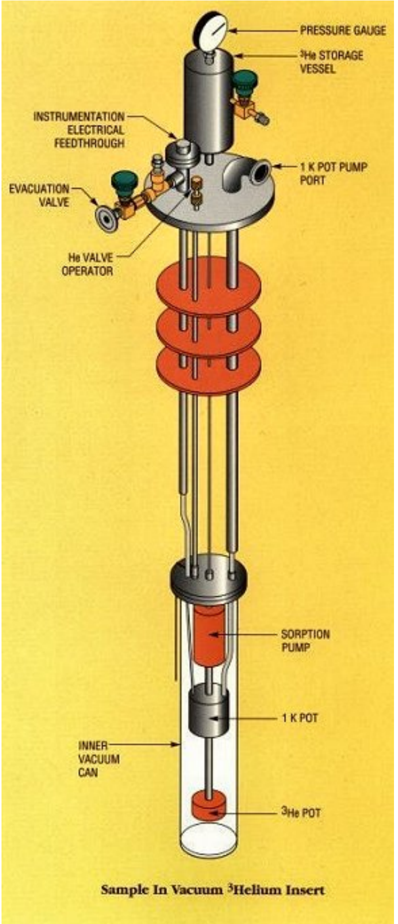
- Temperature range 0.3 K - 80 K (up to 300 K with VTI)
- Achieve a base temperature less than 0.3 K for more than 40 hours
- Maintain a base temperature less than 0.35 K for more than 6 hours with a 50 µW heat load
- Temperature stability of ± 0.003 K below 1.2 K

Dilution Refrigeration Insert

- Temperature range 0.03 K - 1.5 K (up to 300 K with VTI)
- Cooling power at least 40 µW at 0.1 K



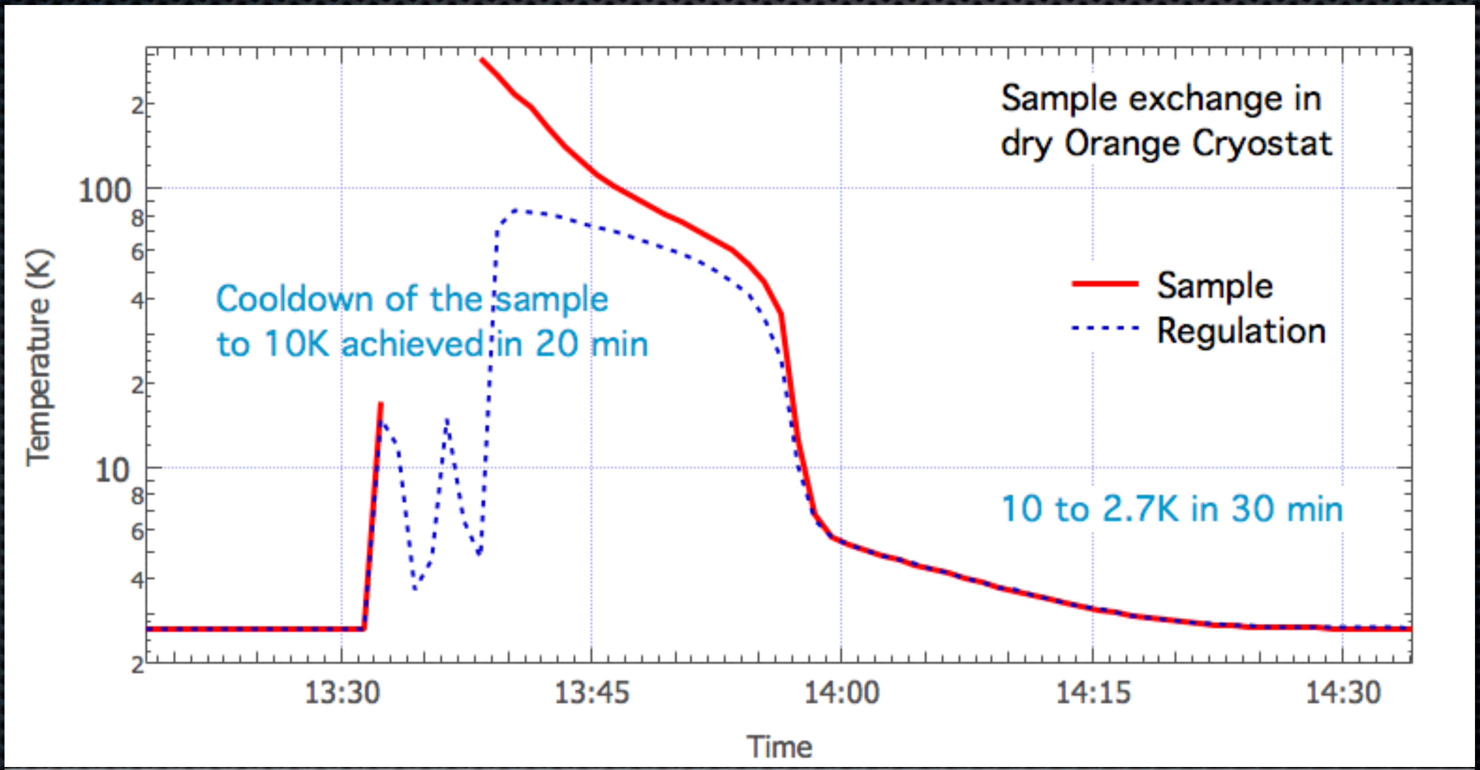




Sample In Vacuum ^{3}Helium Insert

Cryogenics

dry cryostats are easy to use and look fast…



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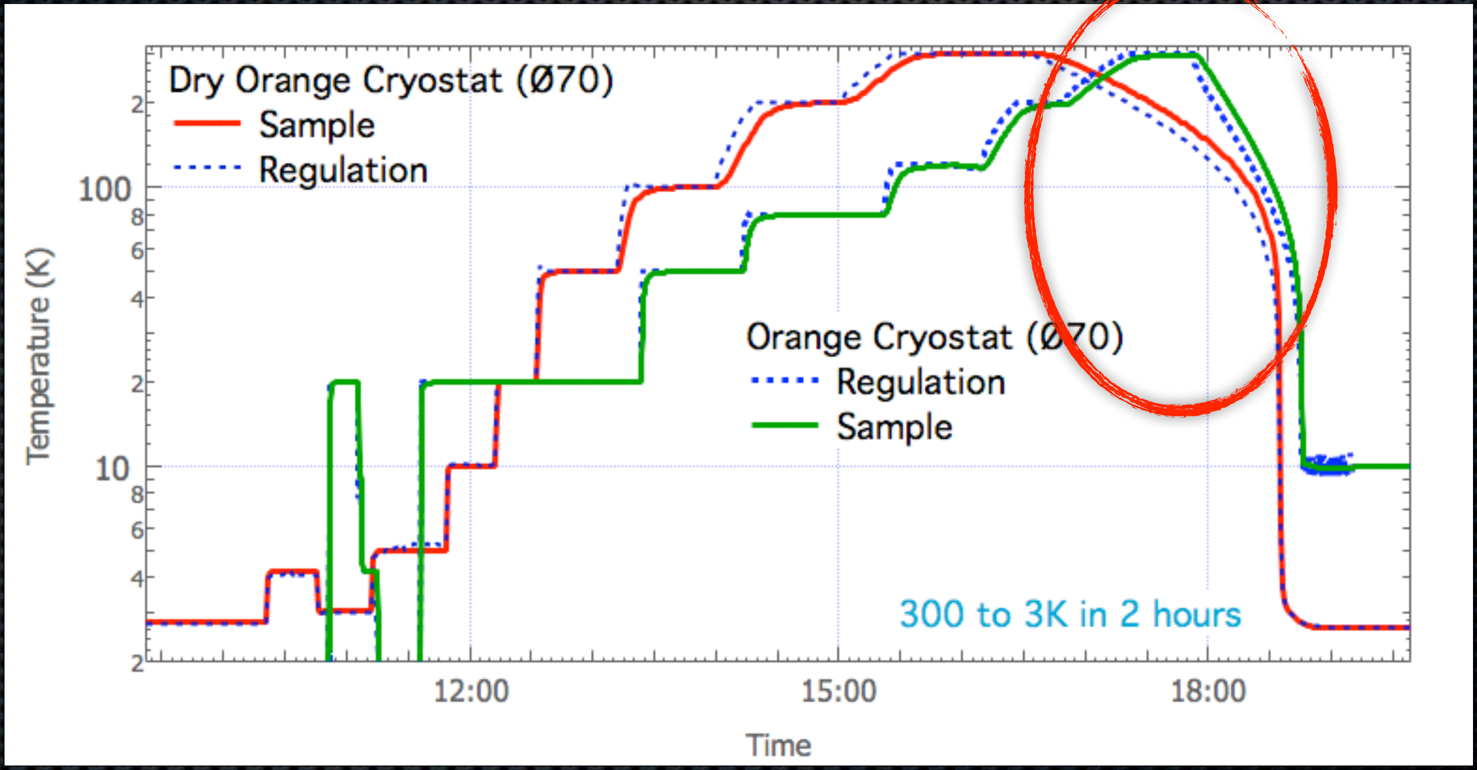
Cryogenics

…but cryogen-free cryostats are 2x slower !



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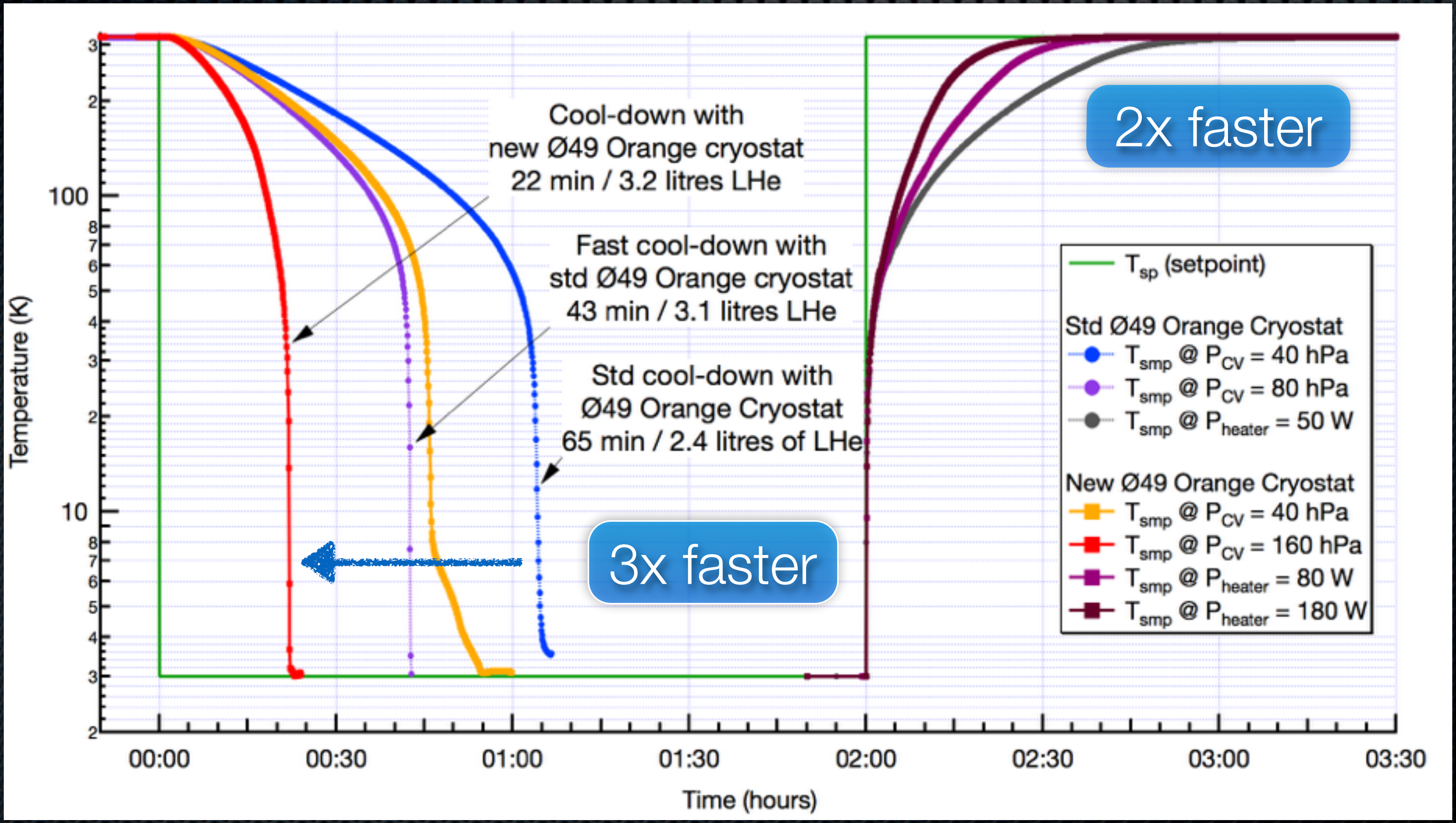
Cryogenics

Faster cryostats for new-generation instruments ?



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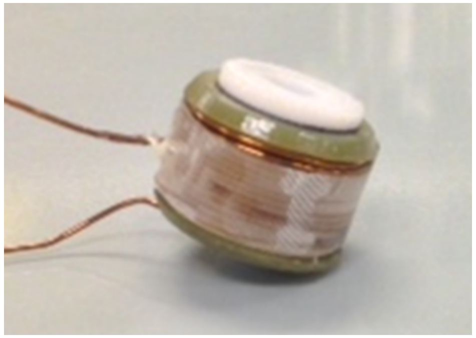
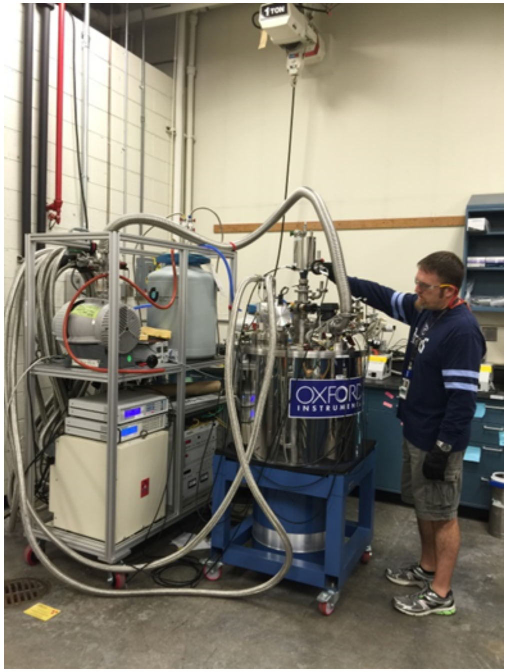
Magnets

Magnetic Field Equipment:

- 0.5-3 T electromagnet: specialized for Reflectometry or SANS
- 3-11 T superconducting cryomagnet, horizontal field
- 5-11 T superconducting cryomagnet, vertical field, symmetric or asymmetric
- 30 T pulsed magnet





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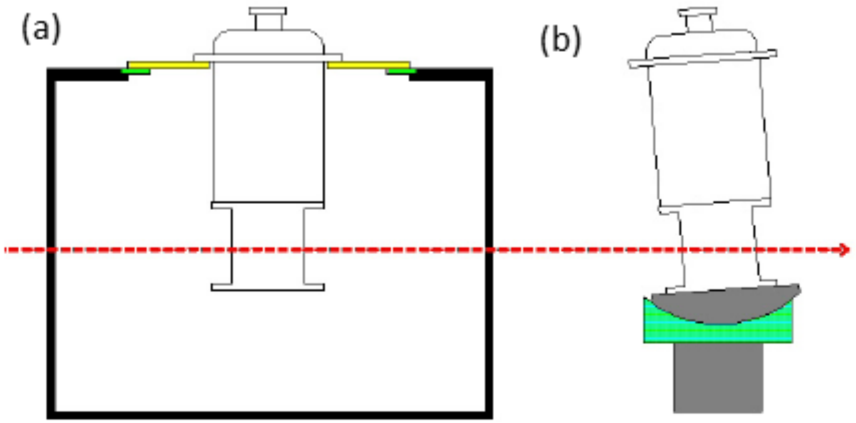
Magnets

Overall Physical Dimensions and Weight

- Flange mount or tail mount
- Flange diameter defines maximum diameter allowed (700 mm typical)
- Define flange: bolt holes, vacuum boundary, etc.
- Tail mount: distance from beam center to bottom of tail
- Maximum overall height (2200 mm typical)
  - Crane access: below the hook to mounting surface
  - Movement around the facility: through doors, etc.
- Total weight (including cryogens 450-680 kg) not to exceed crane capacity



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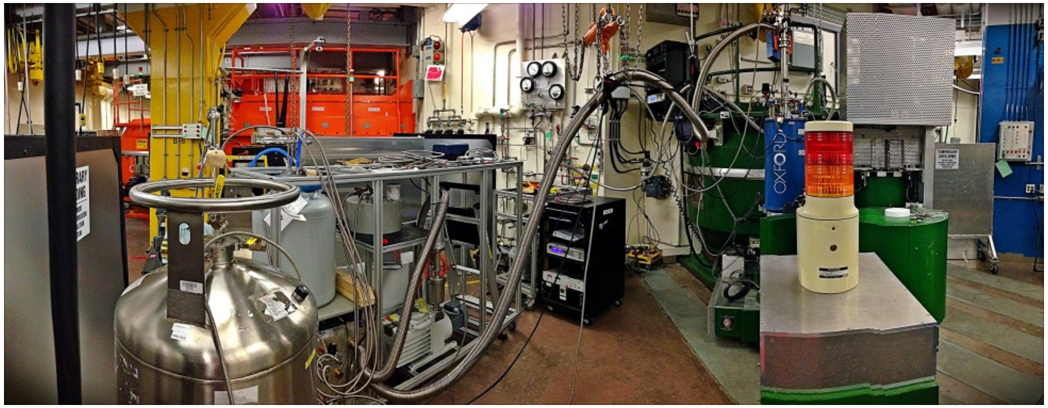
Magnets

Real Estate and Utilities

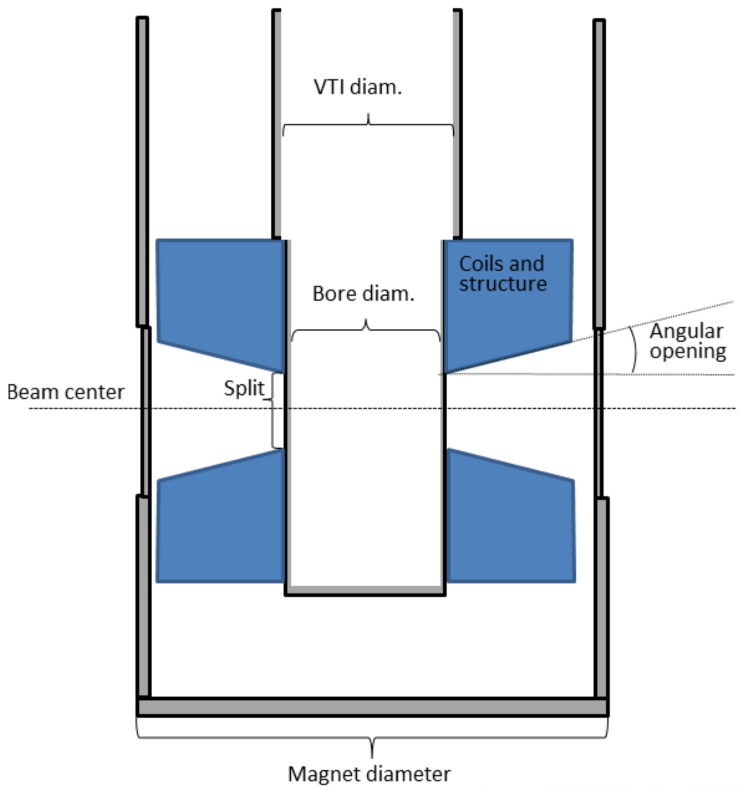
- Ancillary equipment such as power supply, Helium recondensing equipment, vacuum pumps, etc. can take up several square meters of space around the instrument
- Routing of vacuum lines, power and signal cables can be a little tricky
- Electrical power (U.S.):
  - 60 Hz at 110 V and 20 A for instrumentation
  - 60 Hz at 208 V and 30 A for power supply
  - 60 Hz at 480 V and 30 A for cold head compressor
- Chilled water



Magnets

Split Pair Magnet Dimensions

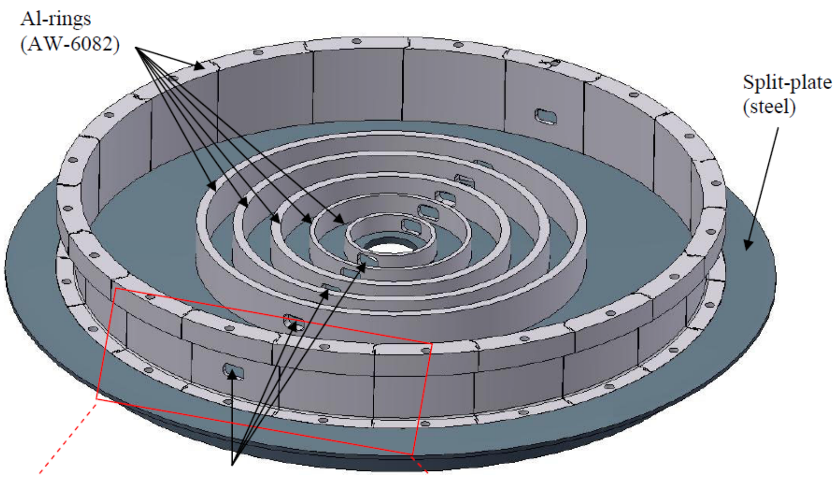
- Magnet diameter increases as magnetic field increases
  - Roughly 600 mm for 14 T uncompensated
  - Diameter can double for compensated
- Angular opening should be chosen to work with detector geometry (a few degrees is typical)
- Bore diameter should be matched to beam width available and low temperature inserts (VTI, DR; 50 mm typical)
- Split is the vertical height available for neutron beam between the magnet poles

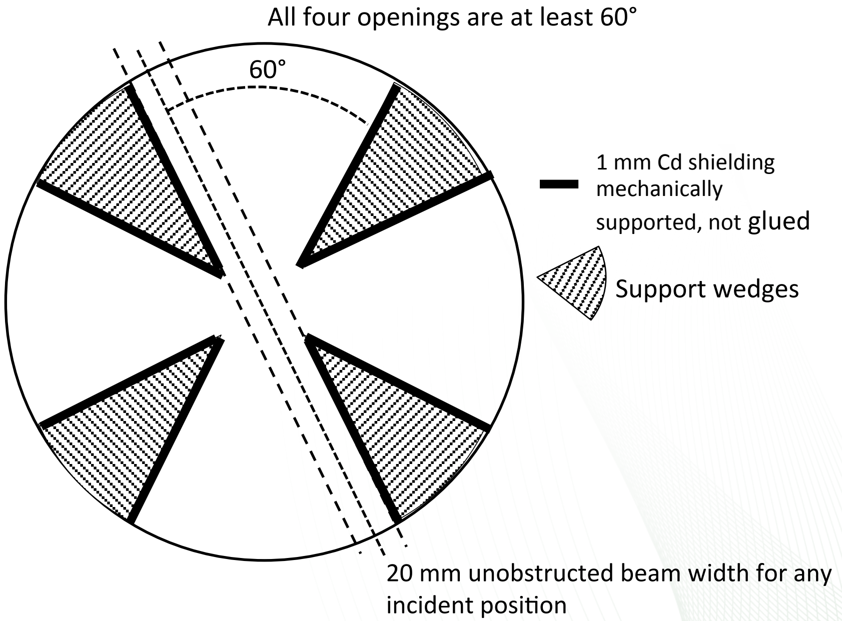


Magnets

Split Pair Magnet Support

- Provide structural support for the weight plus force from magnetic field
- Goal is to minimize the amount of Aluminum in the incident and scattered neutron beams
- Positioning of wedges to take up “dark angle” (where there is no detector coverage)





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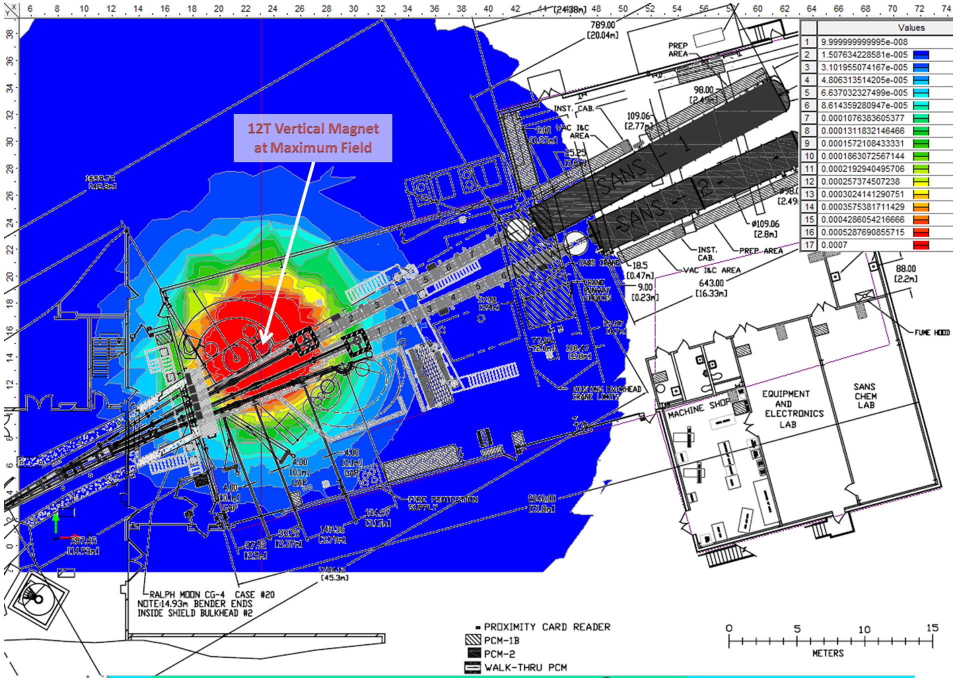
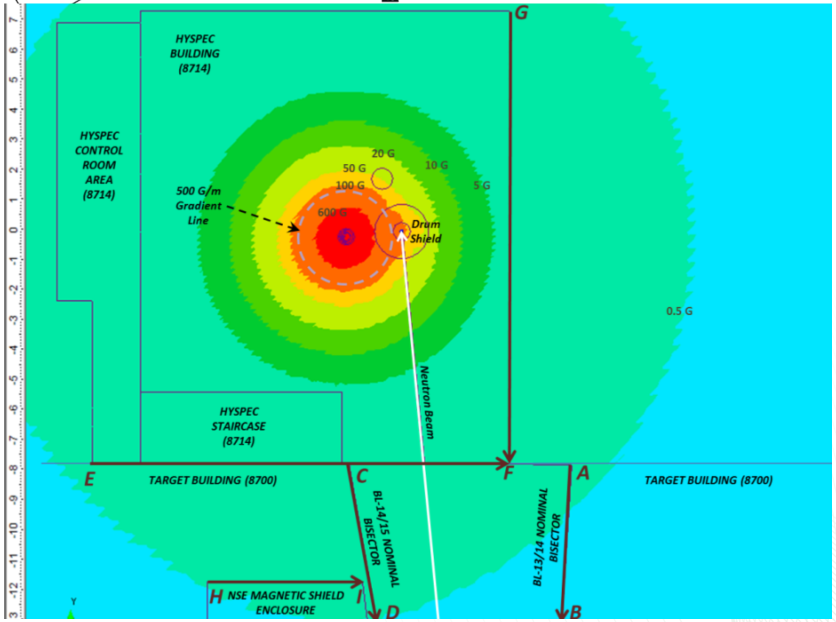
Magnets

Stray Fields

- Stray field plots usually provided by vendor
- Intensity decreases by the cubed of the distance
- What impact in the immediate area
  - 1 gauss: vacuum gauges, etc.
  - 5 gauss: pacemakers
  - 10 gauss: computers
  - 20 gauss: magnetic storage media
  - 50 gauss: magnet power supply, stepper motors





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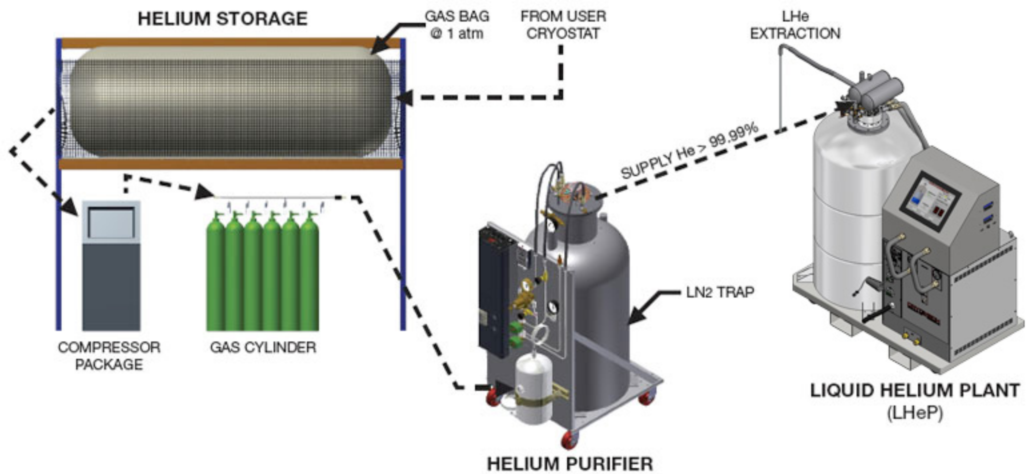
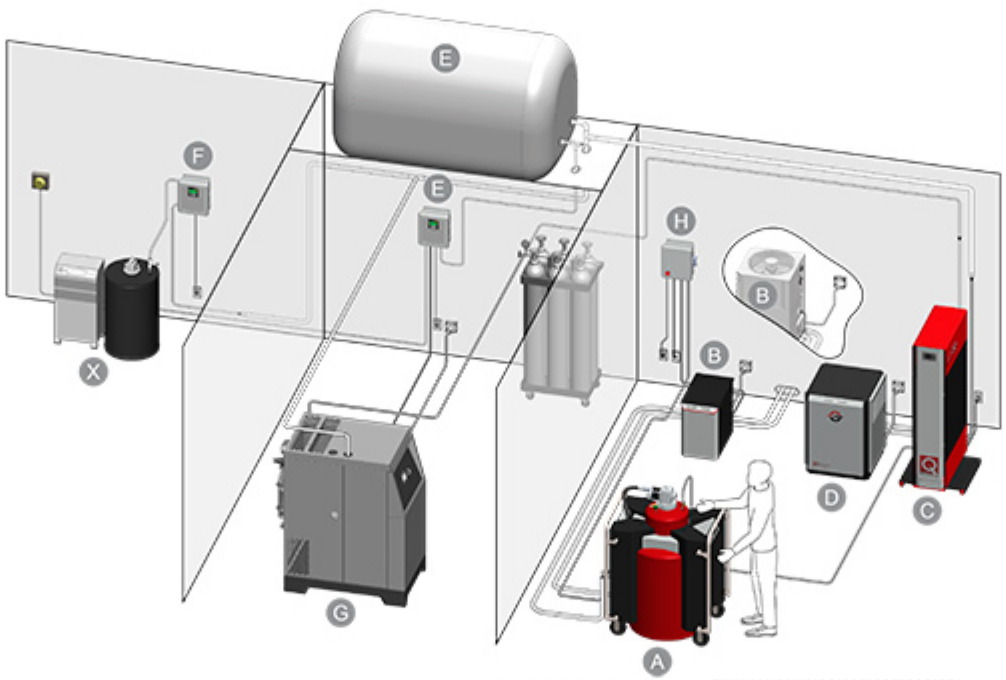
Cryogen Free?

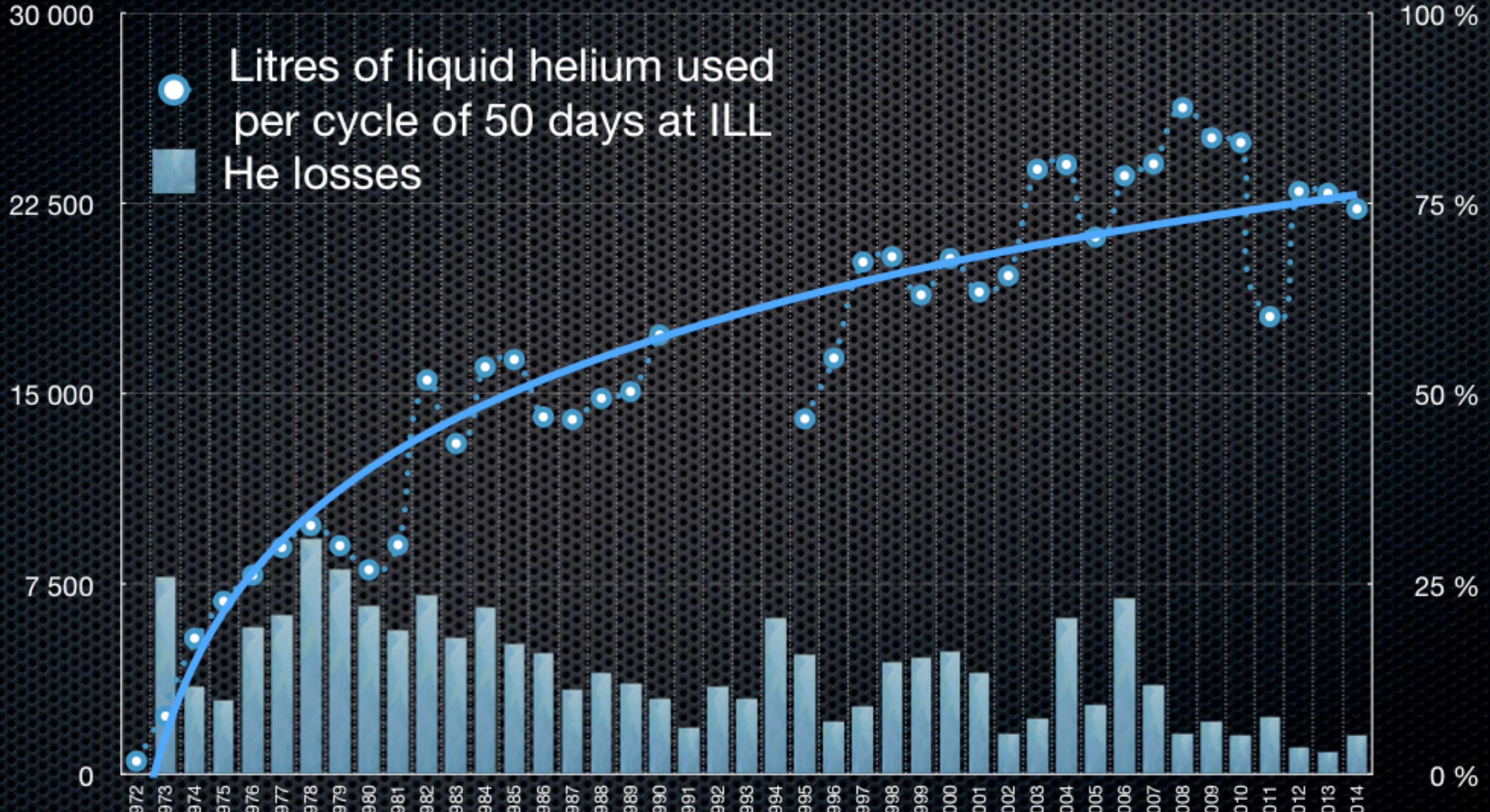
- In the U.S., helium supply is not a problem
- Liquid Helium usage about 15,000 Liters per year
- At a cost of $14.25 per Liter, $213,750 total expense
- Liquid Helium recovery system capital cost of $1,400,000
- 10 years for recovery of capital and operating costs
- Solar panels to provide power?





To summarise

NEUTRONS FOR SCIENCE



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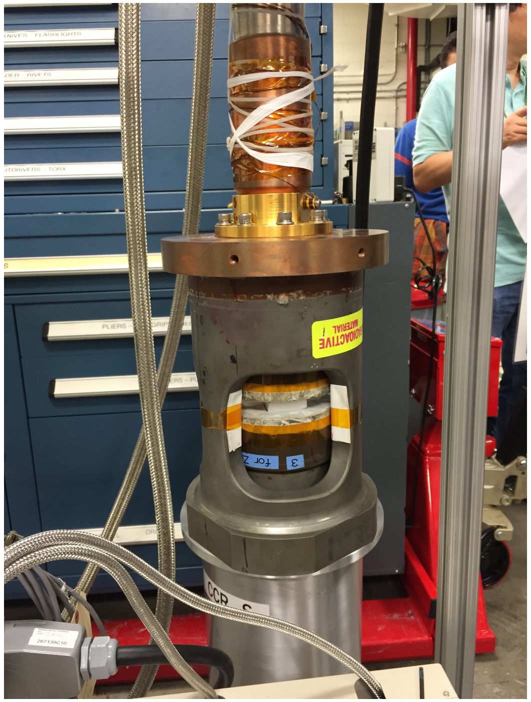
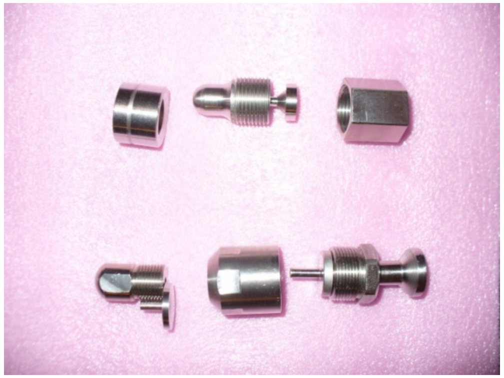
Pressure

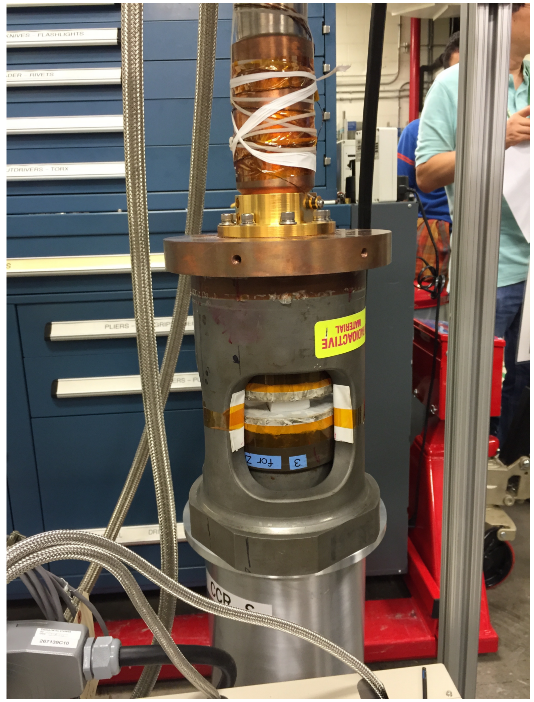


Pressure Equipment:

- 400 - 1300 bar V and TiZr cells for diffraction
- 6 kbar and 1.5 - 300 K Helium gas cells
- 4 GPa and 3.5 K Palm Cubic Anvil
- 1 - 3 GPa and 0.3 - 1.5 K Clamp cells for inelastic
- 10 - 40 GPa and 15 - 300 K Diamond Anvil Cells for diffraction



Pressure



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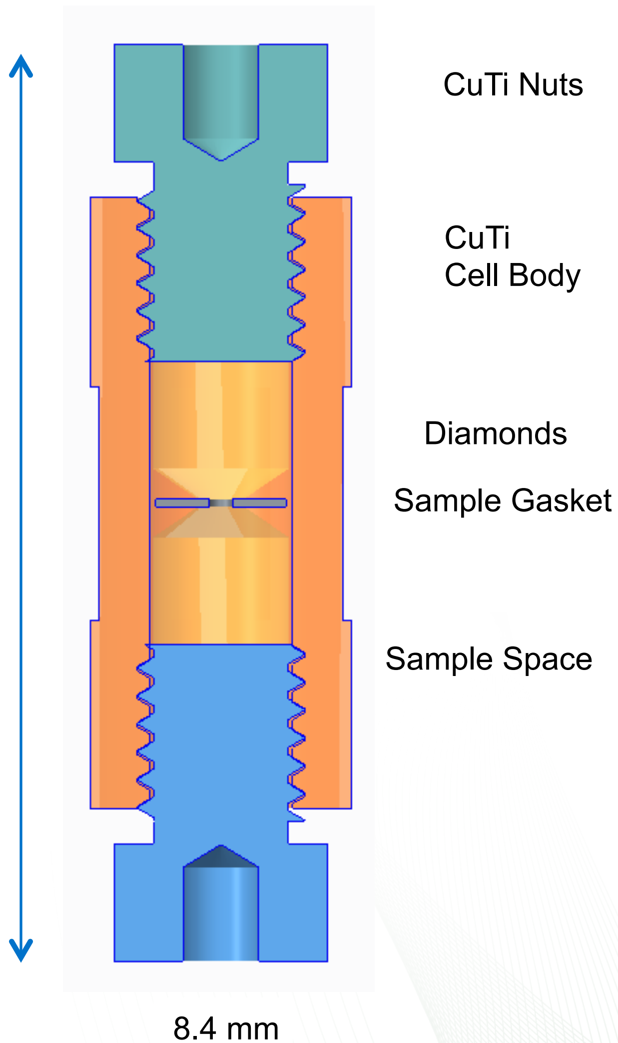
1 GPa = 10 kbar

Pressure

Clamp Cell

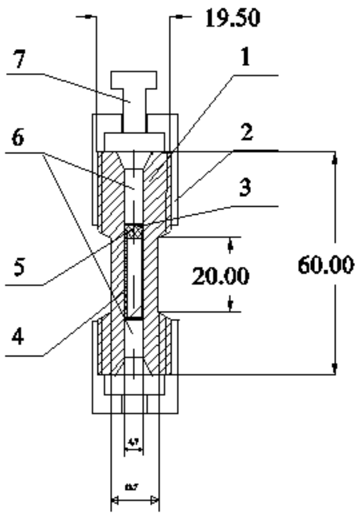
- Max. Load : 3.5 Tons for a wall thickness of 2mm.
- Sample Space: max. 0.3 mm diameter and 0.2 mm height.
- Pressure will depend on the culet size of the diamonds
- For a culet size of 1.7mm, Max Pressure approx.: 10GPa
- Disadvantages: Peaks from the Material
- Choices of Material for High Pressure Cells:
  - NiCrAl yield strength 2 GPa
  - CuBe yield strength 1.2 GPa
  - CuTi yield strength 1.2 GPa
  - Maraging Steel yield strength 0.8 GPa
  - TiZr yield strength 0.7 GPa



Pressure

Clamp Cell Design for Inelastic Scattering

- 500 mm^3 sample volume for inelastic scattering
- Fit in bore of magnet
- Non-magnetic material
- High thermal conductivity material to cool below 4 K
- Cell components have similar coefficients of thermal expansion
- Yield strength of material sufficient to pressurize up to 1.5 GPa



1- Body-nonmagnetic HNU (Ni-Cr-Al) alloy
2- Clamping nut-nonmagnetic Ti alloy
3- Extrusion ring-CuBe alloy
4- Capsule for sample (teflon or lead)
5- Capsule cap (teflon or lead)
6- Piston of a cell- nonmagnetic HNU alloy
7- Piston for pushing out the sample and for generating pressure- nonmagnetic HNU alloy

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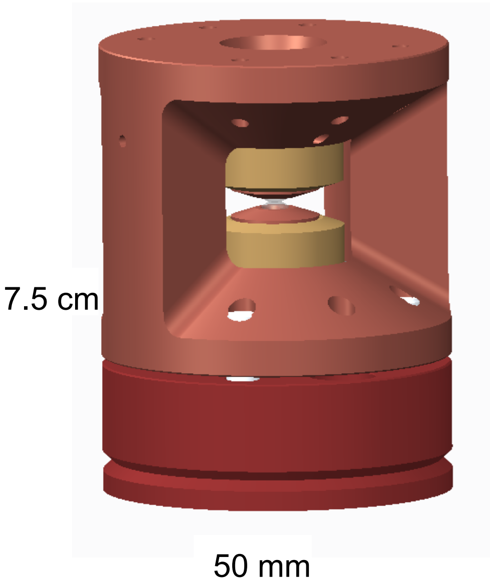
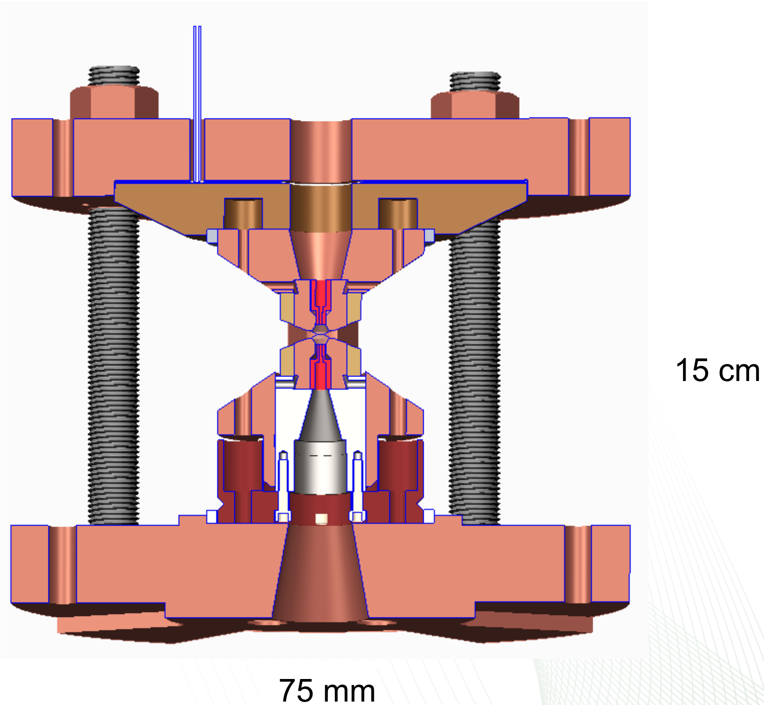
Pressure

Diamond Anvil Cell

- Max recorded Pressure for Neutrons: 94GPa.
- DAC can be made of Steel with Diamond anvils. For low temperatures, CuBe is used.
- Sample volume is limiting, in the order of 0.7mm in diameter and .16mm in gasket thickness
- Beamline Background reduction and collimation is of utmost importance for diamond anvil cell measurements.

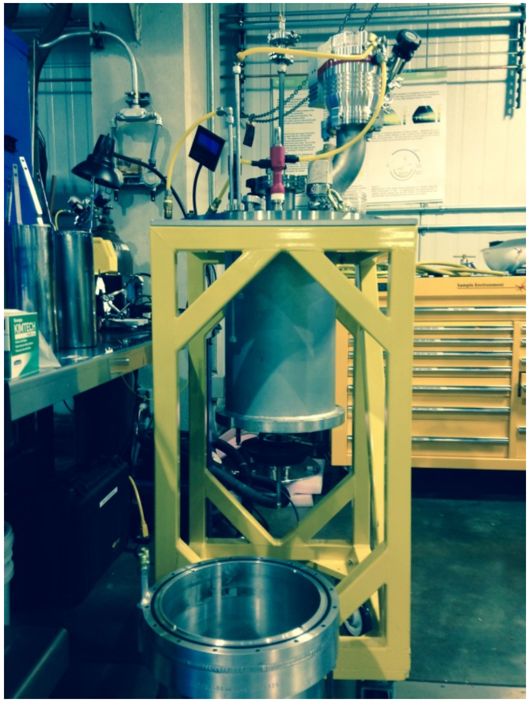


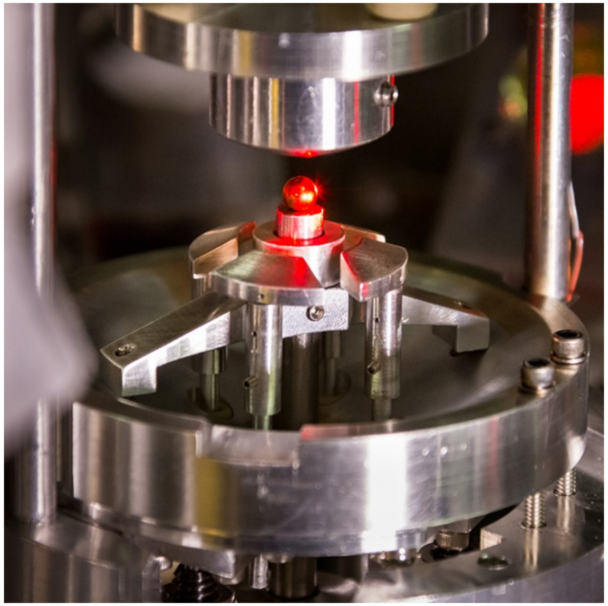


High Temperature

High Temperature Equipment:

- 1200 °C Vanadium ILL
- 1600 °C Niobium ILL
- 1200 °C or 1600 °C MICAS2
- 1500 °C Controlled Atmosphere
- 500 - 2000 °C Electrostatic Levitator





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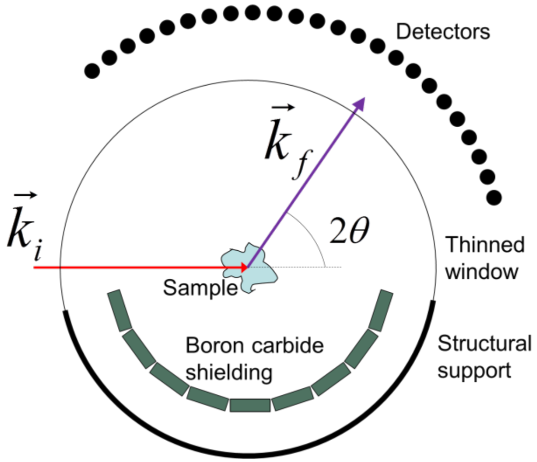
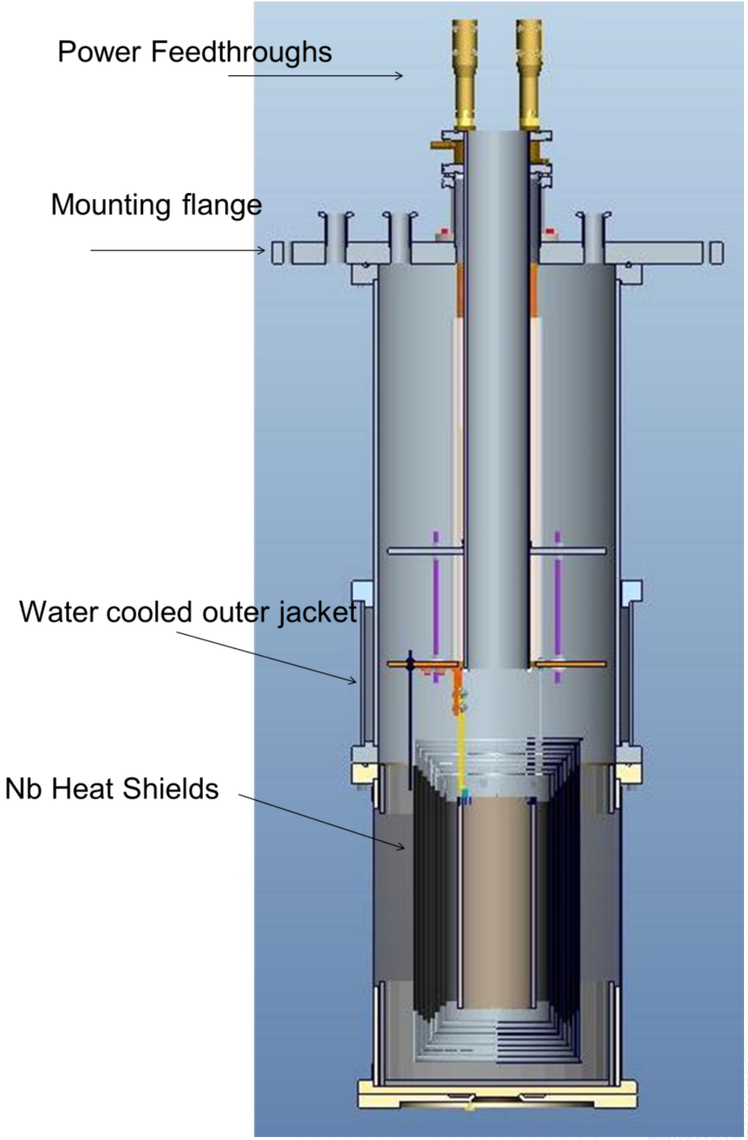
High Temperature

Radiative Heating Furnace

- Customize Outer Vacuum Chamber for detector coverage
- Minimize background using a thin (0.05 mm) Niobium window instead of Aluminum on Outer Vacuum Chamber
- Use of Boron Carbide to prevent multiple scattering





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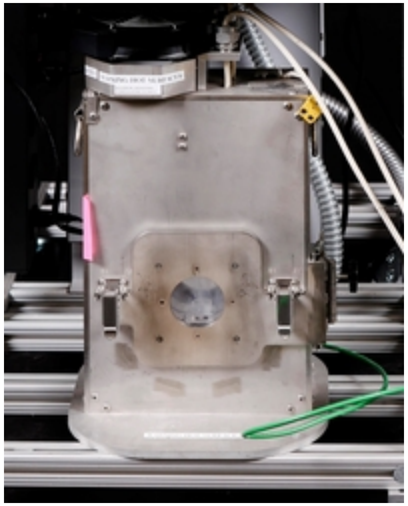
SANS

SANS Equipment:

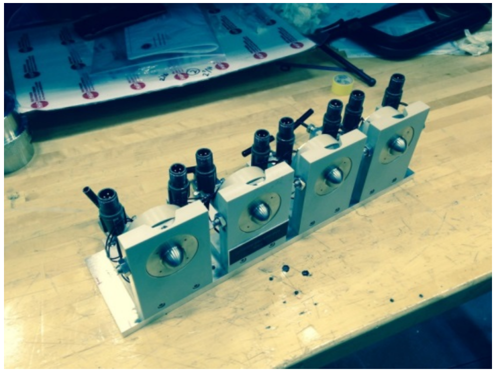
- 30 °C - 800 °C tube furnace with quartz windows
- 4.5 T superconducting cryomagnet, horizontal field, silicon windows
- Liquid Helium Cryostat 1.5 K - 300 K with sapphire windows, 200 bar pressure cell

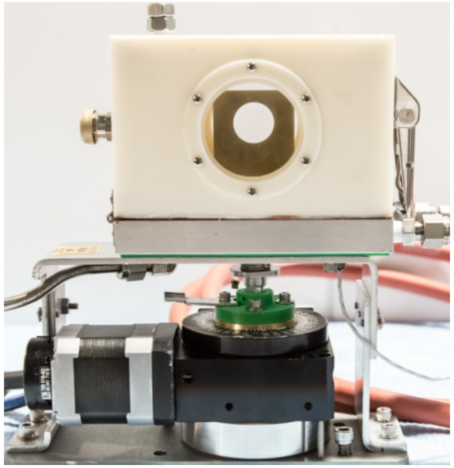


SANS

SANS Equipment:

- 1 kbar pressure cell, Tantalum lined
- 1 kbar 4-position pressure cells
- Sample tumbler
- Relative humidity controlled cell





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- Project management: budget, scope and time

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Sample Environment Design Considerations

- Build in as much flexibility as possible in the sample area: Experiments are continuously more complex
- Don’t skimp on the utilities: Electrical power, crane coverage, compressed air, chilled water, Helium recovery
- Remember your neighbors: Stray magnetic fields, neutron background

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