User Cryogenics at the NHMFL DC Field Facility

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Outline

• Helium (He) – the little atom that could.
• Getting to low temperatures using LHe.
• Some cryogenic sample environments at the MagLab.
• Getting your experiment cold & keeping it cold in high fields.
• Safety- (don’t turn your thesis into an I.E.D.)
• Thermometry options.
• Thoughts on basic probe design.
• Helium is a product of radioactive decay in the earth’s crust.
• Helium gas is present in some natural gas wells in the western United States at a concentration level of 3~7%.
• Helium is lighter than air—once released it eventually drifts into space.
• Recover helium!!!!!!
$^3\text{He}$

- $^3\text{He}$ is an isotope of $^4\text{He}$: one less neutron in the nucleus.
- It is a product of the radioactive decay of tritium.
- Very small abundance present in nature—very expensive.
- The supply of $^3\text{He}$ in the US comes from...
He at Low Temperatures:

- $^4\text{He}$ at 1020 mbar, $T=4.2$ K
- $^4\text{He}$ at 4.7 mbar, $T=1.5$ K
- $^3\text{He}$ at 200 mbar, $T=2.0$ K
- $^3\text{He}$ at 0.036 mbar, $T=0.4$ K

$^4\text{He}$ costs $5.60$/liter (liquid)

$^3\text{He}$ costs $640$/liter (gas STP) = $423,680$/liter (liquid)
Thermal Isolation-Less is More!

James Dewar invents the vacuum flask in 1892 as part of his work to liquefy the “permanent” gases.

Glass vacuum flasks can (and do) implode! Safety glasses would have saved the eyesight of a couple of lab assistants...

In honor of this invention, vacuum insulated cryogenic containers are called *dewars*...
How Important is Vacuum as an Insulator?

• Surface temperature of the sun = 5778 K
• Room temperature = 292 K
• Lowest MagLab Temperature = 0.007 K

• Factor of 19.8 between room temperature & sun’s surface.
• Factor of ~41,000 between room temperature & lowest temperature at the MagLab.
• Outer Vacuum Can (OVC) provides insulation

• Multi-Layer Insulation (MLI) a.k.a. superinsulation wrapped to reduce radiative losses.

• LN$_2$ shields LHe space

• Long neck reduces heat load into LHe space.

• NEVER PUT HELIUM GAS INTO THE OVC!!!!!!!
1.5 \text{ K} < T < 300 \text{ K}

Sample temperature is controlled by controlling the evaporator temperature.
Top Loading, sorbtion pumped, $^3$He insert

$0.3 \, K < T < 70 \, K$
Dilution of $^3$He into $^4$He

$T>0.85$ K $^3$He mixes into $^4$He homogeneously-like milk and water

$^3$He + $^4$He

$T<0.85$ K Phase separation occurs-like ice floating on $H_2O$

100% $^3$He

90% $^4$He + 10% $^3$He

$T=$absolute zero: lower phase is 93.6% $^4$He and 6.4% $^3$He
Continuous Cooling using Dilution of $^3\text{He}$ into $^4\text{He}$

100% $^3\text{He}$

90% $^4\text{He}$ + 10% $^3\text{He}$

Upside down evaporator!

Lowest temperature ever achieved with a DR $\sim$2.5 mK
Dil. Fridge *The Movie*

20 mK < T < 1.5 K

- Vacuum Pump
- 1 K Pot
- Condenser
- Flow Impedance
- Mixing Chamber
- Experiment
- Magnet
- IVC
- Still
- Heat Exchangers

**Symbols:**
- $^4\text{He}$
- $^3\text{He}$
- $^4\text{He} + ^3\text{He}$
At low temperatures heat is transmitted through two possible channels:

1. **Conduction Electrons:** High electrical conductivity materials are also high thermal conductivity materials according to the Wiedemann-Franz law: $K = \sigma \mathcal{L} T$ ($\mathcal{L} = 25 \text{nW} \Omega^{-1} \text{K}^2$)

2. **Phonons:** Vibrations in the lattice of a solid or through liquid helium. The phonon spectrum becomes very limited at $T < 100 \text{ mK}$. Scattering due to boundaries is the dominant limitation to phonon transmission.

How do we ensure good thermal contact to the measurement?

I. Heat sink wires leading to sample/low temperature region.

II. Have the sample in liquid/vapor environment.

III. Minimize # of interfaces that heat flow will experience.
Thermalization Pitfalls....

- **High Conductivity Wiring**: Copper is a great choice for carrying current and a lot of heat - thus wires need to be heat sunk very well or they will put heat directly into your measurement.

- **Kapitza Resistance**: Thermal boundary mismatch between two materials. Phonons moving between materials are reflected at the boundary. Becomes a very large effect below 100 mK. Can be overcome with large surface areas.

- **Self Heating**: When the detection method used heats the sample above the bath temperature, i.e. too much current through a resistor. Noise put out by the detection equipment can also result in self heating that is not accounted for in the measurement current.

- **Eddy Current Heating**: Low resistance metals in rapidly changing magnetic fields have an induced current which dissipates energy in the metal.

- **Levitation**: Helium is magnetic. It will levitate at a high field in a high gradient. We have both at the NHMFL....
Heat Sinking Wires

Kapitza Resistance

From Snell’s Law:

\[
\frac{\sin \theta_i}{\sin \theta_t} = \frac{C_i}{C_t}
\]

For He \(C_i=238\) m/s
& Cu \(C_t=5000\) m/s

For minimum transmission into Cu \(\theta_t=90^\circ\).

\[
\theta_i = \sin^{-1} \left[ \frac{C_i}{C_t} \right]
\]

\(\theta_i<3^\circ\) !!
Self Heating - a cautionary tale...

Self heating test in the portable dilution refrigerator using a calibrated ruthenium oxide sensor measured with a LS 370 bridge. RuO test sensor is located at field center position.

Temperature (mK)

Test Sensor Power (W)

2 K!

50 mK!

Probe Thermometer

Test Thermometer

I increased stepwise

100 mm
...but I am using a $^3$He system with a lot of cooling power and I am at 500 mK, so I don’t need to worry because the force is strong with me...

![To the hand you talk](quickmeme.com)
Self Heating - The Silent Experiment Killer

2 sensors glued together

Graph showing temperature vs. power with markers indicating differences in temperature:
- Sample Temperature
- Temperature Sensor (glued to sample)
- Liquid Temperature

Temperature (K) vs. Power (W) graph with markers showing:
- ΔT = 97 mK
- ΔT = 268 mK
- ΔT = 81 mK
- ΔT = 953 mK
...but my sample is only milliohms of resistance....

\[ V_s = 0.5 \, \mu V \]
\[ R_S = 1 \, m \Omega \]
\[ R_C = 5 \, \Omega \]
\[ I = 500 \, \mu A \]

\[ P = 2.5 \, \mu W \]
... and The Agony of Self Heating

\[ P = 2.5 \, \mu W \]
• $^3$He and $^4$He are diamagnetic—pushed to lower field when exposed to a gradient.

• Since liquids are more dense than gases they are more magnetic

• Bubbles are trapped at field center while liquid is expelled when $F_H > mg$ by the following: $F_H \propto [H^*dB/dZ]$  

• Levitation occurs when $[H^*dB/dZ] > 21T^2/cm$ which is around 20T in resistive magnets.
Eddy Current Heating

\[ P_e = \frac{\pi r^4 L \left( \frac{dB}{dT} \right)^2}{8\rho} \]

Use high resistivity materials when possible

Minimize the o.d. of metal parts

Can use eddy currents to reduce ripple in applied magnetic fields-Faraday shield
Thermoacoustic oscillations that occur in tubes that have one end in LHe and the other end at a high (room) temperature.

Can carry watts of heat into the bath.

As demonstrated here:
Safety-Where things can go wrong
Not a comprehensive list...

- **Cryogenic burns**: severe damage to living tissue-eyes are especially susceptible.
- **Asphyxiation**: $V_{\text{gas}}/V_{\text{liq}} = 701$ for He.
  - Canonical laboratory size- 20’ X 20’ X 12’ = 4800 ft$^3$=1.36 X $10^5$ L= 193 L of LHe to displace all the air!
  - 2 breaths of a 0% oxygen atmosphere result in immediate unconsciousness-equivalent to getting hit in the head with a hammer.
  - We do not need to displace all the air, only drop the O$_2$ concentration by ~10% for bad things to start happening.
- **Embrittlement**: Not all materials are suitable for use at low temperatures-consult one of the many references available on this topic.
- **Pressure Buildup**: High pressure gas stores great amounts of energy. Beware solid air condensed inside the OVC of a dewar after being cold for a long time....
- **Oxygen Enrichment on Cold Surfaces**: At $T<82$ K metal surfaces will condense a 50% N$_2$-50% O$_2$ mixture. Can be a hazard depending where it is dripping.
Small Scale Pressure Buildup

• 20 cm$^3$ of $^4$He liquid = 13 liters of gas @ STP when the $^4$He is warmed to 300K.

• If the volume is held constant then $P=71$ bar or 1035 psi!

• When your experiment warms up the gas will find a way to escape. Planned release is better than the materials test approach....

• Passive pressure reliefs allow the cryogenic system & researcher to survive unforeseen operation hazards.
Large Scale Pressure Buildup

Dilution refrigerator expelled from top of dewar by overpressure. Bolts ripped from dewar top plate.

50 L LHe = 43,300 L He gas

Dewar support bolt completely ripped out

Concrete anchor pulled out along with concrete!
Large Scale Damage

Note: Bottom of dewar has been blown off by force of expanding gas.
Sample in vacuum: High thermal conductivity coldfinger connects experiment to source of cooling. Useful for specific heat measurements, thermal conductivity and optical measurements.

- **Pros:** Well defined thermal path, no liquid or gas to interfere with optical beam path or heat decay.
- **Cons:** More difficult to cool samples, self heating becomes a larger issue, much more metal (Cu or Ag) in high fields resulting in eddy current heating.
Cryogenic Sample Environments-
Sample in Vacuum- Cell 5

- Top loading VTI-in vacuum.
- Sample loads in 30 min.
- 5 K - 300 K
- Direct optical access!
- OVC=IVC=Magnet Bore!
• **Sample in liquid:** Sample is immersed directly in the cryogenic fluid; $^4$He and/or $^3$He.

  • **Pros:** Good thermal contact between the sample and the cooling medium, larger measurement currents possible, really fast sample changes.

  • **Cons:** Poorly defined thermal path—not suitable for specific heat measurements, can be trouble for optical experiments, influence of liquid on dielectric constant for capacitance based measurements.
• Pull out probe to change samples instead of cryostat out of dewar.

• 0.3 < T < 70 K $^3$He insert

• 1.5 < T < 300 K VTI

• Typically no leak check on probe change.

• As little as 30 minutes to load probe.

• 1.5 hour total cycle time for probe change.
Choosing a Thermometer for High Fields

- Cernox resistance sensors: (325 K-0.3 K) have low magnetoresistance to ~ 4 K.

- RuO$_2$ resistance sensors: (1.5 K- 0.01 K) have ~10% magnetoresistance at low temps.

- Most capacitance thermometers are nominally field insensitive but temperature sensitivity is low below 500 mK. Drift for ~ 24 hours after cooling, useful within 4 hours. Cannot be calibrated.

- Vapor pressure: (4.2 K - 0.4 K) field independent, lack of sensitivity to local heating effects.
Cernox Sensors: 0.5K<T<300K

Advantages:
• Sensors are easy to use.
• Wide temperature range
• Magnetoresistance can be calibrated

Disadvantages:
• Significant magnetoresistance below 2K.
• At 0.25 K & 18 T $\Delta T/T = 100\%$

Ruthenium Oxide: 0.010K<T<1.5K

- Good sensitivity below 1.5 K.
- Weak localization is a problem at low fields & low temps.
- Possible to calibrate magnetoresistance.

Some Parts of the Road are Paved...

• **6 months in the lab can save you 6 hours in the library...**

• *Experimental Techniques for Low-Temperature Measurements*, Jack W. Ekin

• *The Art of Cryogenics*, G. Ventura and L. Risegari

• *Handbook of Cryogenic Engineering*, J. G. Weisend II

• *Experimental Techniques In Condensed Matter Physics At Low Temperatures*, R. Richardson and E. Smith

• *Experimental Principles and Methods Below 1K*, O.V. Lounasmaa

• *Experimental Techniques in Low Temperature Physics*, G. K. White
Probe Design I: Functionality... (what’s it supposed to do?)

- Temperature Range?
- Liquid or Vacuum?
- Magnetic Field Range?
- Number of Wires?
- Motion?
- Optical Access?
- How Much Space?

Everything Starts Here!
‘Everything should be kept as simple as possible but no simpler.’ - Einstein constraint

Simple to Use ≠ Easy to Design!
Probe Design III: Modularity

Flanges are your friend!

flat tire ≠ new car!!!!
Murphy’s Laws predate Newton’s Laws....

If you build it-you will fix it.

*Test* the finished product-don’t assume

Don’t be this guy!
Conclusions

• Details are the evil flying monkeys of low temperature, high magnetic field measurements!

• For the user systems at the NHMFL many of the low temperature details have already been taken care of but...

• You must understand your measurement to ensure that the results are correct!

• Successful designs should reflect: functionality, simplicity, modularity and durability.

• Be safe and have fun!