Measurements of ultralow temperatures

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21.3.2006 Measurements of ultralow temperatures

Outline

- Motivation
- Thermometry below 1K
- Methods below 1K
- (Adiabatic melting experiment)

Differential pressure gauge disconnected from the cell and thermally anchored to mixing chamber

Pressure gauge volume connected to cell via a superleak

Vibrating wire resonators replaced by quartz tuning fork resonators

Another platinum NMR thermometer in thermal contact with liquid

Liquid $^3$He–$^4$He mixture

Sintered silver

Liquid pure $^3$He

Solid $^4$He

Filling line

Reference volume, pure $^4$He

Platinum NMR thermometer

Superleak entrance

Liquid $^4$He bath (4.2 K)

$^4$He pot (1.2 K)

Stil (0.7 K)

Heat exchangers

Mixing chamber (3 mK)

Adiabatic melting experiment cell (~300 mK)

9 T superconducting solenoid

Vacuum

Copper nuclear stage (<100 mK)
Motivation

• Why tedious refrigeration is worthwhile?
  – Reduced thermal noise
  – Many fundamental properties of matter were found/understood only after matter had been cooled to the kelvin range or even lower.
    • Quantization of lattice vibrations
    • Electronic excitations leading to linear temperature dependence of specific heat
    • Superconductivity, superfluidity
    • Magnetism related phenomena
    • …
History

Low temperature research started in the 19th century with two aims:
• Refrigerate meat for its journey from other continents to Europe
• Discover whether permanent gases exists

<table>
<thead>
<tr>
<th>Temperature range</th>
<th>Refrigeration technique</th>
<th>Available since</th>
<th>Typical $T_{\text{min}}$</th>
</tr>
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<tbody>
<tr>
<td>Kelvin</td>
<td>Helium-4 evaporation</td>
<td>1908</td>
<td>1.3 K</td>
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<tr>
<td></td>
<td>Helium-3 evaporation</td>
<td>1950</td>
<td>0.3 K</td>
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<td>Millikelvin</td>
<td>$^3$He-$^4$He dilution</td>
<td>1965</td>
<td>10 mK</td>
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<td></td>
<td>Pomeranchuk cooling</td>
<td>1965</td>
<td>3 mK</td>
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<tr>
<td></td>
<td>Electronic magnetic refrigeration</td>
<td>1934</td>
<td>3 mK</td>
</tr>
<tr>
<td>Microkelvin</td>
<td>Nuclear magnetic refrigeration</td>
<td>1956</td>
<td>50 µK</td>
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</table>
Thermometry

• It is important to relate low temperature measurements to thermodynamic temperature scale
  – Compare experimental and theoretical results
  – Comparability of experimental results from different institutes

• Thermometers can be divided into two classes
  – Primary thermometers (no calibration needed)
  – Secondary thermometers (needs to be calibrated)

• In many cases thermometry is as difficult as attainment of the temperature
• A good thermometer has to fulfill several requirements;
  – The property to be measured, \( x \), must be easily, quickly and exactly accessible to an experiment.
  – The temperature dependence of the measured property, \( x(T) \), should be expressible by a reasonably simple law.
  – The sensitivity \( (\Delta x/x)/(\Delta T/T) \) and the dynamic range should be high.
  – The thermometer should reach equilibrium in a "short" time, both within itself and with its surroundings whose temperature it is supposed to measure. Therefore it should have a small heat capacity, good thermal conductivity and good thermal contact to its surroundings. In particular, the thermal contact problem is ever present for thermometry at \( T < 1 \) K.
  – The relevant measurement should introduce a minimum of heat to avoid heating of the surroundings of the thermometer and, of course, above all, heating of itself; this becomes more important lower the temperature.
• Some of the above mentioned requirements are contradictory with each other in low temperature experiments. When designing the thermometry for the experiment, it is important to carefully inspect what are the most essential requirements and emphasize those when implementing the thermometer.
Temperature standards

- **ITS-90**
  - In the standard temperatures from 0.65 to 5 K are defined by helium vapor pressure
  - Lowest fixed point: triple point of e-H₂ at 13.8033 K
- **PLTS-2000**
  - From 0.9 mK to 1 K temperature defined by melting curve of ³He
  - Lowest fixed point: Néel transition (in solid ³He) at 0.902 mK
- In addition some national laboratories of standards (namely NIST) have made devices consisting of arrays of superconductors to be used as fixed points, also noise thermometry and josephson junction devices
  - SC transitions not reliable, too dependent on external fields and small fractions of impurities (also does not go to sufficiently low temperatures), other two can’t really say
- **No chain of calibration!**
  - Many times in the low temperature literature only relative temperatures are reported, for example 0.2 Tₖ
Thermometers

- During cooling from room temperature
  - Pt-100, RT – 4 K
  - Germanium resistor, 50 K – 50 mK

- When operating at low temperature
  - Carbon resistor thermometers, 5 K – 10 mK
  - Co-60 nuclear orientation thermometer, 40 – 2 mK
  - Platinum NMR thermometer 50 mK – 10 µK

- For liquid mixture
  - Melting pressure thermometry, 100 mK – 300 µK
  - Oscillating temperature sensors, 100 mK – 300 µK

The world record low (nuclear spin) temperature of 100 pK set in year 2000 was determined by tedious analysis based directly to the second law of thermodynamics:

\[ T = \frac{\Delta Q}{\Delta S}, \]

Where \( \Delta S \) is the entropy change and \( \Delta Q \) a known amount of heat.

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Resistance thermometry

- Is based on temperature dependence of resistivity of selected materials
- Carbon resistors and semiconductors are used
- For carbon resistors
  \[ R(T_m) = R_i e^{(T_0/T_m)^p} + R_0, \]

where \( T_0 \) is the temperature of the resistor, \( R_i \) resistance at infinite temperature and \( R_0 \) the temperature independent resistance (\( T_0 \) and \( p \) fitting parameters)
- Heating causes a temperature difference between the resistor and studied substance
  \[ T_m - T^n = nr\dot{Q}, \]

where \( n \) depends on the nature of thermal contact and \( r \) is the thermal boundary resistance
- And finally combining previous equations
  \[ R(T) = R_i e^{[T_0/(nr\dot{Q}+T^n)^{1/n}]^p} + R_0. \]

- Unstable against thermal cycling
- Saturates at about 10 mK (works to some extent to lower temperatures)
- Response time increases as temperature decreases
**Co-60 thermometer**

- **Important as primary thermometer**
  - We use it to calibrate more sensitive thermometers
- **Thermometer is based on measuring the anisotropy of γ-rays emitted from polarized nuclei**
  - Nuclear levels are split by magnetic field into \((2I+1)\) hyperfine levels (\(I\) is the spin quantum number of nucleus)
  - The population numbers of the sublevels follow Boltzmann statistics and so depend on temperature
  - On the other hand orientation of nuclei vary from level to level -> temperature depended angular radiation pattern \(W(\phi, T)\)
- **Sensitivity is reasonable between 3 and 30 mK, at higher temperatures all energy levels are equally populated and at lower temperatures only the lowest level is populated.**
- **Measuring one spectrum takes roughly one hour**

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Pt-NMR thermometer

- Sample is placed in a static magnetic field $B_0$
- Susceptibility of nuclear spins follows the Curie law
  \[ \chi_n = \Lambda_n / T \]
- We measure the magnetization $M_n$ by applying an excitation pulse at the Larmor frequency to tip the magnetization by a small angle $\theta$
- After excitation pulse there is a component
  \[ B_y = B_1 \sin(\omega_n t) \]
  precessing in the XY-plane
- This component induces a voltage
  \[ U = \alpha \omega_n M_n \sin \theta_n, \]
  to pick-up coil.

This signal is called the free induction decay and its amplitude is directly proportional to magnetization and thus inversely proportional to temperature.
21.3.2006 Measurements of ultralow temperatures 12

Fid signal

- Two important timescales
  - $\tau_1$ determines nuclear spin systems relaxation time to electron system’s temperature
    $\tau_1 = \frac{\kappa}{T_e}$, where $\kappa$ is Korringa constant (0.030 sK for Pt, $\tau_1 \sim 5$ min at 100 $\mu$K)
  - $\tau_2$ determines spin-spin relaxation time. Sufficient time is needed for measurement to be made. For Pt $\tau_2 \sim 1$ ms
- In addition Pt has only one magnetic isotope $^{195}$Pt with nuclear spin $\frac{1}{2}$ -> simplifies signal
- Pt is a good sample material
Pt sample

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Pt-NMR limitations

- Although the method itself would work to extremely low temperatures (at least 0.1 µK) the thermal contact with the sample becomes a problem.
- In our last cooldown measuring the liquid temperature by Pt-NMR was not reliable at low millikelvin temperatures (although we could measure temperature of cell wall where the probe was connected more accurately).
Measuring the mixture temperature

• There are some additional difficulties in measuring the mixture’s temperature
  – Extremely high thermal resistance between the liquid and any metal prevents the use of most methods -> temperature must be deducted from some property of the liquid itself
  – The experiment is very sensitive to heatloads introduced directly to liquid -> thermometry should introduce less than pW of heating
**Melting pressure thermometry**

- Melting pressure thermometry using pure $^3$He is a widely approved and reliable method for measuring temperatures at the millikelvin range.
- The same concept can be applied for the melting pressure of the crystal forming in the $^3$He/$^4$He mixture.

\[
\left( \frac{\partial p_m}{\partial T} \right) = \left( \frac{\partial \Delta \mu}{\partial T} \right)_p = \left( \frac{\partial \Delta \mu}{\partial p} \right)_T
\]

\[
\frac{\partial p_m}{\partial T} = \frac{(1 - x_s)(S_4^L - S_4^S) + x_s(S_3^L - S_3^S)}{(1 - x_s)(v_4^L - v_4^S) + x_s(v_3^L - v_3^S)}
\]

\[x_s \approx 10^{-25}\]

\[S_F = \frac{\pi^2 k_B T}{2T_F}\]

\[S \approx N_3 \frac{\pi^2 k_B T}{2T_F}\]

\[\frac{dp_m}{d(T^2)} = \frac{1}{2T} \frac{dp_m}{d(T)} = \frac{\pi^2 x_3 R}{6 \Delta v T_F} \propto x_3^{1/3}.\]
Melting pressure thermometry

- We measure capacitance by an Andeen-Hagerling AH 2500A capacitance bridge.
- The pressure sensing diaphragm is a circular BeCu membrane with a 29 mm diameter and 0.2 mm thickness.
Melting pressure thermometry

- The calibration measurement for pressure was done at 2.5 MPa pressure in the reference volume against a Bourbon-Haenni EDD 575 differential pressure transmitter at room temperature.
- The maximum measurable pressure difference was 41 kPa, at which the capacitor plates were shorted.
Melting pressure thermometry

- The mixture’s melting pressure is measured against the melting pressure of pure $^4$He.
- Peak to peak fluctuations of the capacitance is only about 20 aF and the sensitivity was 3.5 pF/Pa at 34.1 kPa, which suggest that thermometry should be possible even below 100 µK.
Melting pressure thermometry

• In the previous cooldown two problems prevented melting pressure thermometry at very low temperatures
  – deviation from the theoretical $T^2$ dependence of pressure
    • The thermodynamical equations must be solved accurately (also the pure $^3\text{He}$ phase must be taken into account)
    • Some properties of the liquid, mainly saturation concentration at melting pressure, must be measured accurately
  – heating caused by the operation of the capacitance bridge
    • capacitance measurement increased the heat load to the liquid by approximately 100 pW
      $\Delta T \sim 30 \, \mu\text{K} \text{ at } 330 \, \mu\text{K}$
Vibrating wire resonators

- Vibrating wire resonators are a widely used tool for studying helium liquids
- In the previous cooldown there were several VWRs in the YKI cryostat, two of which are inside the experimental cell
  - One in the pure $^3$He for thermometry ($\phi 50 \, \mu\text{m NbTi}$)
  - Other in the mixture for detection of the possible superfluid transition ($\phi 125 \, \mu\text{m Ta}$)
Vibrating wire resonators
Vibrating wire resonators

• The wire can be considered as a driven damped harmonic oscillator.

\[ m\ddot{x} + m\lambda \dot{x} + m\omega_0^2 x = C I_w B e^{i\omega t} \]

• Damping force is written as \( m\lambda \dot{x} \). The parameter \( \lambda = \lambda_2 + i\lambda_1 \) relates the studied fluid properties to a measurable quantity.

\[ \Delta f_1 = \frac{\lambda_1}{4\pi} \]
\[ \Delta f_2 = \frac{\lambda_2}{2\pi} \]
Vibrating wire resonators

\[ V_{0r} = \frac{C}{K} \frac{I_w B^2 D}{m} \frac{\omega^2 \lambda_2}{(\omega^0_0^2 - \omega^2 - \omega \lambda_1)^2 + \omega^2 \lambda_2^2} \]

\[ V_{0i} = \frac{C}{K} \frac{I_w B^2 D}{m} \frac{\omega(\omega^0_0^2 - \omega^2 - \omega \lambda_1)}{(\omega^0_0^2 - \omega^2 - \omega \lambda_1)^2 + \omega^2 \lambda_2^2} \]

\[ \text{Angular velocity (s}^{-1}\text{)} \]

\[ \text{Absorption} \]

\[ \text{Dispersion} \]

\[ \Delta_1 \]

\[ \Delta_2 \]

\[ \text{Vacuum peak} \]

\[ \text{Liquid peak} \]
Vibrating wire resonators

• For some geometrically simple cases the damping force and thereby the frequency shift $\Delta f_1$ and the resonance width $\Delta f_2$ of the mechanical resonator can be calculated.

• In the ballistic regime, the damping force for the pure $^3$He phase can be approximated by the formula:

$$F = A n f a v (Y_0 Y_2)^{1/2}$$

where $A$ is a numerical prefactor and $n$ the number of quasiparticles per unit volume, with fermi momentum $p_f$. The temperature dependence comes from the Yosida functions $Y_0$ and $Y_2$ that are related to the normal fluid density and defined as:

$$Y_n \left( \frac{\Delta}{kT} \right) = \frac{1}{2} \int_0^\infty dx \left( \frac{x}{\xi} \right)^n \text{sech}^2 \left( \frac{\xi}{2} \right)$$

• At temperatures below $0.4T_c$, the product $(Y_0 Y_2)^{1/2}$ is simply proportional to $\exp(-\Delta/kT)$, where $\Delta$ is the energy gap.
Vibrating wire resonators

- Measurements were possible with viscous heating at fW range
- In superfluid $^3$He the energy gap $\Delta$ according to the BCS theory is $\Delta_{BCS} = 1.75k_B T_c$, we measured $1.2k_B T_c$
Quartz tuning forks

- We consider replacing VWR’s by quartz tuning forks
- Several advantages
  - No need for magnetic field, independent of external magnetic fields
  - Simpler measurement configuration
  - Very high Q-values \((10^9)\), temperature independent intrinsic damping
  - High signal quality (one point measurement)
  - Cheap mass products
- Some questions/disadvantages
  - More complicated geometry
  - Higher frequency (acoustic losses)
  - Heating?
- We are still testing these
Cooling methods

- Helium-4 evaporation
- Helium-3 helium-4 dilution refrigerator
- Adiabatic nuclear demagnetization stage

Costs of cryoliquids:

- LN ~1 EUR / liter of liquid
- L⁴He ~ 20 EUR / liter of liquid
- L³He ~ 1000 EUR / liter of gas NTP
  ~750 000 EUR / liter of liquid!
Helium-4 evaporation

Pump $^4$He bath. As pressure goes down temperature follows saturated vapor pressure curve (can be understood easily by thinking that the most energetic atoms are pumped away -> cooling)

At about 1.3 K vapor pressure is so low that it is not possible to reduce pressure anymore by pumping, minimum temperature.
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The mixing of helium isotopes produces cooling, primarily due to zero point energy and quantum statistics of $^3$He.

- **Maximum cooling power** $Q_{\text{max}} = 84n_3T_{\text{mc}}^2$, (~10 µW at 10 mK)
- **Minimum temperature** about 4 mK ($T^2$ dependence)
- Very important and widely used cooling method
- **Closed $^3$He circulation**, rate ~1 mmol/s
- **Heat exchanger** is very important and the most complicated part

$^3$He- $^4$He dilution refrigerator

Diagram:
- From 1.5 K condenser to pump
- Almost pure $^3$He and $^4$He mix
- Heating and cooling processes
- Concentrated phase, dilute phase
- Secondary flow impedance
- Heat exchangers
- Still heat exchanger
- Heat flow
Adiabatic nuclear demagnetization

• At a given temperature the entropy of a paramagnetic spin system is lower the higher the magnetic field is.
• Nuclear spins remain paramagnetic down to very low temperatures and are thus suitable for adiabatic demagnetization cooling
• Entropy of the system must be removed at lowest possible temperature at highest possible magnetic field
• Our 100 mole copper nuclear stage can maintain temperature at around 0.1 mK for a period of few weeks after precooling at 9T field to about 10 mK temperature and demagnetization to about 10 mT field
The YKI cryostat

Liquid $^4$He bath (4.2 K)

$^4$He pot (1.2 K)

Still (0.7 K)

Heat exchangers

Mixing chamber (3 mK)

Adiabatic melting experiment cell (~300 μK)

Vacuum

9 T superconducting solenoid

Copper nuclear stage (<100 μK)

1 m
Measurements of ultralow temperatures

- Helium-4 bath
- Helium-4 evaporation
- Helium-4 Helium-3 Dilution
- Nuclear demagnetization

Summary

- ITS-90: H2O triple point, 273.16 K
- ITS-90: Hg triple point, 234.3156 K
- ITS-90: Ar triple point, 83.8058 K
- ITS-90: O2 triple point, 54.3584 K
- ITS-90: Ne triple point, 24.5561 K
- ITS-90: e-H2 vapour pressure, 17 K-20 K
- ITS-90: e-H2 triple point, 13.8033 K
- ITS-90: He vapour pressure, 3K-5K

- PLTS: 3He superfluid A phase transition (at melting curve), 2.44 mK
- PLTS: 3He superfluid A to B phase transition (at melting curve), 1.87 mK
- Néel transition, 0.902 mK

Platinum resistance t.
Carbon resistors
Germanium t.
Vibrating wire t.
Melting pressure t.
Co-60 nuclear orientation t.
Platinum NMR

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Adiabatic melting experiment

• Finding the superfluid transition of $^3$He diluted in superfluid $^4$He is one of the greatest questions still open in low temperature physics
  – Fermions in boson environment
  – $^3$He concentration 6.5 – 9% depending on pressure
• In previous experiments liquid was indirectly cooled by nuclear demagnetic refrigeration
  – Extremely large thermal resistance prevented cooling the liquid to temperatures below 100µK
• We have a new method capable of cooling the liquid directly
Adiabatic melting experiment

- The entropy of $^3$He drops exponentially below $T_c$
- The cooling power of dilution process decreases as $T^2$
- The heat leak to the cell sets the minimum temperature
Adiabatic melting cell

- Pressure is controlled via a superleak
- Pressure gauge thermally connected to mixing chamber
- Top flange: oscillators (VWRs of forks), possibly a NMR-probe
- Experimental volume 78 cm$^3$
- Reference volume 3 cm$^3$
Tentative results

- We have demonstrated that the new method works
- Several technical difficulties prevented using the full power of the method
- Now we are making modifications to the setup and thermometry

\[ Q = \frac{T_{\text{Wall}}^3(t) - T^3(t)}{3R_K}, \]

where \( R_K \) is the thermal resistance
EXTRA
Vibrating wire resonators

- Preliminary measurements at superfluid $^4$He and vacuum
Vibrating wire resonators

- Preliminary measurements at superfluid $^4$He and vacuum
Vibrating wire resonators

- Transition from dilute mixture to pure $^3$He at 12 mK.
Vibrating wire resonators

- Summary of measurements by upper VWR