CRYOGENICS 2

Going below 1 K

Welcome to the quantum world!

590B

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History

He3 systems

Properties of He-3 and He-4

Dilution refrigerator

Demagnetization refrigeration
Short history of temperatures below 1K

1908 LHe liquefaction by Kamerlingh Onnes
1926 The idea of demagnetization cooling by Debye
1927-31 Realization of demagnetization cooling, <100mK
1945 He-3 liquefaction
1950 The idea of Pomeranchuk refrigerator (cooling by adiabatic solidification of He-3)
1962 The idea of dilution process (Heinz London) with G. R. Clarke, E. Mendoza
1965-66 First dilution refrigerators build in Leiden, Dubna and Manchester reaching 25 mK
1971 Superfluidity of He-3
The kid of the 20th century, parallels space rocket and nuclear studies

Similar to space rockets Tsiolkovskiy train generation of low temperatures uses several stages
- 4K stage, usually referred to as He bath or main bath
- 1K stage, or 1K pot
- low temperature unit

The cooling power rapidly decreases with each stage, stages are activated in sequence, functioning of each stage impossible before full activation of the preceding

Important:
To reduce cryogen liquid consumption use to a maximum extent cooling power of the first stages
Cooling with cryogenic liquids

Useful range of cryogens

The lighter the better!
The lightest is stable He-3 isotope

Operation below 4.2 K completely relies on vacuum pumping
Pumping oil creates characteristic smell of low-temperature laboratories!
Natural He contains 0.000137% of He-3. Thousands of liters of He-3 are used annually in cryogenic applications.

He-3 is produced, not mined! Main source: nuclear fusion.

Earth gravity is not strong enough to keep He in atmosphere. Once released, He leaves to space.

Proposals as exotic as mining He-3 on Moon surface, where it accumulates due to exposure to Solar wind.
<table>
<thead>
<tr>
<th>Isotope</th>
<th>Atomic mass (ma/u)</th>
<th>Natural abundance (atom %)</th>
<th>Nuclear spin (I)</th>
<th>Magnetic moment (µ/µN)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^3$He</td>
<td>3.016 029 309 7(9)</td>
<td>0.000137 (3)</td>
<td>$^1/2$</td>
<td>-2.127624</td>
</tr>
<tr>
<td>$^4$He</td>
<td>4.002 603 2497(10)</td>
<td>99.999863 (3)</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

**Physical properties**

<table>
<thead>
<tr>
<th>Property</th>
<th>Helium-3</th>
<th>Helium-4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boiling (1atm)</td>
<td>3.19 K</td>
<td>4.23 K</td>
</tr>
<tr>
<td>Critical point</td>
<td>3.35 K</td>
<td>5.19 K.</td>
</tr>
<tr>
<td>Density of liquid (at boiling point, 1atm)</td>
<td>0.059 g/ml</td>
<td>0.12473 g/ml</td>
</tr>
<tr>
<td>Latent heat of vaporization</td>
<td>0.026 kJ/mol</td>
<td>0.0829 kJ/mol</td>
</tr>
</tbody>
</table>
Cooling power of evaporating cryogenic liquid

\[ Q = n \Delta H = nL, \]

- \( Q \) cooling power
- \( n \) rate of evaporation, molecules/time
- \( \Delta H \) enthalpy of evaporation
- \( L \) latent heat of evaporation

For a pump with constant volume rate \( V \)

\[ Q = VP(T)L \]

\( L \) approximately constant

Latent heat \( L \) [J/mole]

Temperature [K]
Cooling power proportional to vapor pressure
\[ Q \sim P(T) \sim \exp(-1/T) \]

Exponentially small at low T
We can get by pumping on
\[ ^4\text{He} \quad T \sim 1\text{K} \]
\[ ^3\text{He} \quad T \sim 0.26\text{ K} \]

Evaporative cooling is used in
1K pot
He-3 cryostat
He-3 refrigerator

Typical features:
- Sample in vacuum (rarely sample in He-3 liquid)
- One shot mode of operation
- Hold time 10-60 hours

Operation sequence:
- Release He-3 from cryopump
- Condense by heat exchange with 1K pot
- Cool condensate to 1.5K
- Start cryopumping to reach base temperature

He-3 is stored in a sealed space to avoid loss
He-3 pump is called Sorb, uses cryopumping
He-4 nucleus has no spin, Boson

No solid phase due to: weak van der Waals inter-atomic interactions, $E_{\text{pot}}$ is low
Large quantum mechanical zero-point energy $E_0 = \hbar^2/8ma^2$
due to small mass, $E_{\text{kin}}$ is high
Bose-Einstein condensate instead of a solid

Quantum liquids, ratio $\lambda = E_{\text{kin}}/E_{\text{pot}}$ He4 $\lambda = 2.64$ He3 $\lambda = 3.05$
Amplitude of vibrations about $1/3$ of interatomic space
He-3 nucleus has spin 1/2, Fermion

Additional spin entropy

Bose-Einstein condensate of pairs, several superfluid phases

Special feature: Below $T_F$ spins in the liquid phase are spatially indistinguishable. Therefore they start obeying Fermi statistics and are more ordered than in the paramagnetic solid phase!
Pomeranchuk cooling (1950)

Isentropic compression of He-3 below 200 mK leads to cooling

Pomeranchuk cooling was used to discover superfluid phases in He-3

Isaak Pomeranchuk
In dilution process of He-3 into He3-He-4 mixture
$\Delta H$ is enthalpy of mixing
$\Delta H = \int \Delta C dT$
Mixture of He3 and He-4

Phase separation of the mixture into He3 rich and He3 poor phases, but not pure He3 and He4

Pure quantum effect classical liquids should separate into pure components to obey 3rd law of thermodynamics, \( S=0 \)

In case of He3-He4 mixture, \( S=0 \) can be for finite concentration because of the Fermi statistics for He3 and Bose statistics for He4

Phase separation starts below \( T=0.867 \) K (max at \( X=0.675 \))
Cooling power:
Power law decrease
Instead of exponential decrease

Enthalpy of mixing uses the difference
In specific heat of two phases

\[ \Delta H = \int \Delta C dT \]
\[ Q \sim x \Delta H \sim T^2 \]
Evaporative cooling

Dilution

Still evaporates He3 from mixture

Mixing chamber

Phase separation line
Dilution Refrigerator: more details

- Condenser line
- Dilute phase
- Almost pure He3
- >90% He3 vapor
- Heater
- Still 0.7K
- Heat exchangers
- Heat flow
- Dilute phase
- Sintered
- Concentric
- 1K pot
- Main flow impedance
- Still heat exchanger
- To He3 pump
- Concentric sintered
- Mixing chamber 0.01K
- Dilute phase
- Phase boundary

Gravity

Temperature

Concentr. phase

Almost pure He3

>90% He3 vapor

Still 0.7K

Heater

Heat exchangers

Dilute phase

Sintered

Concentric

1K pot
Kapitza resistance

A discontinuity in temperature across the interface of two materials through which heat current is flowing.

Acoustic impedance mismatch at a boundary of two substances. Phonons have probability to be reflected.

Kapitza resistance, \( \sim T^3 \)

Important effect as \( T \) tends to 0

1K vs 10 mK

6 orders of magnitude change!

\[
\hat{Q} = \kappa_K \Delta T
\]

\( \kappa_K \) - Kapitza conductance

Good Thermal contact at low temperatures needs conduction electrons.
Dilution refrigerator: heat exchanging

Need big surface area contacts!

Concentric heat exchanger
High temperatures

Welded Cu-Ni foil
Sintered submicron silver powder
Close to mixing chamber
Dilution refrigerator: Experiment cooling

Do not rely on insulating contacts!

Vibration

RF heating
Dilution refrigerator: gas handling at room temperature

Key elements:
- He3-He4 Gas storage “Dump”
- Vacuum pump for 1K pot
- Vacuum pump for He3 circulation
- Roots (booster) pump for Still line pumping
- Cold traps for mixture cleaning

Very demanding to vacuum leaks
To avoid loss of mixture, all operation goes at $P<P_{\text{atm}}$
Leaks in, not out!
Dilution refrigerator: gas handling system

Front view

Back view

He3  He4
Peter Joseph William Debye

Adiabatic magnetization
Isomagnetic enthalpic transfer
Adiabatic demagnetization
Isomagnetic entropic transfer

Weakest point: needs heat switch working at 1K
Demagnetization fridge

"High"-temperature salts:

MAS: Mn\textsuperscript{2+} SO\textsubscript{4} \cdot (NH\textsubscript{4})\textsubscript{2} SO\textsubscript{4} \cdot 6H\textsubscript{2}O ; \quad T_c \approx 0.17 \text{ K}

FAA: Fe\textsubscript{7}\textsuperscript{3+} (SO\textsubscript{4})\textsubscript{3} \cdot (NH\textsubscript{4})\textsubscript{2} SO\textsubscript{4} \cdot 24H\textsubscript{2}O ; \quad T_c \approx 0.03 \text{ K}

"Low"-temperature salts:

CPA: Cr\textsubscript{2}\textsuperscript{3+} (SO\textsubscript{4})\textsubscript{3} \cdot K\textsubscript{2} SO\textsubscript{4} \cdot 24H\textsubscript{2}O ; \quad T_c \approx 0.01 \text{ K}

CMN: 2Ce\textsuperscript{3+} (NO\textsubscript{3})\textsubscript{3} \cdot 3Mg(NO\textsubscript{3})\textsubscript{2} \cdot 24H\textsubscript{2}O ; \quad T_c \approx 0.002 \text{ K}.

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Fig. 9.2: Curves S (divided by the gas constant $R$) of four salts suitable for paramagnetic demagnetization as a function of temperature in zero field (solid lines) and in 2 T (dashed lines). (For the chemical formula of the salts see the text)
Experimental realization, $T < 100 \text{ mK}$
Experimental verification of 3rd law of thermodynamics, $S \to 0$ when $T \to 0$

William F. Giauque

Commercially available from Janis, CMR, Dryogenics
~60mK
Short experiment time, residual magnetic fields
Relatively inexpensive
Flesh may freeze and stick to cold surfaces

Ref. [6]

DON’T PANIC
<table>
<thead>
<tr>
<th>Safety</th>
<th>Known cases in my experience</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Kyoto: 15T magnet on energization attracted rotary pump, which destroyed vacuum Dewar</td>
</tr>
<tr>
<td></td>
<td>Loss of the magnet</td>
</tr>
<tr>
<td></td>
<td>Kyoto: due to an air leak to cryogenic center</td>
</tr>
<tr>
<td></td>
<td>Liquefier was damaged, 0.5 mln $ loss</td>
</tr>
<tr>
<td></td>
<td>Sherbrooke: quench due to He</td>
</tr>
<tr>
<td></td>
<td>magnet bath exhaust damaged magnet</td>
</tr>
<tr>
<td></td>
<td>Critical field reduced from 15T to 0.7T</td>
</tr>
<tr>
<td></td>
<td>Sherbrooke: due to a leak, dipper</td>
</tr>
<tr>
<td></td>
<td>accumulated He liquid inside and exploded</td>
</tr>
<tr>
<td></td>
<td>on warming, fortunately no one was injured</td>
</tr>
<tr>
<td></td>
<td>Cornell: closed 1K pot with He liquid</td>
</tr>
<tr>
<td></td>
<td>inside and relieve valve frozen, destroyed DF</td>
</tr>
<tr>
<td></td>
<td>Bristol: on a day like today, student opened OVC</td>
</tr>
<tr>
<td></td>
<td>vacuum valve</td>
</tr>
<tr>
<td></td>
<td>1 million worth of equipment</td>
</tr>
<tr>
<td><strong>Temperature</strong></td>
<td></td>
</tr>
<tr>
<td><strong>Gas Pressure</strong></td>
<td></td>
</tr>
<tr>
<td><strong>Energy (SC magnets)</strong></td>
<td></td>
</tr>
<tr>
<td><strong>Damage to expensive equipment</strong></td>
<td></td>
</tr>
<tr>
<td><strong>Loss of mixture</strong></td>
<td></td>
</tr>
</tbody>
</table>
Reading materials

*Matter and methods at low temperatures*
Author: Frank Pobell; Springer, 2007

*Experimental techniques in low-temperature physics*
Author: Guy K. White; Clarendon Press, 1979

*Experimental low-temperature physics*
Author: Anthony Kent; American Institute of Physics, 1993

*Experimental techniques in condensed matter physics at low temperatures*
Author: Robert C Richardson; Eric N Smith Addison-Wesley Pub. Co., 1988

*Experimental techniques for low-temperature measurements: cryostat design, material properties, and superconductor critical-current testing*
Author: J. W. Ekin; Oxford University Press, 2006

*Hitchhiker’s guide to dilution refrigerators,*
Nathaniel Creig and Ted Lester