CRYOGENICS

PHY 590B F18

Sergey L. Bud’ko

History
Materials
Design
Cryogenics
The branches of physics and engineering that involve the study of very low temperatures, how to produce them, and how materials behave at those temperatures.

Cryonics
The emerging medical technology of cryopreserving humans and animals with the intention of future revival.
## Some typical low temperatures

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Celsius (°C)</th>
<th>Absolute (K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tropics</td>
<td>45</td>
<td>318</td>
</tr>
<tr>
<td>Human body</td>
<td>37</td>
<td>310</td>
</tr>
<tr>
<td>Room temperature</td>
<td>20</td>
<td>293</td>
</tr>
<tr>
<td>Ice point</td>
<td>0</td>
<td>273</td>
</tr>
<tr>
<td>Salt + water (cryogen)</td>
<td>-18</td>
<td>255</td>
</tr>
<tr>
<td>Antarctic winter</td>
<td>-50</td>
<td>223</td>
</tr>
<tr>
<td>Solid carbon dioxide</td>
<td>-78</td>
<td>195</td>
</tr>
<tr>
<td>Liquid oxygen</td>
<td>-183</td>
<td>90</td>
</tr>
<tr>
<td>Liquid nitrogen</td>
<td>-196</td>
<td>77</td>
</tr>
<tr>
<td>Liquid helium</td>
<td>-269</td>
<td>4</td>
</tr>
<tr>
<td>Absolute zero</td>
<td>-273</td>
<td>0</td>
</tr>
</tbody>
</table>

Iowa record: +48°C
Iowa record: -44°C
Logarithmic temperature scale

- Lowest Temperatures Recorded
  - $^3$He Superfluid $T_c$
  - $^4$He Superfluid $T_c$
  - BOILING POINTS
    - $^4$He
    - N$_2$
    - H$_2$O
    - W

- Cryogenics
- High Temp.
- Plasmas
- Fusion
- High Energy Particles

Temperature Scale:
- $10^{-8}$ to $10^{12}$ K
cryo-surgery and veterinary medicine

cryo-transport of natural gas

100 t LH$_2$ + 600 t LO$_2$

research - physics

medicine

space

life
Historical cryogenics events

1850 Mechanical refrigeration first applied
1877 Cailetet & Pictet liquefied O₂ (90 K).
1892 James Dewar developed the vacuum flask
1908 Kamerlingh Onnes liquefied He (4.2 K).
1911 K. O. discovered superconductivity.
1926 Giauque reached 0.25 K by magnetic cooling.
1946 Collins developed commercial He liquefier.
1995 Connell & Wiemann achieved Bose-Einstein condensation at 2x10⁻⁸ K.
## Characteristic temperatures of low-energy phenomena

<table>
<thead>
<tr>
<th>Phenomenon</th>
<th>Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>Debye temperature of metals</td>
<td>few 100 K</td>
</tr>
<tr>
<td>High-temperature superconductors</td>
<td>~ 100 K</td>
</tr>
<tr>
<td>Low-temperature superconductors</td>
<td>~ 10 K</td>
</tr>
<tr>
<td>Intrinsic transport properties of metals</td>
<td>&lt; 10 K</td>
</tr>
<tr>
<td>Cryopumping</td>
<td>few K</td>
</tr>
<tr>
<td>Cosmic microwave background</td>
<td>2.7 K</td>
</tr>
<tr>
<td>Superfluid helium 4</td>
<td>2.2 K</td>
</tr>
<tr>
<td>Bolometers for cosmic radiation</td>
<td>&lt; 1 K</td>
</tr>
<tr>
<td>Low-density atomic Bose-Einstein condensates</td>
<td>~ µK</td>
</tr>
</tbody>
</table>
Materials change at low temperatures

Structural phase transition (brittle-ductile – Ti alloys)

CW paramagnetism, long range magnetic order, spin glass

Superconductivity (annoying traces of it – phosphor bronze, Ti-alloys, many solders)

Freezing/glassing (liquids, oils, greases)

Condensation/liquefaction (gases)

Change of mechanical properties (rubber, plastics)

see appendix
Cooling with cryogenic liquids

Useful range of cryogens

- Helium
- Hydrogen
- Neon
- Nitrogen
- Argon
- Oxygen

T [K]

- Below Patm
- Above Patm
Cooling with cryogenic liquids

Vapour pressure at cryogenic temperatures

1 atm
# Normal boiling point and latent heat of fluids

<table>
<thead>
<tr>
<th>Fluid</th>
<th>Normal Boiling Point (K)</th>
<th>Latent Heat (kJ/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>373</td>
<td>2256</td>
</tr>
<tr>
<td>Ethylene</td>
<td>169</td>
<td>481</td>
</tr>
<tr>
<td>Krypton</td>
<td>120</td>
<td>116</td>
</tr>
<tr>
<td>Methane</td>
<td>111</td>
<td>512</td>
</tr>
<tr>
<td>Xenon</td>
<td>110</td>
<td>99</td>
</tr>
<tr>
<td>Oxygen</td>
<td>90</td>
<td>213</td>
</tr>
<tr>
<td>Argon</td>
<td>87</td>
<td>162</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>77</td>
<td>199</td>
</tr>
<tr>
<td>Neon</td>
<td>27</td>
<td>86</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>20</td>
<td>443</td>
</tr>
<tr>
<td>Helium</td>
<td>4.2</td>
<td>21</td>
</tr>
</tbody>
</table>

## Cooling with cryogenic liquids
# Cooling with cryogenic liquids

Amount of cryoliquids (in liters) to cool 1 kg of metal if only latent heat (latent heat + enthalpy of the gas) is used.

<table>
<thead>
<tr>
<th>Cryoliquid</th>
<th>Temperature change (K)</th>
<th>Al</th>
<th>SS</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\text{N}_2$</td>
<td>300 - 77</td>
<td>1.0 (0.63)</td>
<td>0.53 (0.33)</td>
<td>0.46 (0.28)</td>
</tr>
<tr>
<td>$^4\text{He}$</td>
<td>77 – 4.2</td>
<td>3.2 (0.2)</td>
<td>1.4 (0.1)</td>
<td>2.2 (0.16)</td>
</tr>
<tr>
<td>$^4\text{He}$</td>
<td>300 – 4.2</td>
<td>66 (1.6)</td>
<td>34 (0.8)</td>
<td>32 (0.8)</td>
</tr>
</tbody>
</table>

- Use LN$_2$ to precool the equipment (caution and patience needed)
- Make use of the enthalpy of the cold He gas (very slow initial transfer, end of the transfer tube close to the bottom)
Heat sources

(i) Heat conduction

Thermal conductivity integrals of selected materials [W/m]

<table>
<thead>
<tr>
<th></th>
<th>From vanishingly low temperature up to</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>20 K</td>
</tr>
<tr>
<td>OFHC copper</td>
<td>11000</td>
</tr>
<tr>
<td>DHP copper</td>
<td>395</td>
</tr>
<tr>
<td>1100 aluminium</td>
<td>2740</td>
</tr>
<tr>
<td>2024 aluminium alloy</td>
<td>160</td>
</tr>
<tr>
<td>AISI 304 stainless steel</td>
<td>16.3</td>
</tr>
<tr>
<td>G-10 glass-epoxy composite</td>
<td>2</td>
</tr>
</tbody>
</table>

Heat conduction in solids

Fourier’s law: $Q_{\text{con}} = k(T) \cdot S \cdot \frac{dT}{dx}$

$k(T)$: thermal conductivity [W/m.K]

Integral form:

$$Q_{\text{con}} = \frac{S}{L} \int_{T_1}^{T_2} k(T) \cdot dT$$

$\int k(T) \cdot dT$: thermal conductivity integral [W/m]

Thermal conductivity integrals for standard construction materials are tabulated

stainless steel
thin walls
G10
Heat sources

(ii) Heat radiation

- Wien’s law
  - Maximum of black body power spectrum
  \[ \lambda_{\text{max}} T = 2898 \, \text{[\mu m.K]} \]
- Stefan-Boltzmann’s law
  - Black body
  - “Gray”body
  - “Gray” surfaces at \( T_1 \) and \( T_2 \)

\[
Q_{\text{rad}} = \varepsilon \sigma A T^4
\]
\[
\varepsilon = 5.67 \times 10^{-8} \, \text{W/m}^2\text{.K}^4
\]
(Stefan Boltzmann’s constant)

\[ Q_{\text{rad}} = \varepsilon \sigma A T^4 \]
\( \varepsilon \) emissivity of surface

\[ Q_{\text{rad}} = E \varepsilon A (T_1^4 - T_2^4) \]
\( E \) function of \( \varepsilon_1, \varepsilon_2 \) geometry

Emissivity of technical materials at low temperatures

<table>
<thead>
<tr>
<th></th>
<th>Radiation from 290 K Surface at 77 K</th>
<th>Radiation from 77 K Surface at 4.2 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stainless steel, as found</td>
<td>0.34</td>
<td>0.12</td>
</tr>
<tr>
<td>Stainless steel, mech. polished</td>
<td>0.12</td>
<td>0.07</td>
</tr>
<tr>
<td>Stainless steel, electropolished</td>
<td>0.10</td>
<td>0.07</td>
</tr>
<tr>
<td>Stainless steel + Al foil</td>
<td>0.05</td>
<td>0.01</td>
</tr>
<tr>
<td>Aluminium, as found</td>
<td>0.12</td>
<td>0.07</td>
</tr>
<tr>
<td>Aluminium, mech. polished</td>
<td>0.10</td>
<td>0.06</td>
</tr>
<tr>
<td>Aluminium, electropolished</td>
<td>0.08</td>
<td>0.04</td>
</tr>
<tr>
<td>Copper, as found</td>
<td>0.12</td>
<td>0.06</td>
</tr>
<tr>
<td>Copper, mech. Polished</td>
<td>0.06</td>
<td>0.02</td>
</tr>
</tbody>
</table>

polished surfaces radiation shields
Heat sources

(iii) Residual gas conduction

getters on cold walls

![Residual gas conduction](image)

- **Molecular regime**
  - At low gas pressure $\lambda_{molecule} >> d$
  - Kennard’s law
  - Conduction heat transfer proportional to pressure, independent of spacing between surfaces
    - $\Omega$ depends on gas species
  - Accommodation coefficient $\alpha(T)$ depends on gas species, $T_1, T_2$

(iv) Thermoacoustic (Taconis) oscillations

standing waves in gas filled tubes with temperature gradient and closed warm end

change in geometry

introduction of damping element

use of Taconis oscillations
Superinsulation

Multi-layer insulation (MLI)

- Complex system involving three heat transfer processes
  - \( Q_{MLI} = Q_{rad} + Q_{sol} + Q_{res} \)
  - With \( n \) reflective layers of equal emissivity, \( Q_{rad} \sim 1/(n+1) \)
  - Due to parasitic contacts between layers, \( Q_{sol} \) increases with layer density
  - \( Q_{res} \) due to residual gas trapped between layers, scales as \( 1/n \) in molecular regime
**Typical heat fluxes at vanishingly low temperature between flat plates [W/m²]**

<table>
<thead>
<tr>
<th>Description</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Black-body radiation from 290 K</td>
<td>401</td>
</tr>
<tr>
<td>Black-body radiation from 80 K</td>
<td>2.3</td>
</tr>
<tr>
<td>Gas conduction (100 mPa He) from 290 K</td>
<td>19</td>
</tr>
<tr>
<td>Gas conduction (1 mPa He) from 290 K</td>
<td>0.19</td>
</tr>
<tr>
<td>Gas conduction (100 mPa He) from 80 K</td>
<td>6.8</td>
</tr>
<tr>
<td>Gas conduction (1 mPa He) from 80 K</td>
<td>0.07</td>
</tr>
<tr>
<td>MLI (30 layers) from 290 K, pressure below 1 mPa</td>
<td>1-1.5</td>
</tr>
<tr>
<td>MLI (10 layers) from 80 K, pressure below 1 mPa</td>
<td>0.05</td>
</tr>
<tr>
<td>MLI (10 layers) from 80 K, pressure 100 mPa</td>
<td>1-2</td>
</tr>
</tbody>
</table>
Dewar flask
Sir James Dewar, FRS (1842-1923)

Developed *Cordite* (smokeless gunpowder alternative) – with Sir Frederick Abel, 1889

Described several formulae for benzene, 1867

Studied physiological action of light, with Prof. J. G. McKendrick (conditions of retina)

Spectroscopical observations, with Prof. G.D. Living

Public liquefaction of oxygen and air, 1884

Cir. 1892 – vacuum flask

Liquid hydrogen, 1898, solid hydrogen, 1899

Gas-absorbing powers of charcoal, 1905

Surface tension of soap bubbles, during and after WW1

Last publication in 1923 (at 80 yrs age)

Was able to engage in quite vitriolic arguments with other scientists: “...argue with Dewar was akin to being a fly in molasses...”

Lost race for He liquefaction

Lost patent case against *Thermos* in court

Was nominated many times but did not get Nobel prize

Refused to retire from Fullerian Professorship in Chemistry at the Royal Institution
Glass cryostats – more details

how would you do this?

LN$_2$ dewar – no valve, LHe dewar - valve: why?
“silver mirror reaction” or Tollens’ test

\[
\text{CH}_3\text{C} = \text{H} + 2\text{Ag}^{+} + 2\text{OH}^{-} \rightarrow \text{CH}_3\text{C}\text{OH} + 2\text{Ag} + \text{H}_2\text{O}
\]

qualitative laboratory test used to distinguish between an aldehyde and a ketone

you can try to see demonstration at one of the ISU’s GenChem or Organic Chemistry courses
Glass research cryostats

Basically extinct by now

At a time:
- Were cheap
- Could be fabricated and repaired in a good glass shop
- Offer visual control of cryogens
- Small amount of cryogens

However
- Not particularly safe (break in multiple glass pieces flying around – unless you use nylon stockings)
- Need to take particular care of overpressure safety features
- Not recommended to leave unattended
# Storage Dewars – LN$_2$

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>V-1</td>
<td>Liquid Fill/Withdrawal</td>
</tr>
<tr>
<td>V-2</td>
<td>Fill/Withdrawal Valve</td>
</tr>
<tr>
<td>V-3</td>
<td>Pressure Building</td>
</tr>
<tr>
<td>V-4</td>
<td>Vent Valve</td>
</tr>
<tr>
<td>RD-1</td>
<td>Rupture Disk</td>
</tr>
<tr>
<td>RV-1</td>
<td>Relief Device</td>
</tr>
<tr>
<td>Pg-1</td>
<td>Pressure Guage</td>
</tr>
<tr>
<td>LG-1</td>
<td>Liquid Level Building</td>
</tr>
<tr>
<td>PCV-1</td>
<td>Pressure Building Regulator</td>
</tr>
<tr>
<td>PBC-1</td>
<td>Pressure Building Coil</td>
</tr>
</tbody>
</table>

---

**CL SERIES**

**CLPB SERIES**

---

**Liquid Level Line**
Storage dewars - LHe

Fig. 5.7. Commercial storage vessel for liquid $^4$He (A: connection for transfer tube, B: overflow valve, C: safety valve, D: manometer, E: vacuum and safety valves, F: gas valve, G: getter material, H: adsorbent material, I: superinsulation)
Dipper

Fast, cheap (and dirty)

Can use storage dewar

Poor temperature control (easy to improve somewhat)
Continuous flow cryostat

- **Cheap**
- **Fast cooling**
- **Relatively low He consumption**
- **Relatively easy optical access**
- **Base temperature?**
- **Mediocre temperature control**
- **Hard to incorporate high field**
Going below 4.2 K – $^4$He

Usually not done like this nowadays.

Still might be considered for objects with large thermal mass or if experiment generates a lot of heat.
**4He cryostat with variable temperature insert**

- Workhorse for LT measurements
- Good temperature control
- Easy magnet accommodation
- Allows for variety of sample holders
- Not so cheap
- Uses fair amount of cryogens
- Needle valves and small impedances could be tricky
Helium transfer

He level detectors: acoustic, resistive (SC), capacitive.

Diagram:
- To helium recovery system
- Measurement probe
- Cold helium gas cools apparatus
- Superconductor magnet
- Test dewar
- Pressure relief valve
- Two-section coupling
- Flexible bellows transfer tube
- Transfer tube fill port
- Transfer tube funnel tube during initial cooldown
- Helium delivered to bottom of test dewar during initial cooldown
- Liquid helium
- Liquid-helium storage dewar
How to get there? Closed cycle refrigerators...

Gifford-McMahon (GM) or Pulse Tube (PT) cycle

To make a refrigeration cycle, need a substance, the entropy of which depends on some other variable than temperature

**Pressure of gas:** Compression/expansion cycle

**Magnetization of solid:** magnetic refr. cycle

\[ \Delta Q_1: \text{heat absorbed at } T_1 \]
\[ \Delta Q_2: \text{heat rejected at } T_2 \]

→ Refrigeration cycle A B C D
Closed cycle refrigerators GM vs PT

1) Price
In general, GM cryocoolers are less expensive than PT cryocoolers of similar cooling power.

2) Vibration
Both GM and PT cryocoolers are mechanical refrigerators that do have some level of vibration. Two locations exist on the cold head where vibration is important: The room temperature mounting flange and the 2nd stage.

3) Orientation
The performance of PT cryocoolers is orientation-dependent. PT cryocoolers only function properly when they are operated in a purely vertical orientation with the 2nd stage pointing down. GM cryocoolers will lose some cooling power when the cold head is not operated vertically, but the base temperature will not be affected. GM cryocoolers can operate in any orientation.
Closed cycle refrigerators

GOOD

Turn-key
Easy optical (etc...) access
No liquid cryogens
Small footprint (but remember compressor)
Sometimes reasonably priced

NOT SO GOOD

“Usually” base $T > 4 \, K$
Noise, vibrations
“Small” cooling power at low temperatures
Long term maintenance cost?
Use in high fields?

Somewhat difficult to tweak
Cryogen free systems

One can buy

14T/100mK measurements platform (combination of PT cryocooler and magnetic refrigeration stage)

SQUID magnetometer with installed liquefaction unit.

Presented as example only. We are not endorsing any particular manufacturer.
Cryogen free systems

One can buy

Presented as example only. We are not endorsing any particular manufacturer.
Safety

Temperature
Pressure
Energy (SC magnets)

Ice blocks
Relief valves
He level – magnet quench
Glass dewars
Liquid oxygen

Think
Learn
Use PPE (!?)

RECOVER!

Take Ames Laboratory “Safe use of cryogens” training
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
Electrical resistivity and heat capacity

Figure 2.1 Specific heat as a function of temperature for several types of material. Typical behaviors are illustrated for metals (aluminum, beryllium, and copper), semiconductors (carbon and silicon), an amorphous inorganic (Pyrex glass) (Corruccini and Gniezek, 1960), and for an organic polymer (polyvinyl chloride) (Chang, 1977). A single point is shown for aluminum in the superconducting state at 1 K.
Yield strength of engineering materials

(1) 2024-T4 Aluminum
(2) Beryllium copper
(3) K-monel
(4) Titanium
(5) 304 Stainless steel
(6) C1020 Carbon steel
(7) 9% Nickel alloy steel
(8) Teflon
(9) Invar-36
Variation of Young's modulus with temperature

Fig. 10.1 Variation of Young's modulus with temperature. (From Ledbetter [10.1].)
A: silver 99.999% pure,
B: copper (OFHC),
C: coalesced copper,
D: electrolytic tough pitch,
E: single crystal aluminum,
F: machining copper,
G: aluminum 1100 F,
H: aluminum 6063-T5,
I: phosphorus deoxidized copper,
J: aluminum 2024-T4,
K: brass
A; 50-50 Pb-Sn solder
B; steel, SAE 1020
C; beryllium copper
D; constantan
E; monel
F; silicon bronze
G; inconnel
H; 347 stainless steel
I; fused quartz
J; teflon (PTFE)
K; polymethylmethacrylate (PMMA)
L; nylon

appendix
Thermal contraction

appendix