

# PHYS-E055102

Low Temperature Physics V D:  
Low Temp Techniques, Course  
2022, 6.9.-7.12.

The students will learn how to construct a successful experiment using low-temperature techniques.

First lecture:

- Introduction
- Short history of cryogenics
- Some phase diagrams
- Cryogenic fluids
- Safety measures



# Three courses under PHYS-E0551

- This course: Low Temperature Physics
  - Lectures on Tuesdays 14 – 16 by Pertti Hakonen (Nanotalo 161)
  - Exercises on XXX by Marco Will (place to be selected)
- Contact: [firstname.lastname@aalto.fi](mailto:firstname.lastname@aalto.fi), or room N175
- Nanoelectronics (Sorin Paraoanu)
- Theory of Superconductivity (Vladimir Eltsov)

# Course material

- F. Pobell, *Matter and Methods at Low Temperatures* (Springer-Verlag 1992, 1996, 2007)
- Lecture notes (will be available at MyCourses)
- Exercises (available as above)

Additional useful reading:

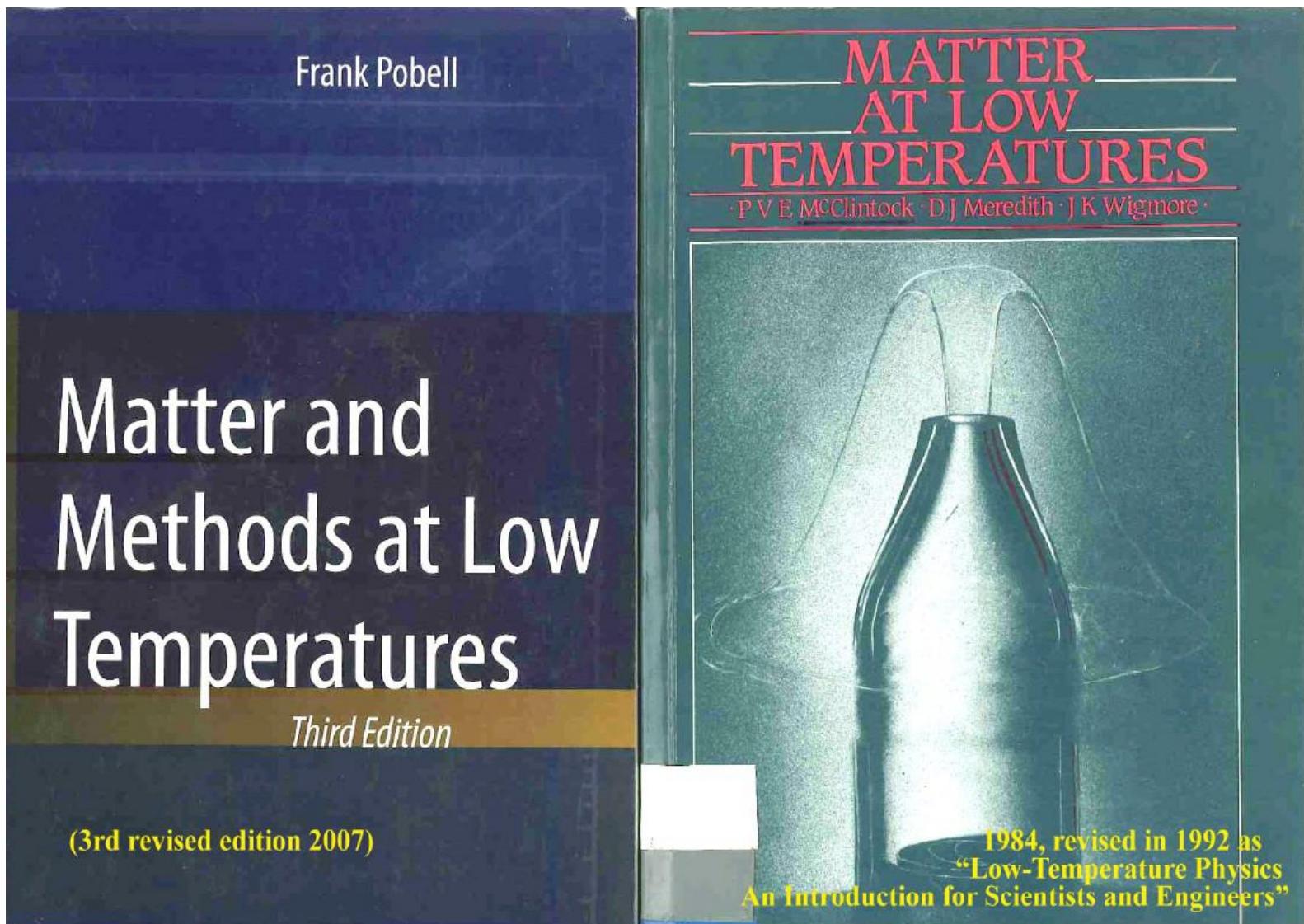
- P.V.E. McClintock, D.J. Meredith, and J.K. Wigmore  
*Matter at Low Temperatures* (1984) / *Low-Temperature Physics An Introduction for Scientists and Engineers* (1992)
- C. Enss and S. Hunklinger, *Low-Temperature Physics* (2005)

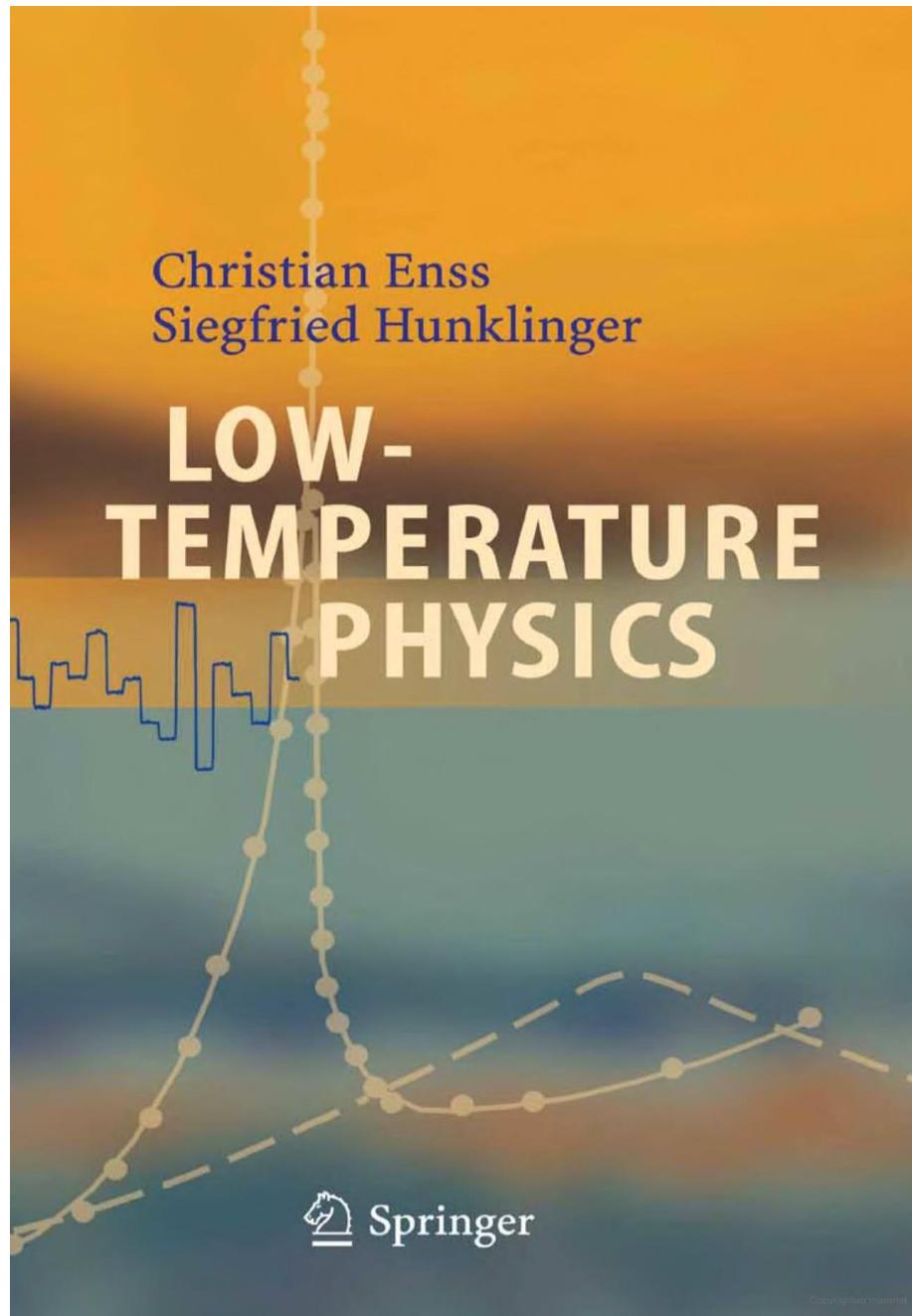
For an enthusiastic reader:

- O.V. Lounasmaa, *Experimental Principles and Methods below 1 K* (1974, <http://ltl.tkk.fi/wiki/images/6/61/Book.pdf>)

For basic things:

- G.K. White, *Experimental Techniques in Low-Temperature Physics*, 3rd Edition (1979)





#### Quantum Fluids

- Helium — General Properties, pp 3-13
- Superfluid  $^4\text{He}$  — Helium II, pp 15-76
- Normal-Fluid  $^3\text{He}$ , pp 77-96
- Superfluid  $^3\text{He}$ , pp 97-146
- Mixtures of  $^3\text{He}$  and  $^4\text{He}$ , pp 147-163

#### Solids at Low Temperatures

- Phonons, pp 167-203
- Conduction Electrons, pp 205-243
- Magnetic Moments — Spins, pp 245-282
- Tunneling Systems, pp 283-341
- Superconductivity, pp 343-446

#### Principles of Refrigeration and Thermometry

- Cooling Techniques, pp 449-504
- Thermometry, pp 505-541

# Objective and Prerequisites

The course is aimed for experimental physicists needing low temperature environment in their study. The objective is to learn

- operating modern refrigerators
- to design working cryostats
- preparing successful experimental setups for work at millikelvin temperatures and below

(also for the theoretically inclined it is good to understand the practical limitations in refrigeration and in methods applicable at very low temperatures)

To follow, you must have basic understanding of

- thermodynamics (entropy, chemical potential, distribution functions, ...)
- quantum mechanics (quantization, wave function, spin, symmetries, ...)
- condensed matter physics (phonons, conduction electrons, magnetism, ...)

# Topics

- Specific heat (insulators, metals, superconductors, helium fluids, ...)
- Thermal conductivity (as above)
- Thermal contact, - isolation, - switching, - expansion  
(technical aspects, sealing, leak detection, pumps)
- Refrigeration:
  - Evaporation cooling (helium bath, refillable pot, pumping capacity)
  - Pomeranchuk cooling (entropy considerations, examples)
  - Dilution cooling (heat of mixing, solubility, osmotic pressure, heat exchangers, etc)
  - Pulse tube refrigerators (dry cryostats)
  - Adiabatic demagnetization (paramagnetic salts, nuclear spins, hyperfine enhanced systems)
- Thermometry:
  - Resistivity (metals, semiconductors)
  - Magnetic susceptibility (paramagnetic salts, spin glasses)
  - Nuclear orientation (gamma emission, NMR, neutron absorption)
  - Noise thermometry
  - Viscometry on helium fluids (vibrating wires and other mechanical oscillators)
  - Melting curves ( ${}^3\text{He}$ , helium mixtures)
  - Superconducting fixed points
  - Temperature scales
- Electromagnetic interference?
- Electronic coolers?

# (Very) short history

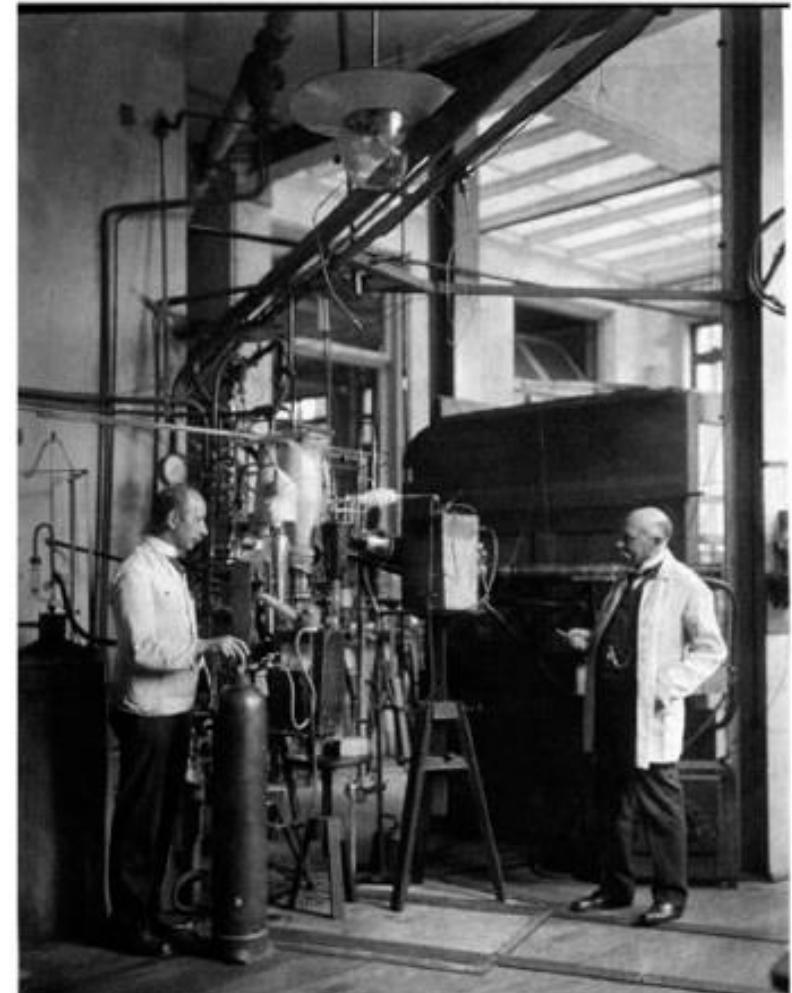
Greek  $\kappa\rho\hat{\nu}\sigma$  (cold)  
 $\gamma\varepsilon\nu\epsilon\varsigma$  (generated from) } cryogenics

Early history: development of concepts, physical relations, and measurements

- around 1700; defining temperature and units, practical thermometers, concept of heat in terms of energy, concept of absolute zero temperature
- around 1800; gas laws, techniques for reducing temperature moderately below the ambient, production of artificial ice

## First era: liquefaction of "permanent gases"

- 1877 Cailletet and Pictet  
(independently from one another)  
liquefy oxygen (90.2 K)
- 1883 Wróblewski and Olszewski  
liquefy nitrogen (77.4 K)
- 1898 Dewar liquefies hydrogen (20.3 K)  
(had invented the vacuum isolated dewar flask in 1892)
- 1908 Kamerlingh Onnes  
liquefies helium (4.2 K)  
(helium had been discovered in 1868 from sun, available on earth since 1903 from gas wells)



Flim & Kamerlingh Onnes in Leiden around 1910 at their helium liquefier

## Modern era: after access to liquid helium

- 1911 Kamerlingh Onnes discovers superconductivity (Hg,  $T_c = 4.2$  K)
- 1930's Giauque and others, adiabatic demagnetization cooling (paramagnetic salts, 0.25 K)
- 1937 Kapitza, Allen, and Misener realize superfluidity of  $^4\text{He}$  at 2.2 K
- 1956 Kurti uses nuclear demagnetization on copper (nuclear spin temperature of  $\sim 20$   $\mu\text{K}$ )
- 1963 Gifford and Longsworth develop the pulse tube cooler
- 1965 First dilution refrigerators
  - (principle introduced in 1951 by London, practical realization inhibited by no availability of  $^3\text{He}$ )
- 1972 Lee, Richardson, and Osheroff discover superfluidity in  $^3\text{He}$  at 0.002 K
- 1978 Wineland and Dehmelt demonstrate laser cooling
- 1986 Müller and Bednorz discover high-temperature superconductivity ( $\text{LaBaCuO}$ ,  $T_c = 30$  K)
- 1995 Cornell and Wieman create Bose-Einstein condensate (magneto-optically trapped  $^{87}\text{Rb}$  at 170 nK)

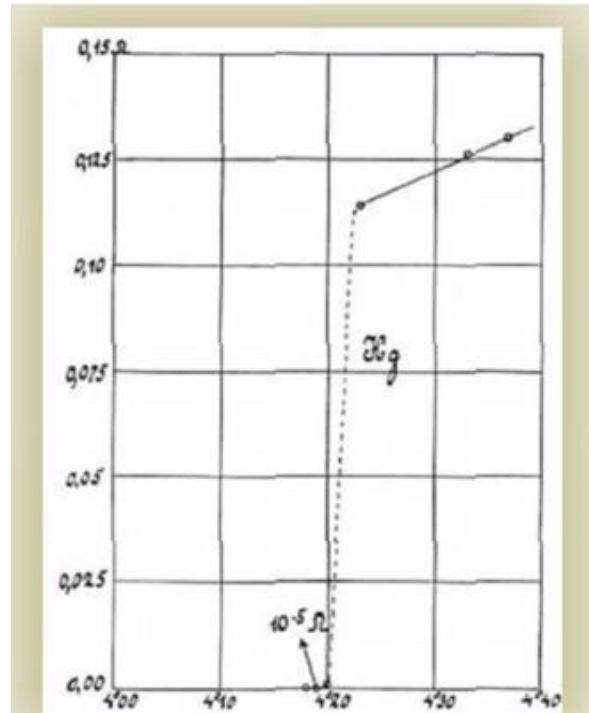


Figure 4. Historic plot of resistance (ohms) versus temperature (kelvin) for mercury from the 26 October 1911 experiment shows the superconducting transition at 4.20 K. Within 0.01 K, the resistance jumps from unmeasurably small (less than  $10^{-5}$   $\Omega$ ) to 0.1  $\Omega$ . (From ref. 9.)

alkali  
metals

# PERIODIC TABLE OF SUPERCONDUCTING ELEMENTS

18

Atomic number  
Symbol /  
Critical temperature of bulk at normal pressure  
Critical temperature under certain conditions  
Condition type (e.g. pressure value, film form)

superconducting element only under certain conditions (pressure or film form)

superconducting element at normal pressure in bulk form

	1 H	alkaline earth metals
	2 Li	
0.4 mK	26 mK	
20 K	9.95 K	film

11 Na	12 Mg
-------	-------

19 K	20 Ca
29 K	216 GPa

37 Rb	38 Sr
4 K	50 GPa

55 Cs	56 Ba
1.66 K	5 K

87 Fr	88 Ra

89 Ac
1.75 K 5 GPa

58 Ce	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb	71 Lu
1.75 K 5 GPa					1.8 K 80 GPa							0.1 K 1.2 K 18 GPa	
90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr
1.4 K	1.4 K	1.3 K 2.2 K 1 GPa			1 K								

21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr		
0.34 K 21 GPa	0.5 K 120 GPa	5.4 K 17.2 K 3 K film		2 K 21 GPa					0.85 K 1.6 K film	1.08 K 8.6 K film	5.4 K 11.5 GPa	2.7 K 24 GPa	7 K 13 GPa	1.4 K 150 GPa			
37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I 54 Xe	
4 K 50 GPa	2.8 K 15 GPa	0.6 K 11 K 9.7 K 30 GPa 4.5 GPa	9.25 K 0.92 K	0.92 K 8.2 K	0.5 K 0.5 mK	0.5 K irradiated				0.52 K		3.4 K 4.2 K film	3.7 K 4.7 K film	3.6 K 8.5 GPa	7.4 K 35 GPa	1.2 K 25 GPa	
55 Cs	56 Ba	57 La	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi	84 Po	85 At 86 Rn	
1.66 K 8 GPa	5 K 20 GPa	6 K 12.8 K 20 GPa	0.38 K 4.4 K 4.5 K 40 GPa	0.01 K 4.5 K 5.5 K film	1.7 K 0.7 K 0.1 K				20 mK powder		4.15 K	2.4 K 7.2 K	0.5 mK 8.7 K 9 GPa				

LaH<sub>10</sub>: Tc=250K

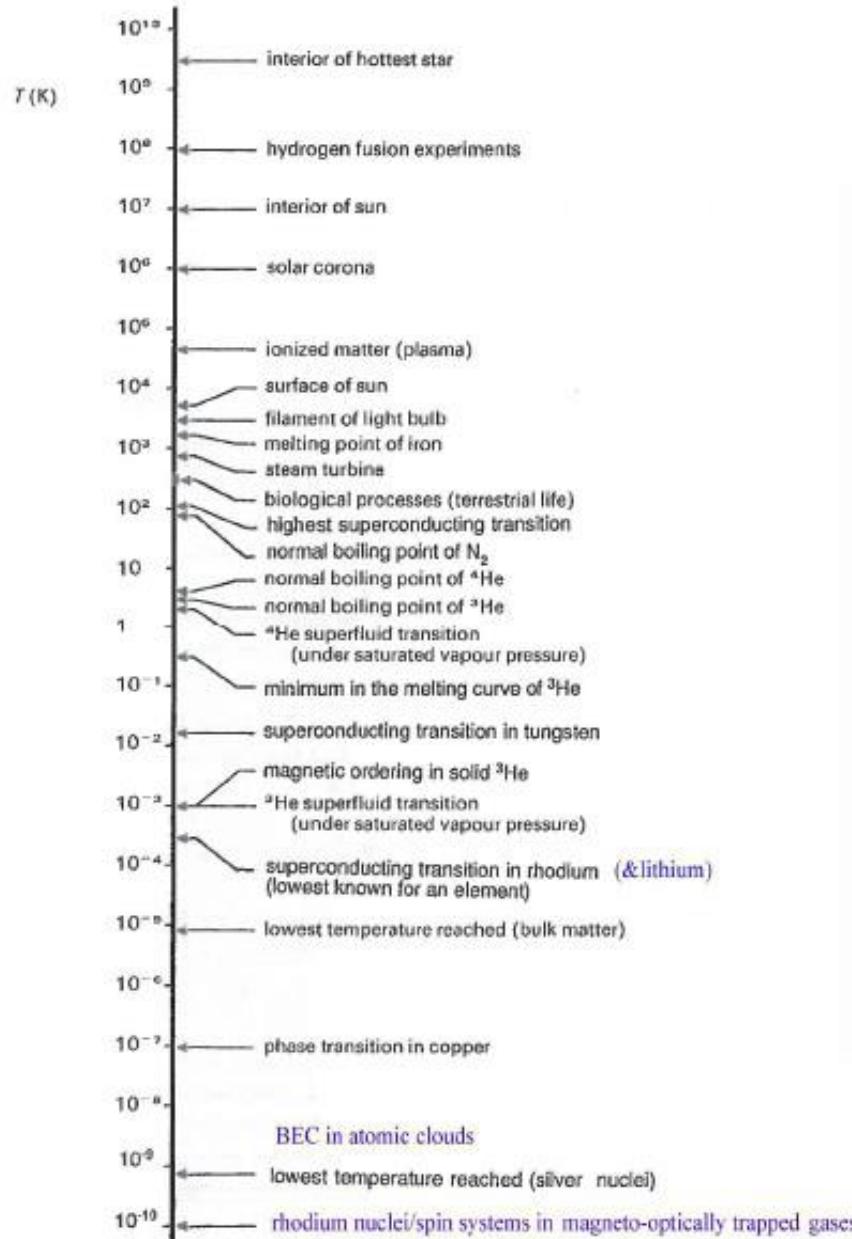
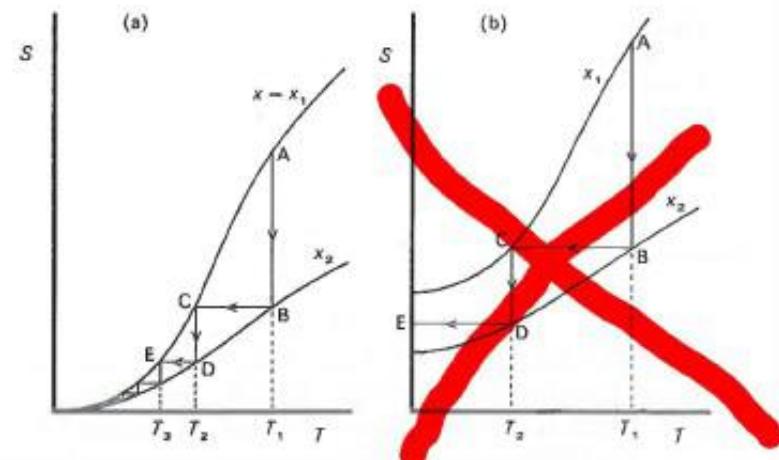


Figure 1.1 The temperatures  $T$  (in Kelvin) at which some selected phenomena occur, illustrating the huge range which is accessible. Note that the scale is logarithmic.

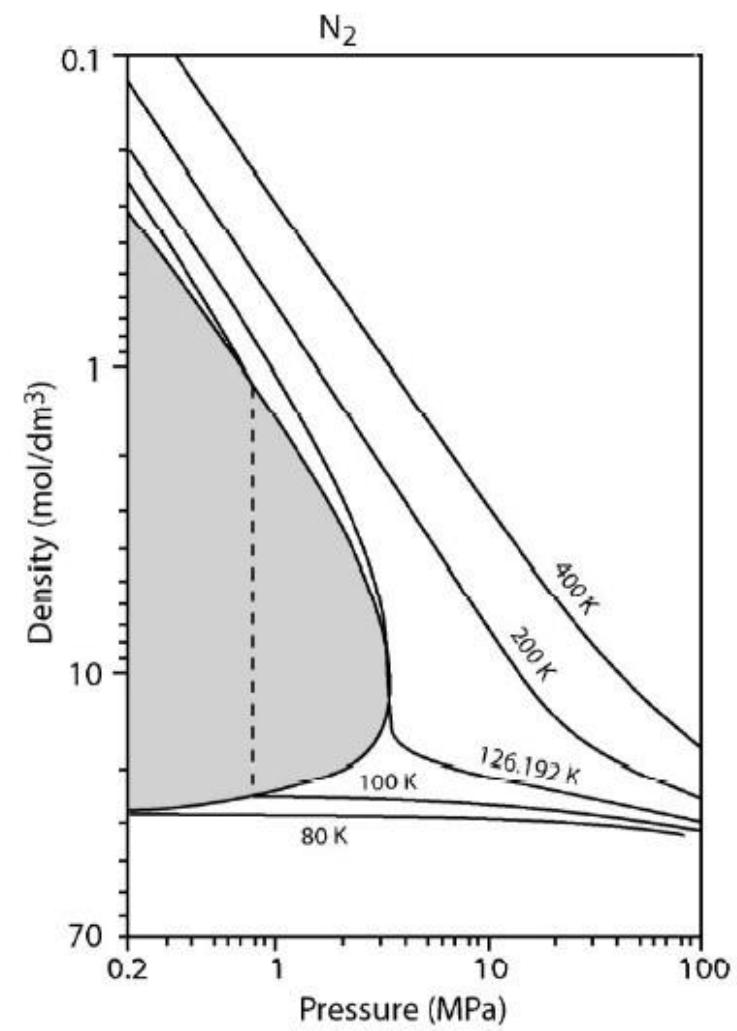
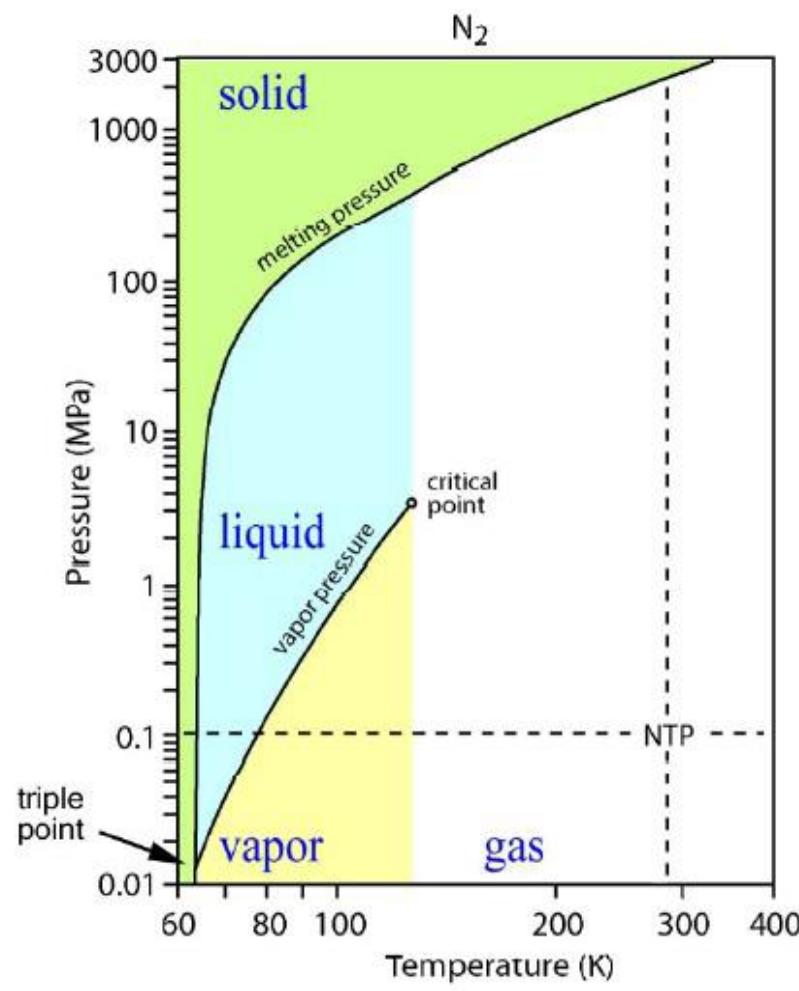
## On unattainability of $T = 0$



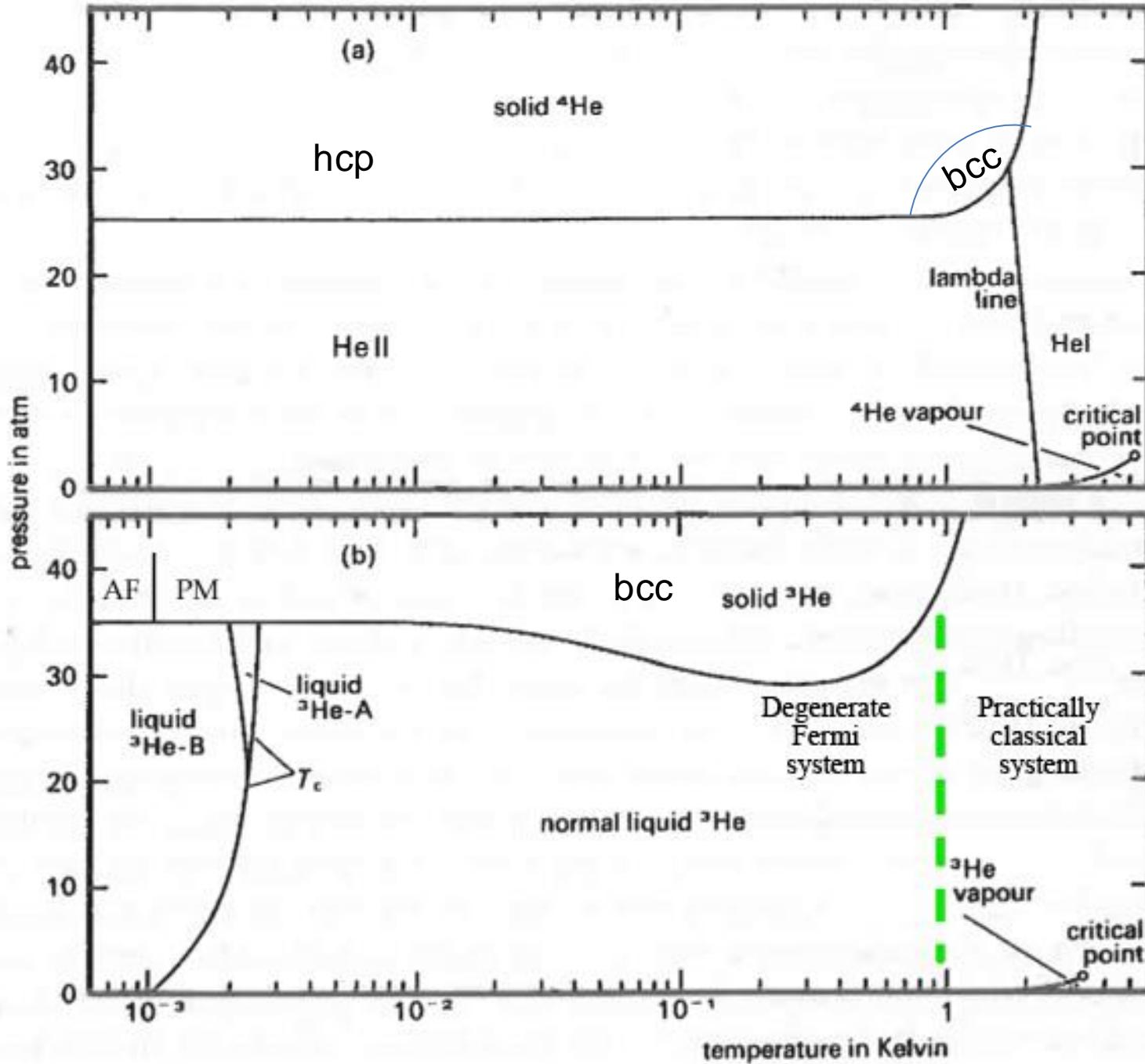
It should be noted that, although the goal cannot be attained, it may nonetheless be approached arbitrarily closely. An interesting comment made by F.E. Simon (1952) is that it must be possible, at least in principle, to reach whatever low temperature may be necessary for the study of any given physical phenomenon. That is, it should be possible to design the experiment in such a way that the entropy changes involved in the phenomenon under study are themselves used to provide the required degree of cooling:

It will only become progressively more difficult to reduce the temperature when the system has lost practically all its entropy. In this case, only a desert lies before us and so we are not interested in going further: we have already to all extents and purposes a system which is hardly distinguishable from one at absolute zero and its properties can be safely extrapolated. If, on the other hand, some new phenomenon is going to occur at lower temperatures, then we will be able to make use of it to reach this oasis of interest. Thus the law of unattainability of absolute zero is no barrier to our knowledge.

# Phase diagram of nitrogen N<sub>2</sub>



# Phase diagrams of helium



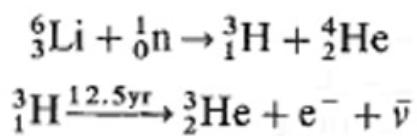
Clausius-Clapeyron equation:

$$\frac{\partial p}{\partial T} = \frac{S_l - S_s}{V_l - V_s}$$

$$S_s > S_l$$

Figure 1.9 Low-temperature phase diagrams (a) for  $^4\text{He}$ , and (b) for  $^3\text{He}$  under zero magnetic field.

Most of the  ${}^3\text{He}$  used for cryogenic purposes is now prepared as a byproduct of tritium manufacture and storage, through the neutron bombardment of lithium:



thermonuclear fuel: the heavy hydrogen isotopes [deuterium](#) and [tritium](#), or in modern weapons [lithium deuteride](#).

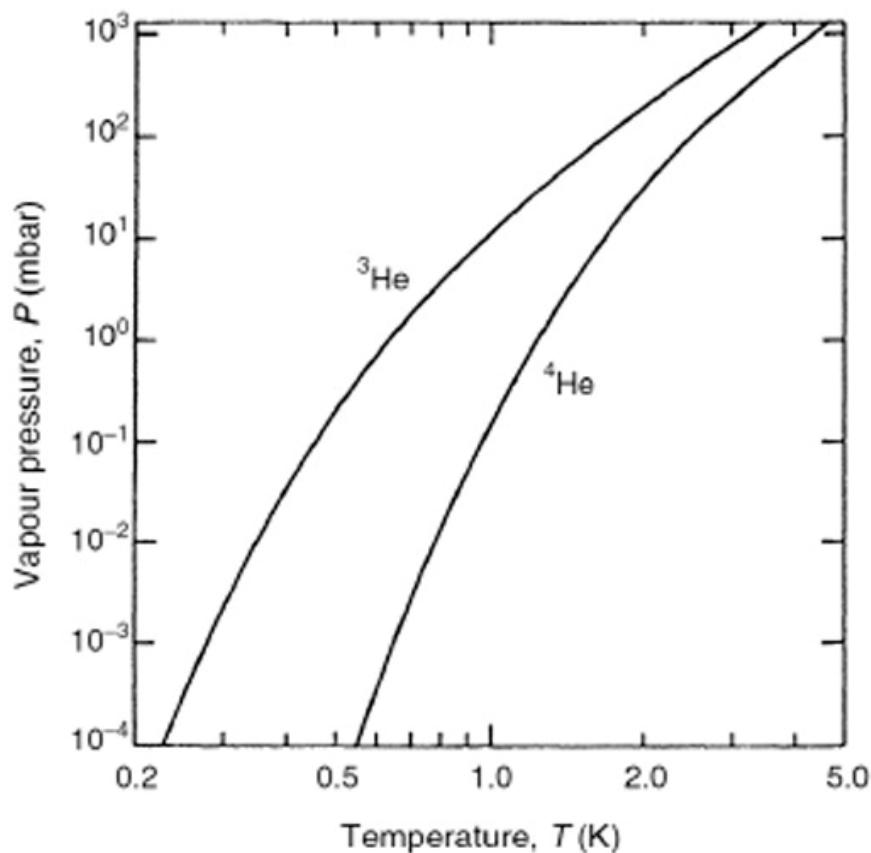


Fig. 2.7. Vapour pressures of liquid  ${}^3\text{He}$  and liquid  ${}^4\text{He}$

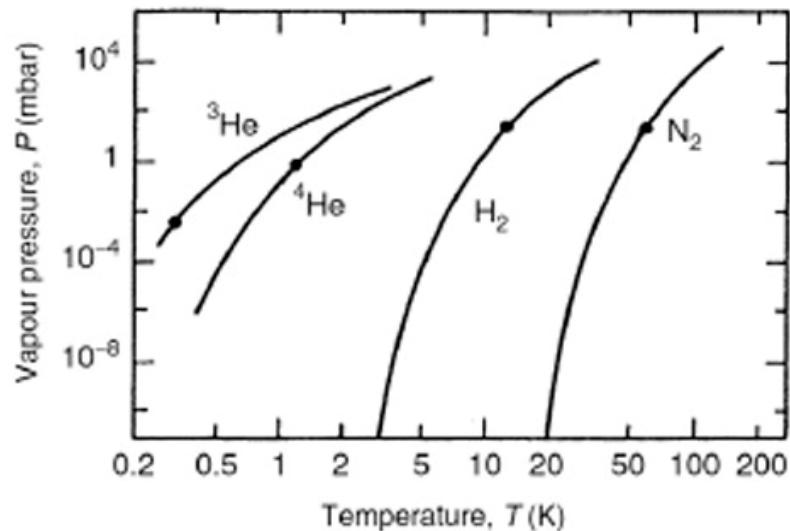
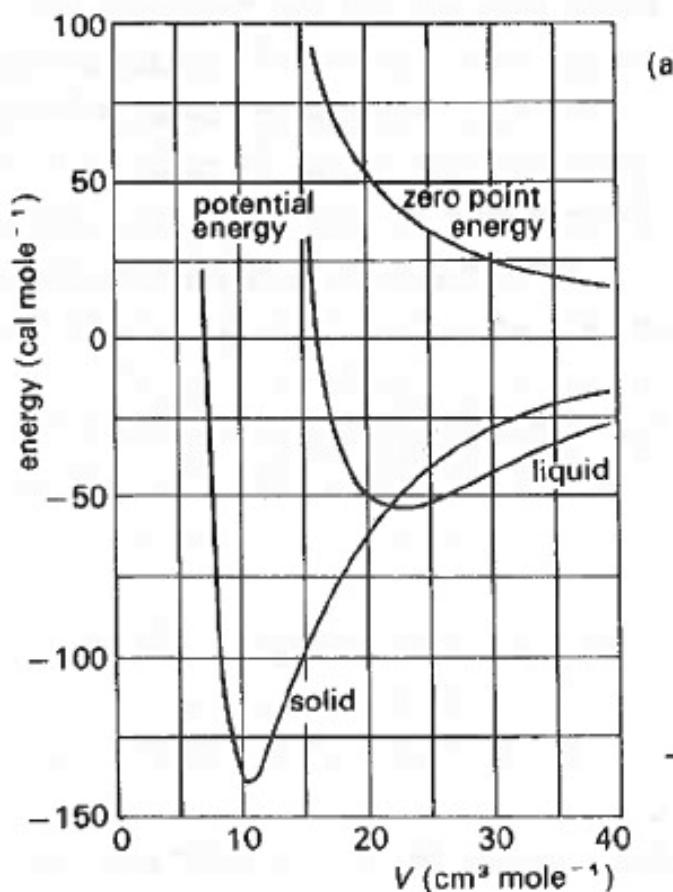


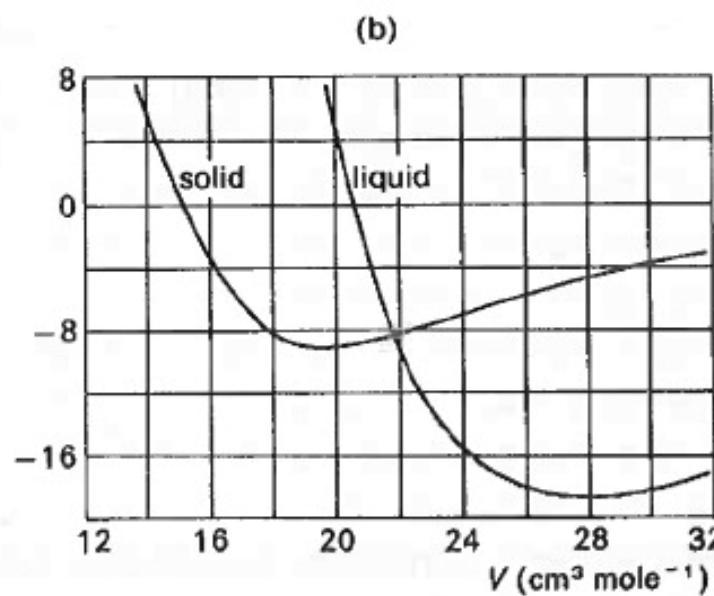
Fig. 2.8. Vapour pressures of various cryoliquids. The dots indicate the practical lower limits for the temperatures which can be obtained by reducing the vapour pressure above these liquids

Why does He remain liquid at  $T = 0$  ?

- weak mutual attraction, only minute dielectric effect (van der Waals force)
- strong zero-point fluctuation (Heisenberg)



(a)



$$\Delta p \geq \hbar / \Delta x \sim \hbar / a$$

$$E_z \sim \frac{\Delta p^2}{2m} \sim \frac{\hbar^2}{2ma^2}$$

$$E_z = \frac{3\pi^2 \hbar^2}{2mV^{2/3}}$$

Remains in  
liquid phase

${}^4\text{He}$ :

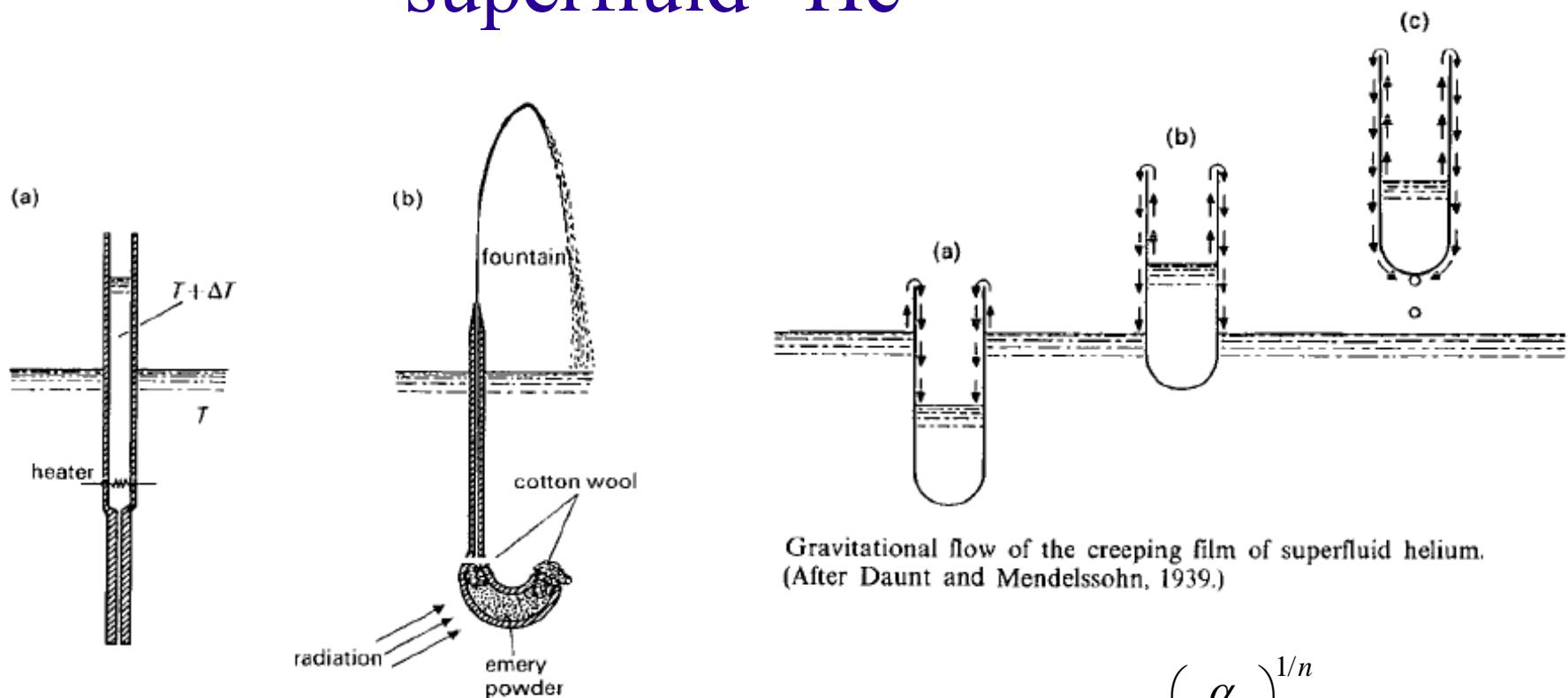
$$V_{\text{molar}} = 28 \text{ cm}^3$$

${}^3\text{He}$ :

$$V_{\text{molar}} = 40 \text{ cm}^3$$

**Figure 1.10** Illustration (after F. London, 1954) of how zero-point energy prevents  ${}^4\text{He}$  from solidifying under its saturated vapour pressure. Note the change of scales between (a) and (b) (and that 1 calorie = 4.2 joules).

# Some peculiar properties of superfluid $^4\text{He}$



Gravitational flow of the creeping film of superfluid helium.  
(After Daunt and Mendelssohn, 1939.)

**Figure 5.6** The thermomechanical (fountain) effect in HeII (Allen and Jones, 1938). (a) When the temperature of the HeII in the vessel is increased slightly above that of the bath, liquid flows in through the capillary tube. (b) The same phenomenon (using radiative heating in the case illustrated) can be used to create a dramatic fountain of liquid helium.

$$\frac{Dv_s}{Dt} = -\frac{1}{m_4} \frac{\Delta\mu}{\Delta x} = -\frac{1}{\rho} \frac{\Delta p}{\Delta x} + \sigma \frac{\Delta T}{\Delta x} = 0 \quad \frac{\Delta p}{\Delta T} = \rho\sigma$$

$$d = \left( \frac{\alpha}{gH} \right)^{1/n}$$

$\alpha$  – van der Waals interaction

$d$  – film thickness

$g$  – gravitational constant

$H$  – height from helium level

$n = 3\dots 4$

# Risks in cryogenic laboratory work

## \* Low temperature "burns"

- don't spill LN2 on your clothes with pockets, shoes or gloves catching some fluid
- do not touch cold transfer tubes without gloves
- easy to avoid if common sense is used

## \* Suffocation

- when large amounts of nitrogen or helium evaporate quickly to open air, the oxygen content may be reduced significantly. Take care of proper ventilation!
- excessive evaporation can happen if vacuum isolation is broken, a big SC magnet quenches, LN2 is overfilled or a vessel is emptied by increasing its boil-off

## \* Fire hazard

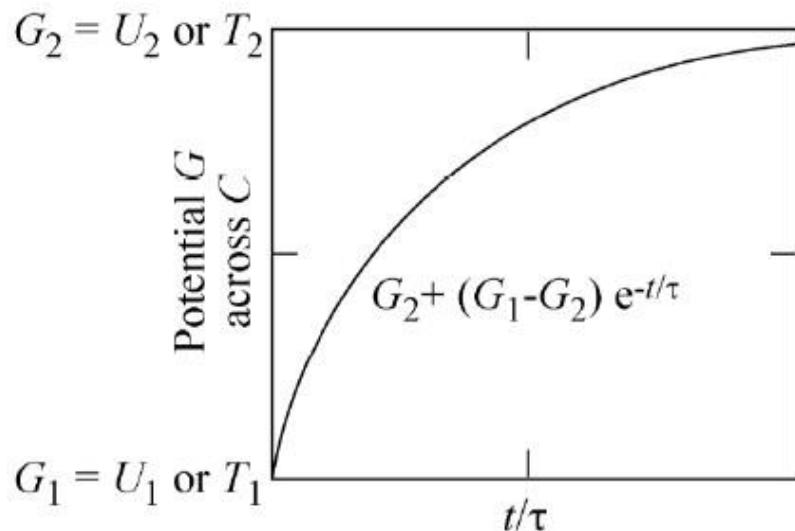
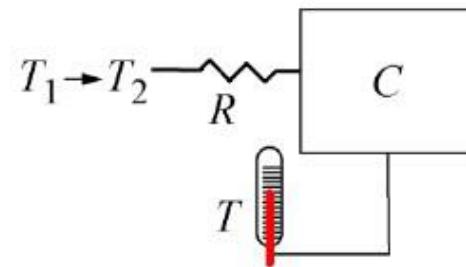
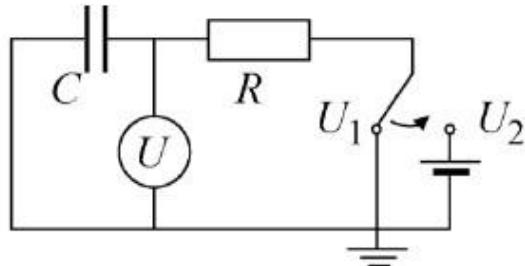
- in presence of LO2, any spark on hydrocarbons or charcoal causes explosion
- open vessels of LN2 may condense some LO2 from air, which remains there until all LN2 has boiled off (can be recognized from the bluish color)
- not so serious problem nowadays, as liquid air or LH2 is used no more

## \* Pressure accumulation in cryogenic vessels or pumps in closed circulation systems

- always make sure that there is an open exhaust line (+ safety valve)
- used to be an issue with glass dewars
- before starting a pump, check the back side
- regulators on high pressure bottles

# Thinking of familiar analogies may help

Potential:	electric	$U$	thermal	$T$
Charge:		$q$		$Q$ (heat)
Current:		$I = dq/dt$		$dQ/dt$
Capacitance:		$C = dq/dU$		$C = dQ/dT$
Conductivity:		$\sigma$		$\kappa$
Resistance:		$R = L/\sigma A$		$R = L/\kappa A$



Ohms law:  
 $\Delta U = RI$   
 $\Delta T = R(dQ/dt)$   
 RC time constant:  
 $\tau = RC$  (both cases)  
 + Kirchhoff's circuit laws  
 can be applied!