Cryogenic Techniques below 1K

How to get cold, stay cold and measure something!

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Initial statement of problem

☐ You need to choose a refrigerator that can get colder than the temperature needed by your experiment T_E

☐ The refrigerator must have sufficient cooling power to absorb the power dissipated by the measurement $\dot{Q}_E$ and still maintain T_E

☐ You need to cool the sample through some thermal link:
- sample must cool in a reasonable time
- thermal contact must be good enough that $\dot{Q}_E$ doesn't warm the experiment above T_E

☐ You need to be able to measure temperature:
- resolution, stability, transferability
- thermal contact
- power dissipation of the thermometer
- response time of the thermometer
- sensitivity to external parameters   e.g. magnetic field

Books:

O V Lounsamaa	Experimental Principles & Methods below 1K Academic Press 1974
D S Betts	Refrigeration and Thermometry below 1K Sussex Univ Press 1976
R C Richardson & E N Smith	Experimental Techniques in Condensed Matter Physics at Low Temperatures, Addison Wesley 1988
F Pobell	Matter & Method at Low Temperatures Springer Verlag 1996

Cooling Methods

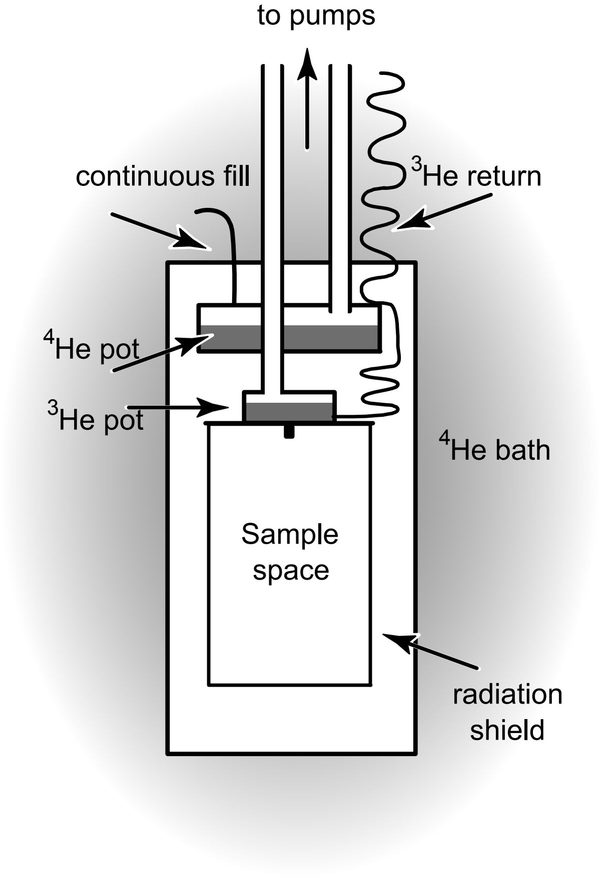
The main choices of refrigeration method:

a)	$T > 0.25$ K	^{3}He evaporation cryostat
b)	$1.0 > T > 0.003$ K	^{3}He - ^{4}He dilution refrigerator
c)	$T < 0.003$ K	Adiabatic nuclear demagnetisation
	Others: *Not discussed further*	Adiabatic electron demagnetisation† $PrNi_5$ demagnetisation Pomeranchuk cooling

† For example, the recently developed Cambridge Magnetic Research mFridge.
$T_{min} < 40$mK, 24 hours @ 100mK

Cryogen-free systems now becoming available which can be combined with one of the above.

a) ^{3}He evaporation cryostat



❑ commercially available
❑ reasonable cooling power

Oxford Instruments:
T_{base}= 250mK,
40μW for 6 hours with T < 300 mK

Temperature dependence of ^{3}He vapour pressure described by Clausius-Clapeyron equation

$$\frac{dP}{dT} = \frac{\Delta S}{\Delta V}$$

Ignoring negligible liquid molar volume, substituting for the (approx constant) latent heat $L=T\Delta S$, find

$$P \propto \exp(-L / RT)$$

Cooling power ∝ mass flow across phase boundary ∝ P. ∴ exponentially falling cooling power.

L/R=2.5 K limits T_{base} to 0.2 – 0.3 K

Costs:

❑ ^{4}He £3 per liquid litre, ^{3}He £60,000 per liquid litre!

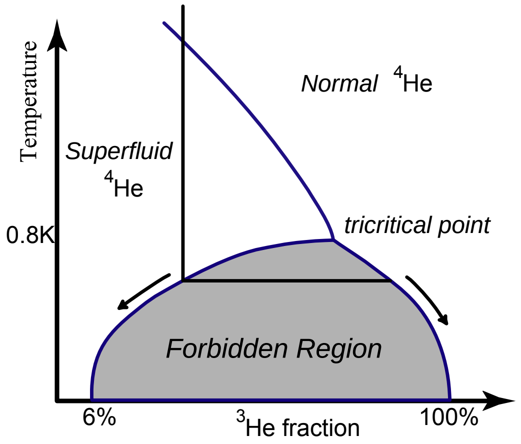
b) ^{3}He-^{4}He dilution refrigerator

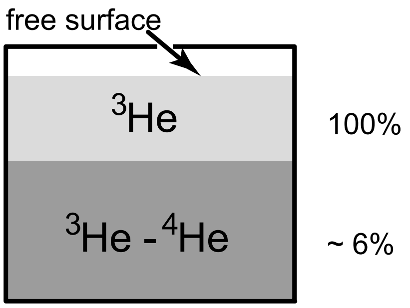
- ☐ commercially available
- ☐ not cheap to buy or run!

Oxford Instruments:
T_{base} < 7mK,
cooling power > 300µW at 100 mK

Just a different form of evaporation cooling.

Well described in literature:
Frossatti, LT15, J de Physique C6,
sup 8, 1578, 1975

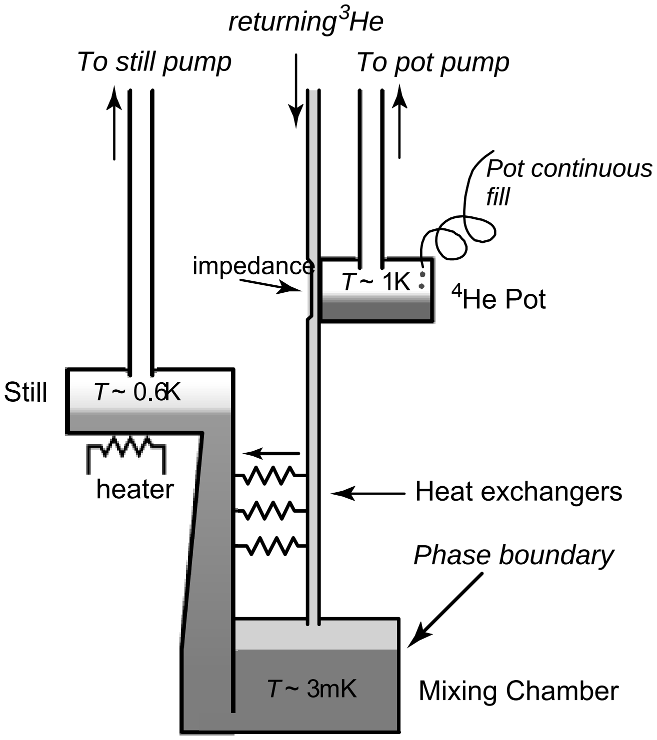


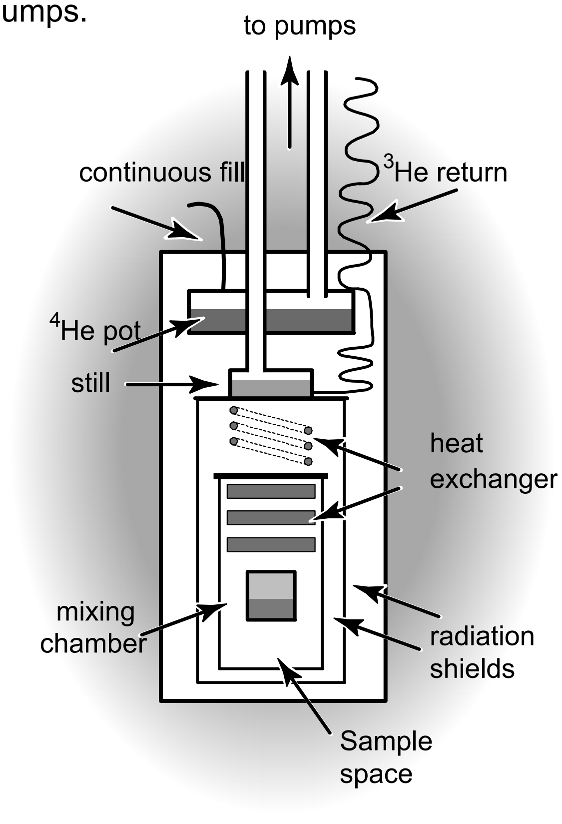


How does it work? ^{3}He-^{4}He mixture phase separates as it cools into a ^{3}He-rich phase floating on top of a ^{4}He-rich phase. At T~0 K, the ^{3}He-rich phase is 100% ^{3}He whilst the ^{4}He-rich phase is ~6.6% ^{3}He. If ^{3}He can be made to 'evaporate' across the phase boundary from the ^{3}He-rich phase to the ^{4}He-rich phase, then cooling can occur. The entropy of each phase is proportional to T (Fermi statistics) with the entropy of the dilute ^{4}He-rich phase being larger than that of the pure phase. (A proper argument uses enthalpies – see Lounasmaa.) For a given ^{3}He circulation rate $\dot{n}_3$, the cooling power at T is approximately

$$\dot{Q} \sim 80\,\dot{n}_3\,(T^2 - T_{base}^2)$$

Circulation rate can be 1000 µmol s^{-1} with *big* pumps.





Problems and solutions

Use a log-book to keep a good record of all changes!

☐ Wiring:
- Use insulated superconducting wire with CuNi cladding, not copper cladding
- Thermally anchor at each intermediate stage
- Beware difficulty in cooling the core of coax cables
- Twisted pairs usually OK for low frequency
- Liquid fill lines anchor at all intermediate stages
- can you get the wiring or fill line out non-destructively?

Common problems:

☐ Fridge initially gets cold then warms up 24h or so later to ~1K
- small leak – look for excess ^{4}He or ^{3}He in vacuum space

☐ Pot empty
- blocked continuous fill
  - ➢ dirty ^{4}He or air leak to ^{4}He bath
- large heat load on pot
  - ➢ touch?
  - ➢ any new wiring?

☐ Base T very high
- touch between shields
- large heat load
  - ➢ diagnostic see if cools if increase circulation rate – if so
    - o touch?
    - o any new wiring?

☐ Condensers keep blocking
- air leak into system
- ‘cold traps’ not cold or contaminated
- poor procedures?
  - ➢ pump carefully at start of run
  - ➢ ensure equal pressure both sides of an impedance

Dilution fridge specific:

☐ Fridge won’t start to cool or cools badly
- Has something changed?
  - ➢ mixture loss
  - ➢ wiring changed
  - ➢ new experimental cell

!! Do you really want to continue?? The following is for experts only - seek advice locally first before doing anything which might be irreversible!!!

Symptom:

☐ no vapour pressure in still

☐ no cooling of still

- still empty
  - ➢ too little mixture – add ^{4}He

Symptoms:

☐ mixing chamber not very cold

☐ mixing chamber not the coldest

- phase boundary in wrong place – difficult to tell!
  - ➢ remove ^{3}He to storage containers and monitor temperature

c) Adiabatic Nuclear Refrigeration

☐ Single shot process

☐ $T < 3$ mK

Needs

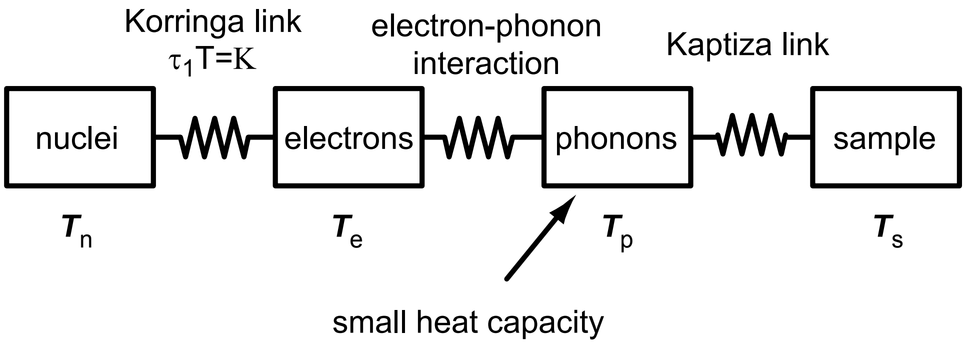
- large magnetic field, B > 6 Tesla
- dilution fridge precool to < 10 mK through superconducting heat switch

see Pickett, Rep Prog Phys 51, 1295, 1988 for a recent review or Pobell's book

Cool nuclear paramagnet, usually copper, in high field, isolate and remove B.

Entropy $S = f(B / T)$; adiabatic S = const $\therefore B \downarrow \quad T\downarrow$

difficulty of thermal contact



Limits:

nuclear spins	$T_{nuc} \sim 100$ pK
electrons	$T_{electron} \sim 1$ μK
^{3}He	$T_3 \sim 80$ μK
^{3}He –^{4}He mixtures	$T_3 \sim 100$ μK ??

Thermal Contact

☐ The lowest sample temperature is often determined NOT by the refrigerator cooling power/base temperature but by the thermal link to the sample

heat flow $\dot{Q}$ through finite thermal resistance *R* causes ΔT

$$\dot{Q} = \Delta T / R$$

Thermal time constant $\tau = RC$ where *C* is the sample heat capacity

Metallic contact:

Thermal conductivity is very difficult to measure. Fortunately, we can use the Weidemann-Franz law which relates the thermal and electrical conductivities. These quantities are related through the constant

$$L_0 = \frac{K}{\sigma T} = 2.45 \times 10^{-8}\,\mathrm{W\Omega\,K^{-2}}$$

where K is the thermal conductivity at temperature T, and σ is the electrical conductivity at T. The room temperature electrical conductivity is usually well known and the low temperature conductivity can be found by a quick measurement of the ratio of the resistivity at room temperature to that at 4K– the Residual Resistance Ratio RRR = ρ_{293K} / ρ_{4K} (connect up a 4-probe resistance measurement and dip the sample into a ^{4}He dewar. The ratio avoids needing to know the sample dimensions accurately.)

typically:

- 50 –100 for untreated copper wire
- 1 for alloys - Stainless Steel, CuNi

if very pure (99.999%) starting material, heat treating can raise this to 1000 – 10000

Thermal resistance $R = l / KA$ where

$$K = L_0 \frac{RRR}{\rho} T \quad \text{where} \quad \sigma = \frac{RRR}{\rho}$$

so

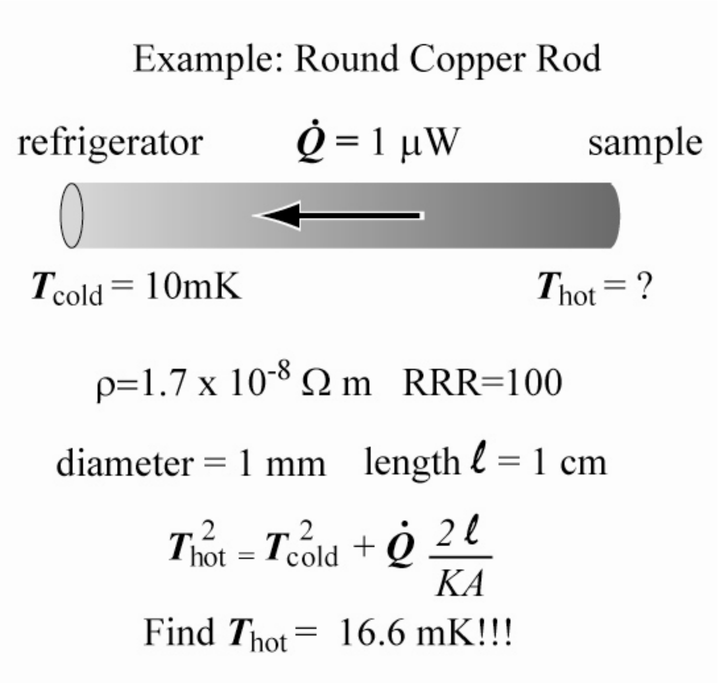
$$\dot{Q} = \frac{KA}{l} \Delta T$$

If the temperature difference ΔT across the conductor is large, you must integrate

$$\dot{Q} = \int_{T_{cold}}^{T_{hot}} \frac{\mathrm{K}A}{l}\, dT$$

$$\dot{Q} = \frac{A}{l} L_0 \frac{RRR}{\rho} \frac{(T_{hot}^2 - T_{cold}^2)}{2}$$

As an example, lets consider a 1mm diameter untreated copper wire straight off a reel connecting your sample to the mixing chamber of a dilution fridge at 10mK. We'll assume 1μW power is dissipated in the sample, the typical cooling poower of a dilution efrigerator at 10mK..



Example: Round Copper Rod

refrigerator $\dot{Q} = 1\ \mu\mathrm{W}$ sample

$T_{\mathrm{cold}} = 10\mathrm{mK}$ $T_{\mathrm{hot}} = ?$

$\rho = 1.7 \times 10^{-8}\ \Omega\ \mathrm{m}$ RRR=100

diameter = 1 mm length ℓ = 1 cm

$$T_{\mathrm{hot}}^2 = T_{\mathrm{cold}}^2 + \dot{Q}\,\frac{2\,\ell}{KA}$$

Find $T_{\mathrm{hot}} =$ 16.6 mK!!!

Metallic sinters

Boundary resistance between fine metal particles (sinters) and the He liquids inversely proportional to the sinter area and depends on T as

^{3}He – ^{4}He		$R \propto 1/T^2$
^{3}He	$T > 10$ mK	$R \propto 1/T^3$
^{3}He	$T < 10$ mK	$R \propto 1/T$

Screw Joints

screw joints can have contact resistance < 0.1μΩ with care. $R \sim 4/T$ K^2/W

Thermal Isolation

Insulators:

Best material

- ☐ Vespel SP22
  - strong, machinable
  - low thermal conductivity $K = 17 \times 10^{-4} T^2$ W m^{-1} K^{-1}Locatelli, Cryogenics **16**, 374, 1976

Superconducting heat switches

	T_C(K)	H_C(mT)	contact method
Sn	3.7	30.4	solder¶
Zn	0.85	5.3	solder with Cd† or In
Al	1.16	10.3	gold plate & clamp or melt

¶ beware changing to grey tin powder

† highly toxic!

Vibrational Isolation

- ☐ think about it especially if you have large magnetic fields
  - use air springs to decouple from floor
  - dump vibrations from pipework, etc in heavy masses

Thermometers

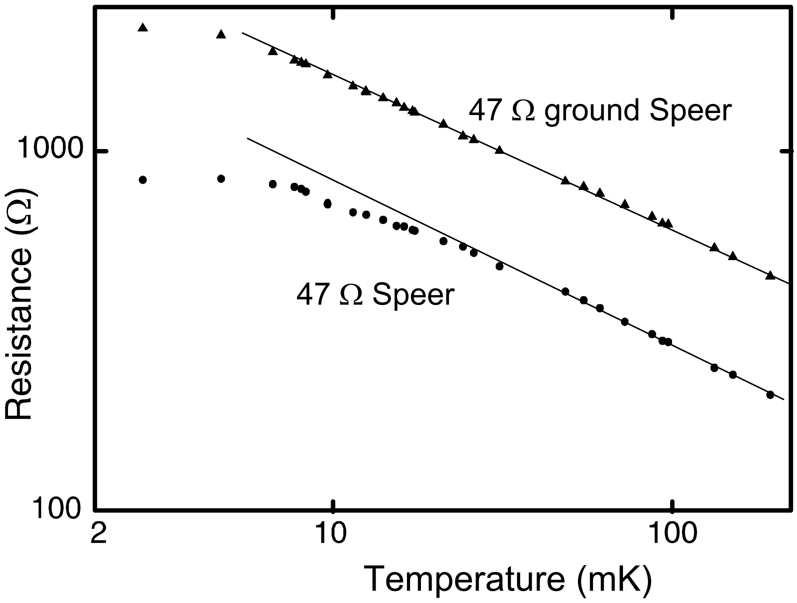
- ➢ thermal contact difficult.
- ➢ want small *C* to get fast response

Resistance thermometry

(Speer) carbon resistor

- ☐ usable to ~10 mK with care
- ☐ power dissipation must be < T^3 nW/K^3 ~10^{-15} W at 10 mK
- ☐ field dependent
- ☐ cooled mainly through the leads
  - glue thin slice to copper plate improves thermal contact.

The examples below are of a 47Ω Speer resistor mounted directly in the liquid in the mixing chamber and a similar 47Ω Speer resistor ground to ~0.5mm thick, glued to a copper plate (insulated by stycast 1266 impregnated cigarette paper, attached by a silver wire to a sinter pad in the mixing chamber. Both resistors have simple low-pass LC filters on the electrical leads. The ground resistor tracks T to much lower temperatures.



Germanium resistor

- ☐ expensive & fragile
- ☐ some OK down to ~ 20 mK
- ☐ field dependent

RuO_2 resistor

- ☐ not very field dependent
- ☐ as Speer resistors otherwise

susceptibility thermometry

CMN

- ☐ Curie-Weiss law $\chi = C/(T-\Delta)$
- ☐ SQUID useful
- ☐ two forms
    - powder form for He liquids
    - slurry with glycerol in fine wires for thermal contact to solid
- ☐ usable down to ~3 mK with care
    - dilute with La to go lower
- ☐ field dependent
- ☐ can order above 3 mk if dehydrated

PdFe (few % Fe) see Pobell's book

- ☐ Curie law $\chi = C/T$
- ☐ ~50 mK to << 1mK
- ☐ SQUID necessary
- ☐ field dependent
- ☐ tunable range by Fe content

NMR Pt wire or powder

- ☐ field independent
- ☐ Curies law $\chi = C/T$
- ☐ pulsed technique
  - τ_2 short τ_1 long (= 0.03/T s)
- ☐ commercially available
- ☐ 20 mK to ~1 μK with care

Capacitance thermometer – Frossatti LT18, p1723

- ☐ field independent
- ☐ 1 K to 1 mK
- ☐ fast response, low dissipation,
- ☐ resolution 10μK at 1mK
- ☐ needs 'fancy' C bridge Andeen-Hagerling

^{3}He melting curve

- ☐ 0.3K – 2 mK
- ☐ difficult to calibrate - needs T_A
- ☐ field independent
- ☐ large heat capacity – slow response

Nuclear Orientation

- ☐ measure γ-ray decay anisotrophy
- ☐ constant heat leak which cannot be turned off!
  - 650 pW per 1μC ^{60}Co
- ☐ slow
- ☐ needs expensive detectors

Vibrating wire resonators

- ☐ useful in ^{3}He or ^{3}He-^{4}He mixtures
- ☐ 50 mK to < 100 μK
- ☐ measures liquid temperature directly
- ☐ requires magnetic field