Dilution refrigerators

European Course of Cryogenics 2022

August 23rd, 2022

Daan Wielens

Quantum Transport in Matter / Interfaces and Correlated Electron systems

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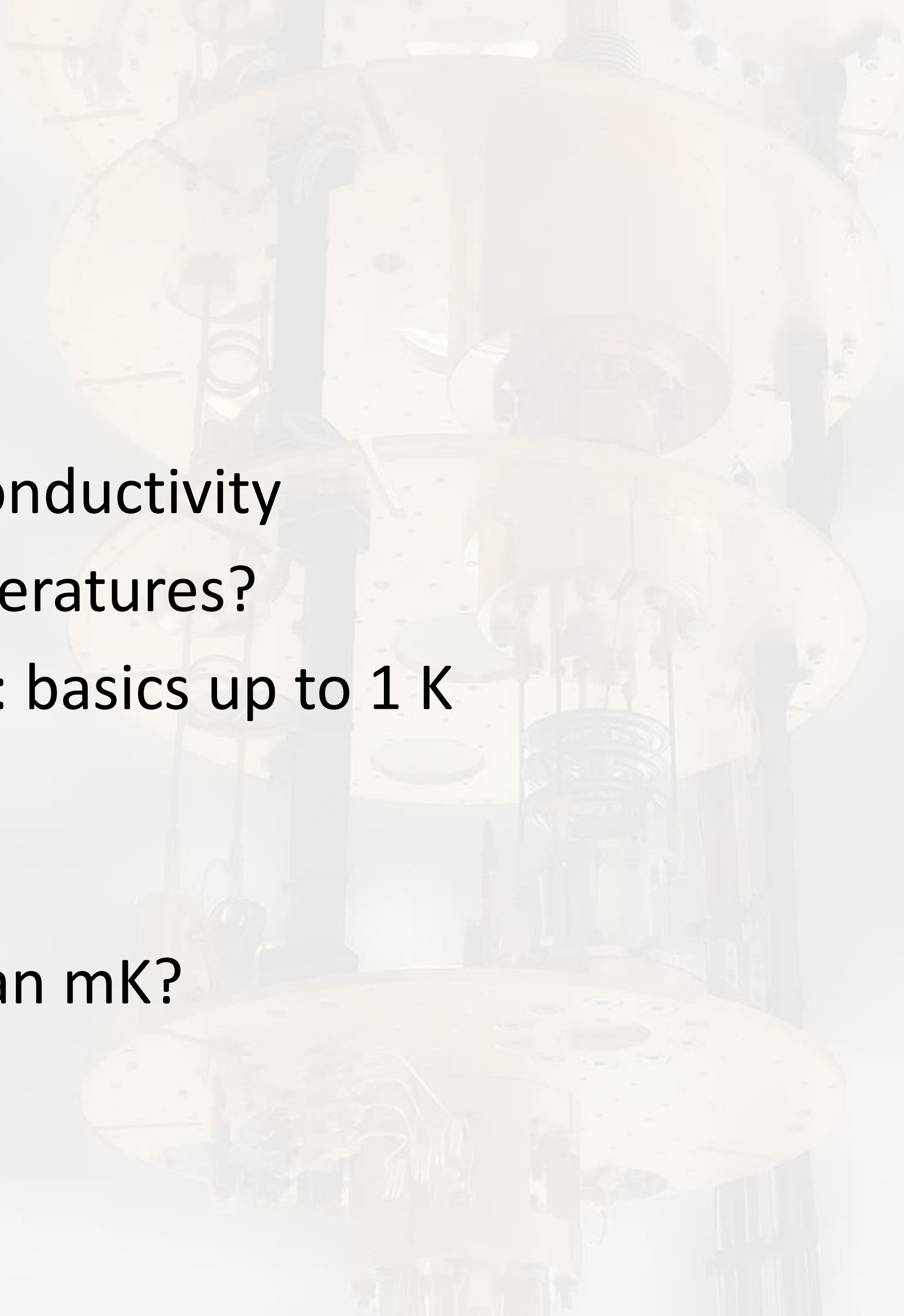
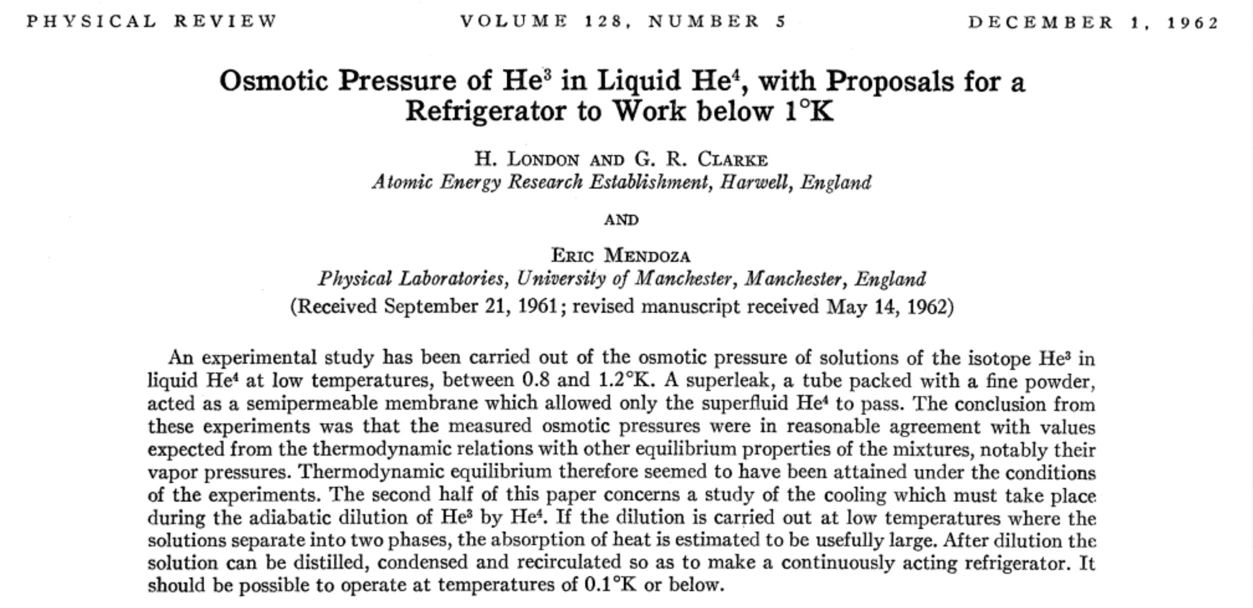


- History
- Intermezzo: superconductivity
- Why go to mK temperatures?
- Wet and dry fridges: basics up to 1 K
- Dilution unit
- Dry fridges
- Can we go lower than mK?

History

- First suggested by ***London*** in **1952**
- ***London, Clarke, and Mendoza*** proposed a continuous refrigerator in **1962**



PHYSICAL REVIEW VOLUME 128, NUMBER 5 DECEMBER 1, 1962

Osmotic Pressure of He^3 in Liquid He^4, with Proposals for a Refrigerator to Work below 1°K

H. London and G. R. Clarke
Atomic Energy Research Establishment, Harwell, England

and

Eric Mendoza
Physical Laboratories, University of Manchester, Manchester, England

(Received September 21, 1961; revised manuscript received May 14, 1962)

An experimental study has been carried out of the osmotic pressure of solutions of the isotope He^3 in liquid He^4 at low temperatures, between 0.8 and 1.2°K. A superleak, a tube packed with a fine powder, acted as a semipermeable membrane which allowed only the superfluid He^4 to pass. The conclusion from these experiments was that the measured osmotic pressures were in reasonable agreement with values expected from the thermodynamic relations with other equilibrium properties of the mixtures, notably their vapor pressures. Thermodynamic equilibrium therefore seemed to have been attained under the conditions of the experiments. The second half of this paper concerns a study of the cooling which must take place during the adiabatic dilution of He^3 by He^4. If the dilution is carried out at low temperatures where the solutions separate into two phases, the absorption of heat is estimated to be usefully large. After dilution the solution can be distilled, condensed and recirculated so as to make a continuously acting refrigerator. It should be possible to operate at temperatures of 0.1°K or below.

- It was realized in **1964** in the **Kamerlingh Onnes Lab** at Leiden University **(220 mK)**

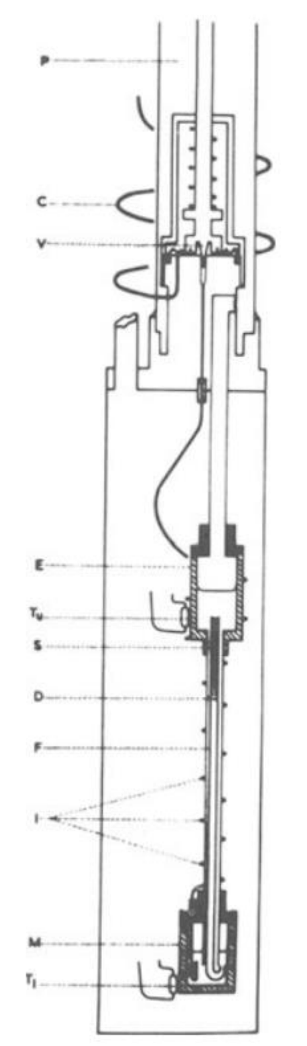
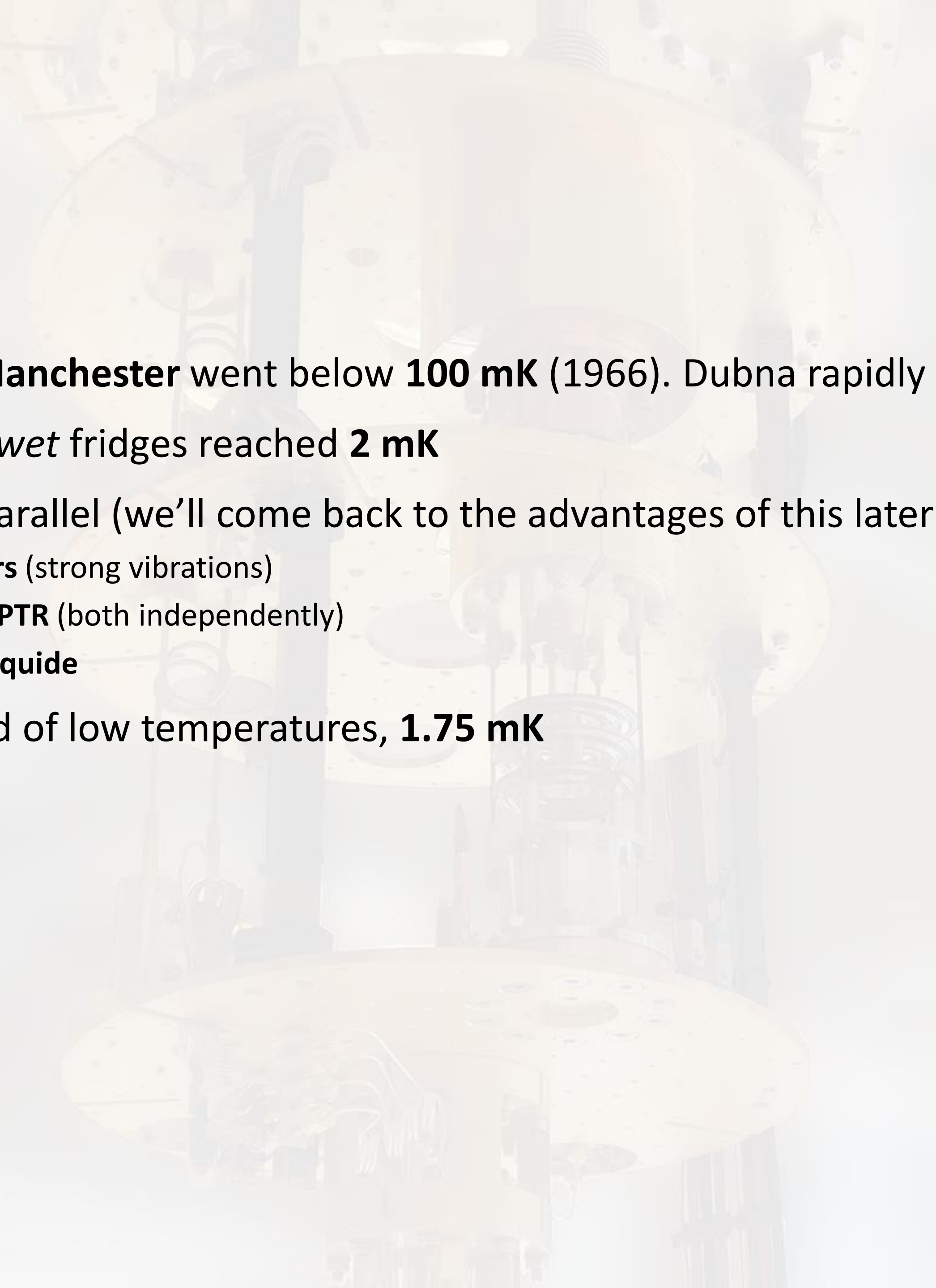


Fig. 1.

History

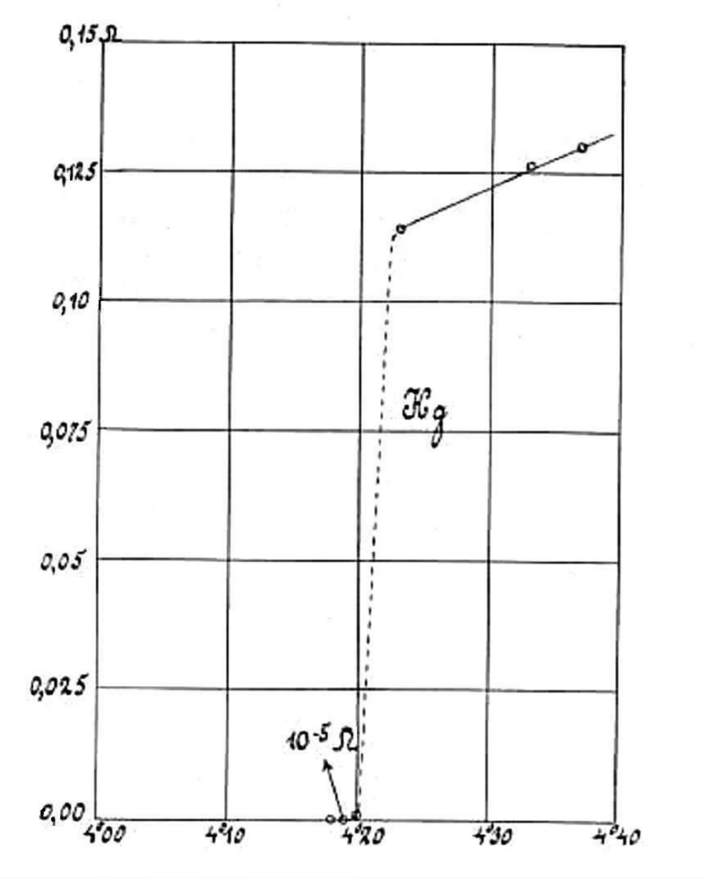


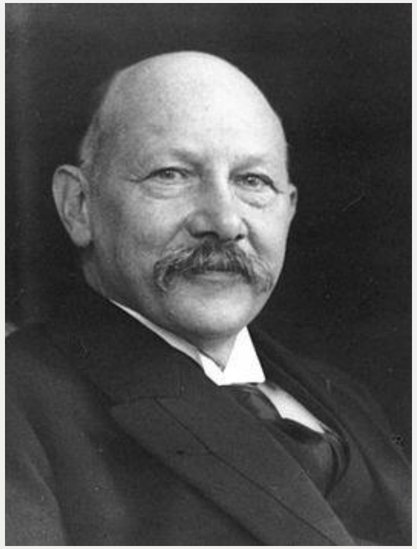
- Neganov in **Dubna** and Hall in **Manchester** went below **100 mK** (1966). Dubna rapidly reached **25 mK**.
- After numerous developments, *wet* fridges reached **2 mK**
- Dry fridges were developed in parallel (we'll come back to the advantages of this later):
    - 1993 by **Uhlig** who used **GM coolers** (strong vibrations)
    - ~ 2002 by **Uhlig** and **Godfrin** using **PTR** (both independently)
    - 2003 first commercial unit by **Air Liquide**
- **Lancaster** has the present record of low temperatures, **1.75 mK**

Intermezzo: superconductivity

- Discovered in Leiden, The Netherlands, in 1911 by H. Kamerlingh-Onnes

- Superconducting state has two properties:
  - **Zero resistance**
  - Zero magnetic field





Intermezzo: superconductivity

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- Superconducting state has two properties:
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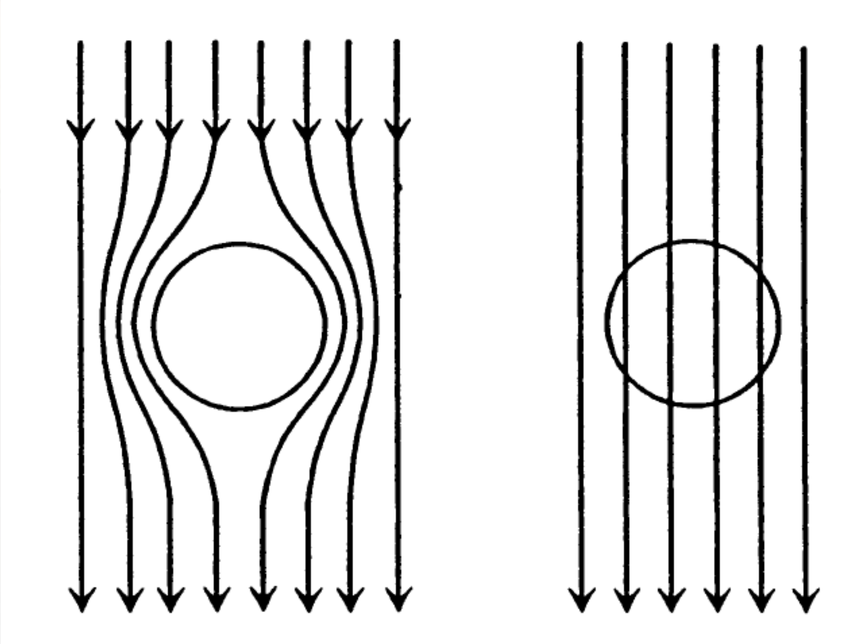
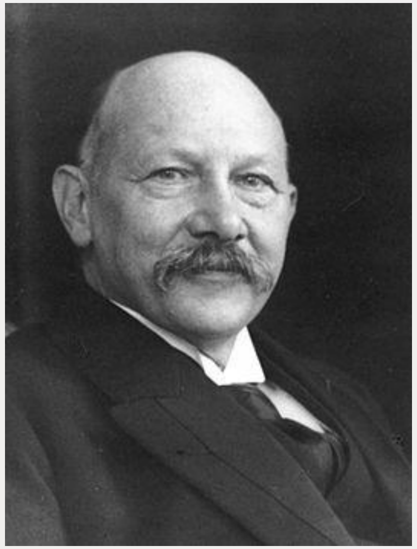
    (Meissner effect)



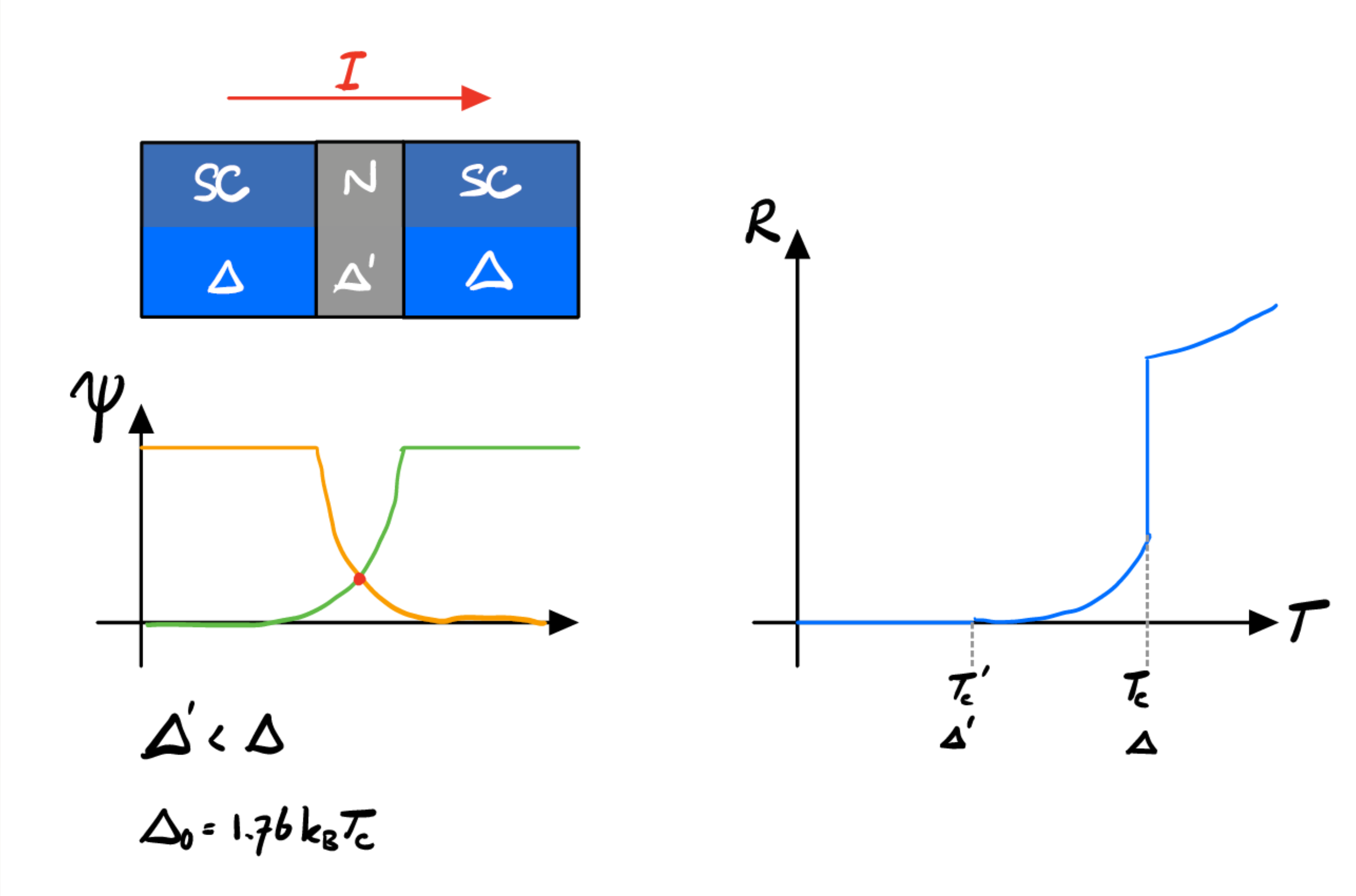


Intermezzo: superconductivity

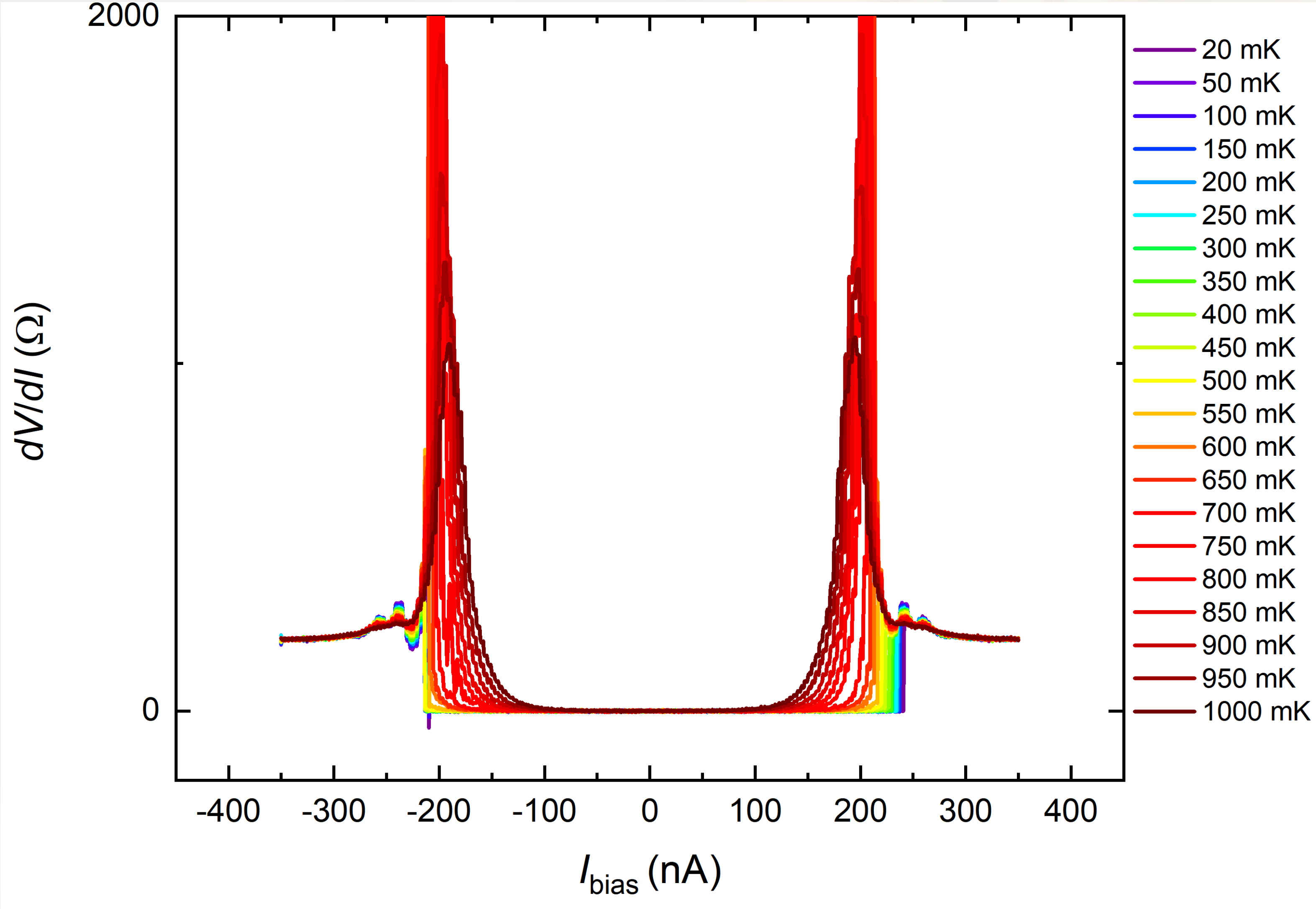
Material	Critical temperature (K)	Critical field (T)	Type	BCS
Al	1.20	0.01	I	Yes
Hg	4.15	0.04	I	Yes
Nb	9.26	0.82	II	Yes
Pb	7.19	0.08	I	Yes
Nb3Sn	18.3	30	II	Yes
NbTi	10	15	II	Yes
YBCO	95	120-250	II	No
BSCCO	104			
H2S @ 155 GPa	203		II	
LaH_{10} @ 150 GPa	250			

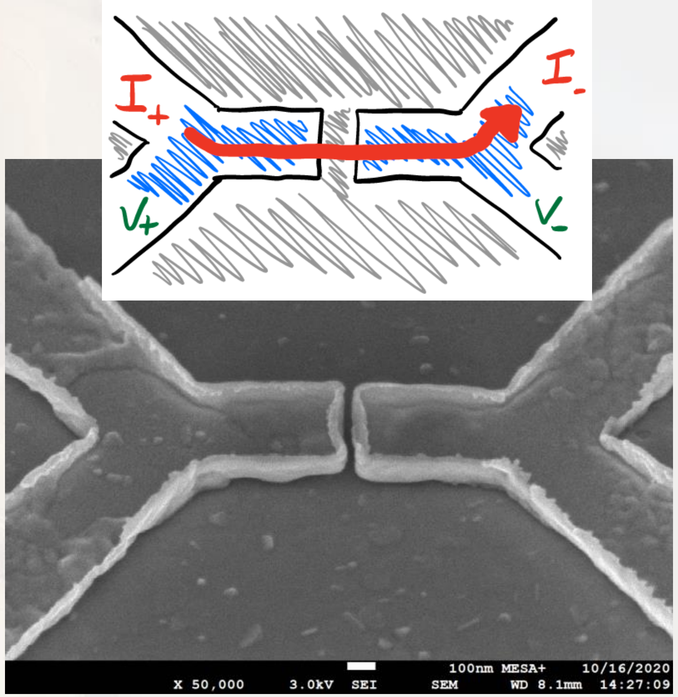
Why mK temperatures?

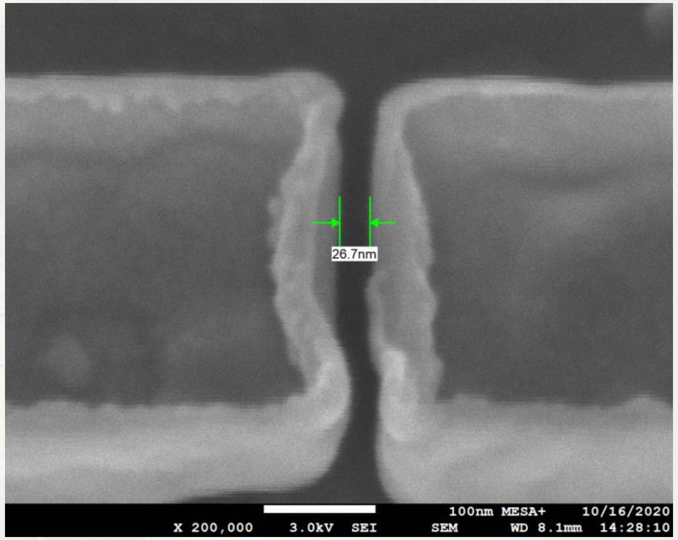
- Josephson junction devices



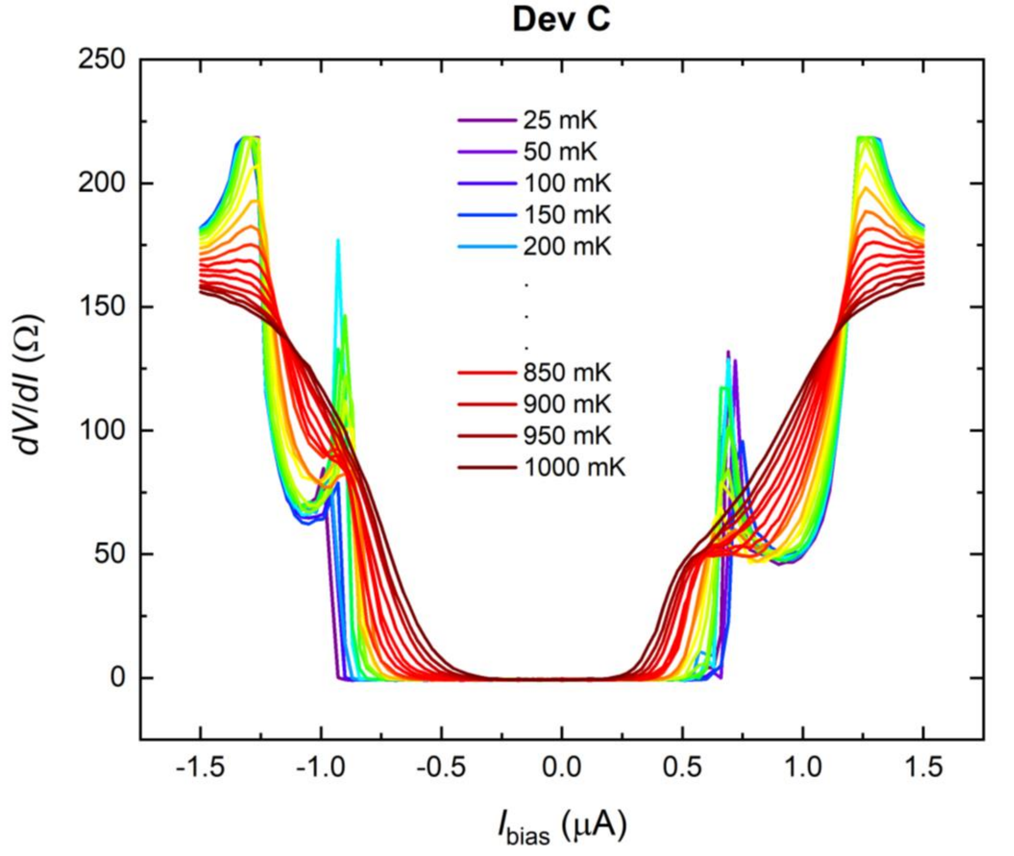
Why mK temperatures?

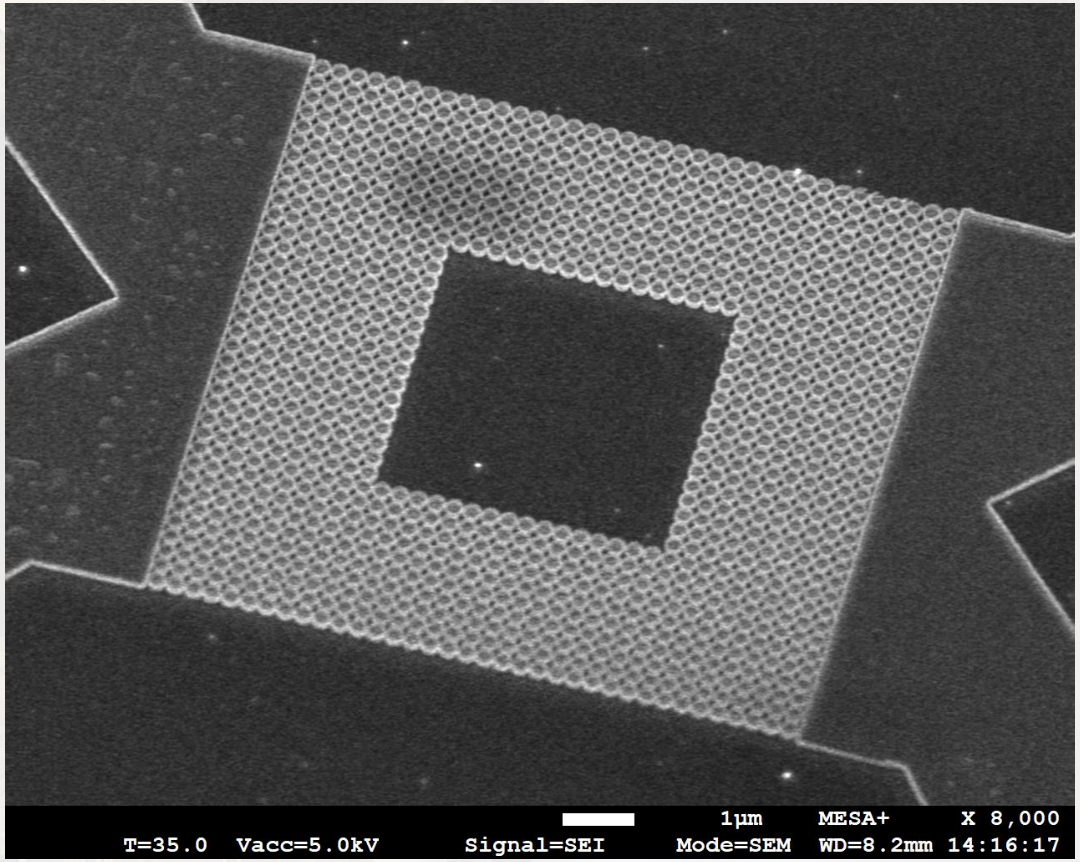






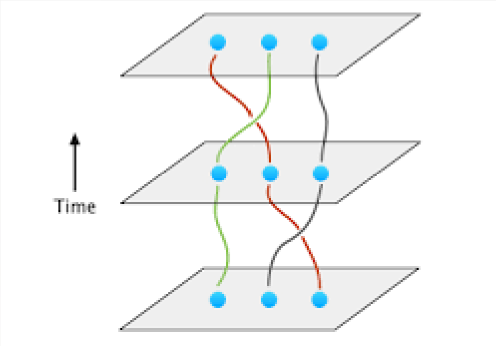
Why mK temperatures?

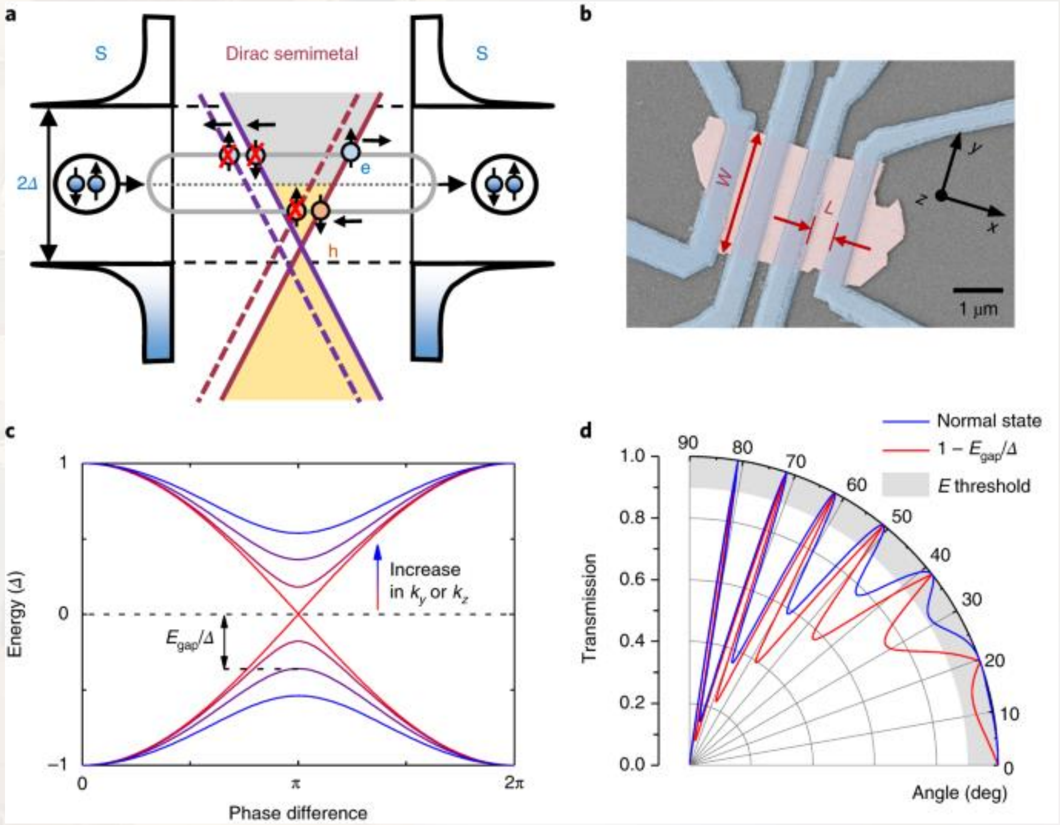




Why mK temperatures?

- Search for Majorana fermions
  - Andreev bound states (ABS)
  - Majorana bound states (MBS)
  - Energy phase relation: 2π vs 4π
- Use MBS for braiding
  - Quantum computing

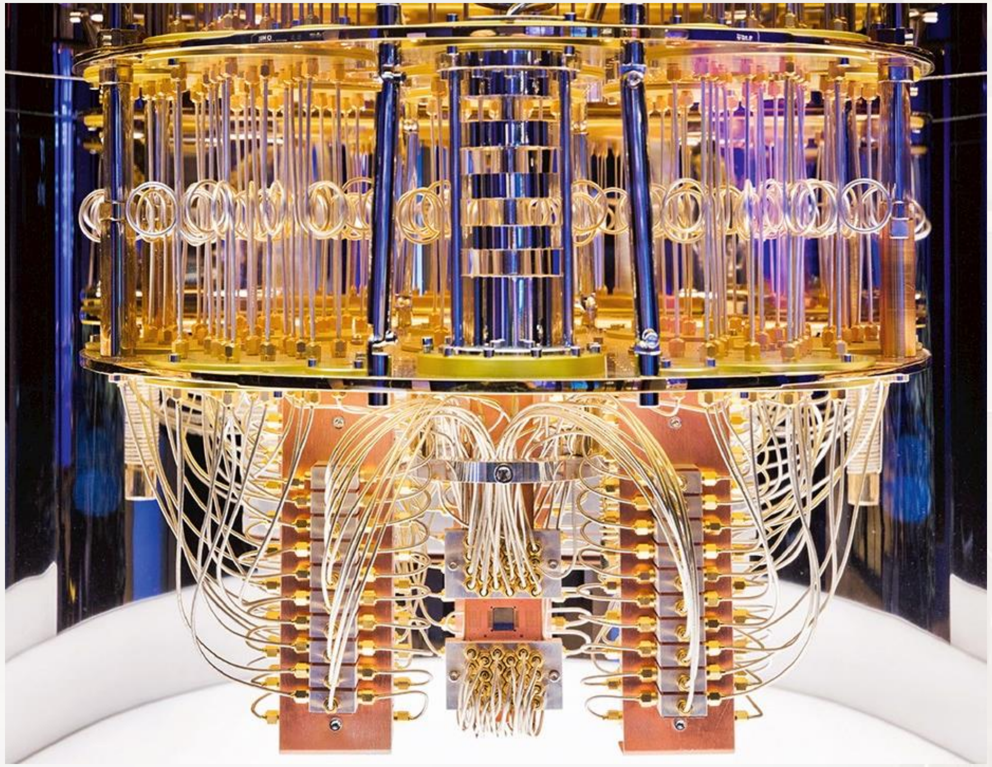


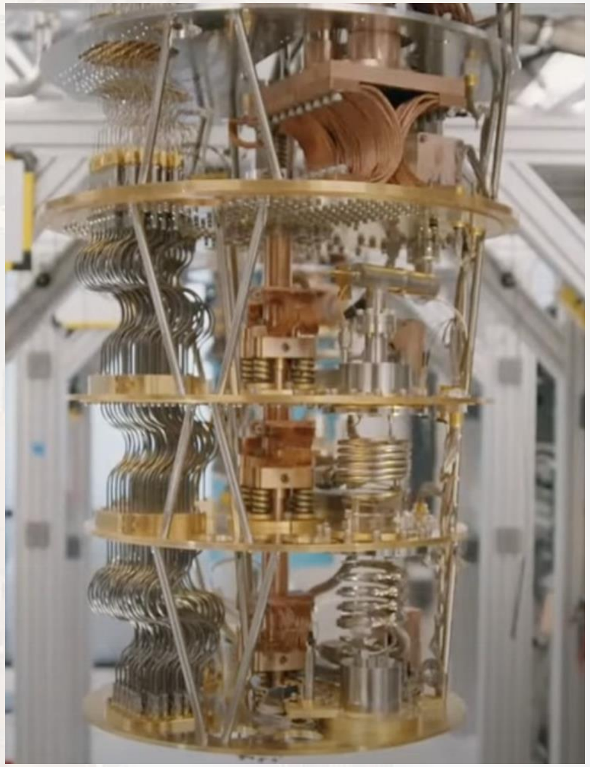


Nature Materials **volume 17**, 875–880 (2018)

Why mK temperatures?

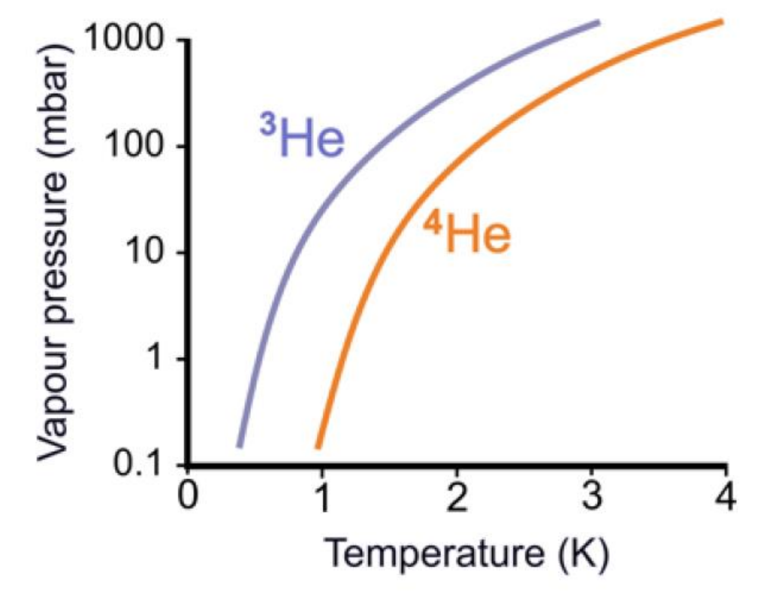
- Quantum computing
  - Heat → vibrating atoms
  - Vibration destroys entanglement of qubits

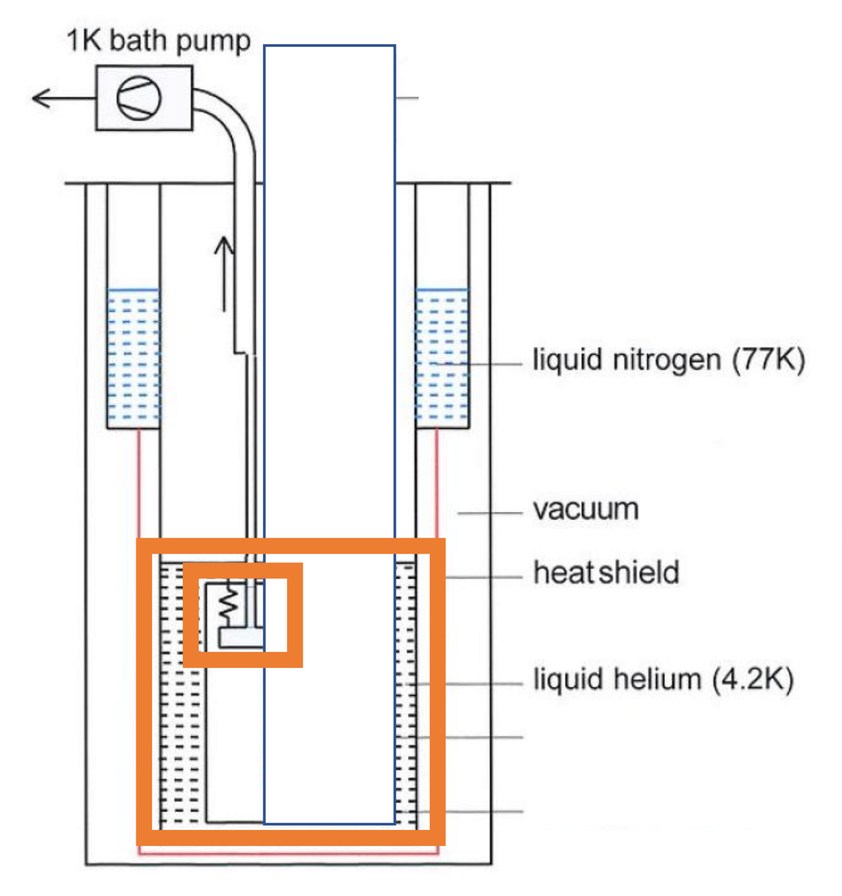




Wet fridges: basics up to 1 K

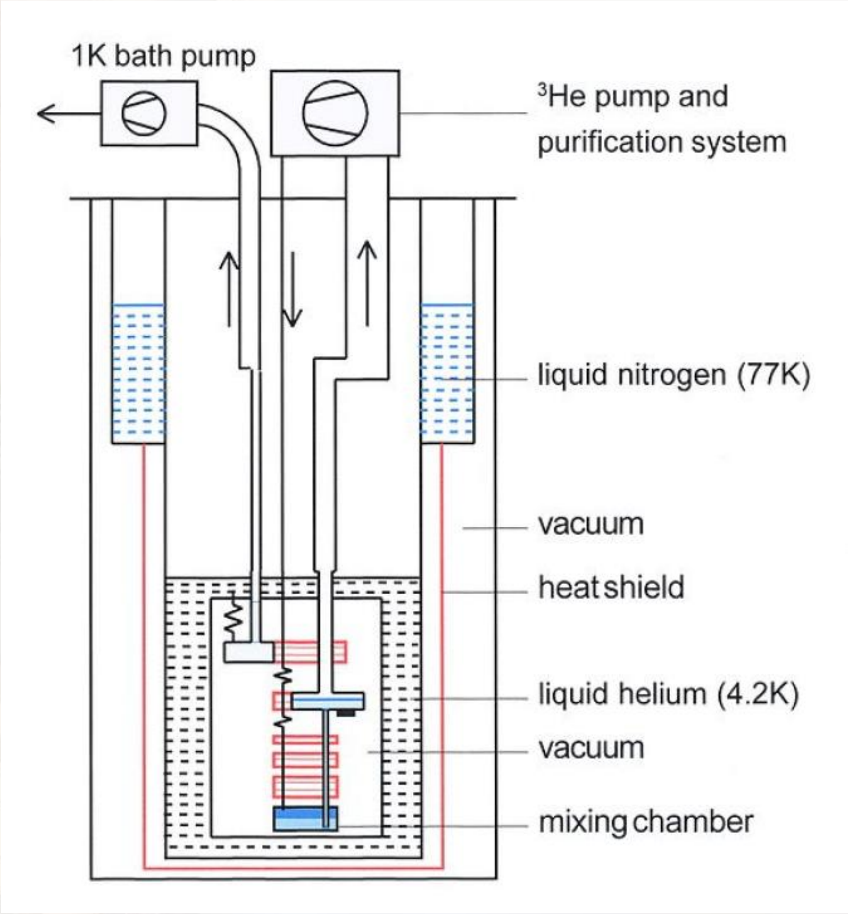
- Start with LHe bath
- Feed a small volume (the **pot**)
- Pump on the pot → reduce vapor pressure
    - ~ 1.2 K for ^{4}He
    - ~ 300 mK for ^{3}He





Wet fridges: basics up to 1 K

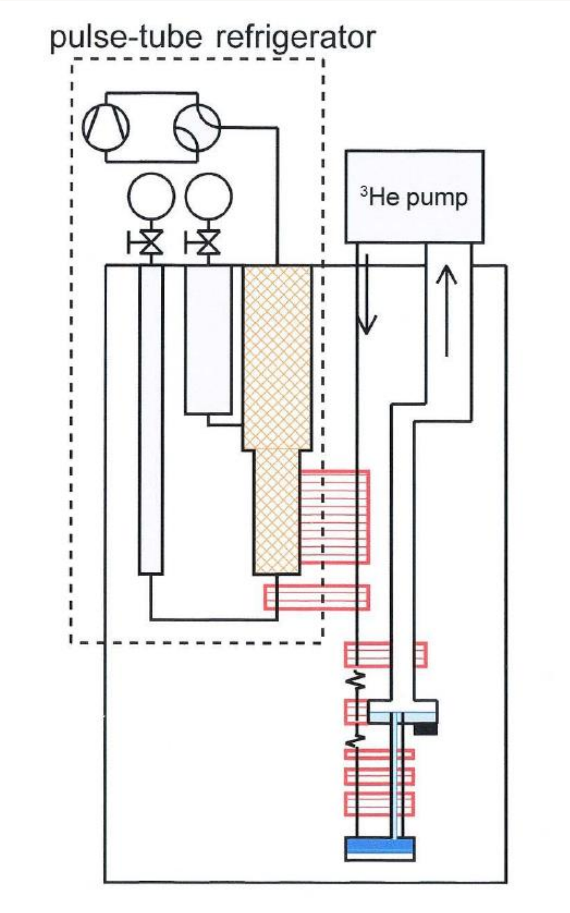
- Start with LHe bath
- Feed a small volume (the **pot**)
- Pump on the pot → reduce vapor pressure
- 1 K pot precools/liquefies mixture



Dry fridges: basics up to 1 K

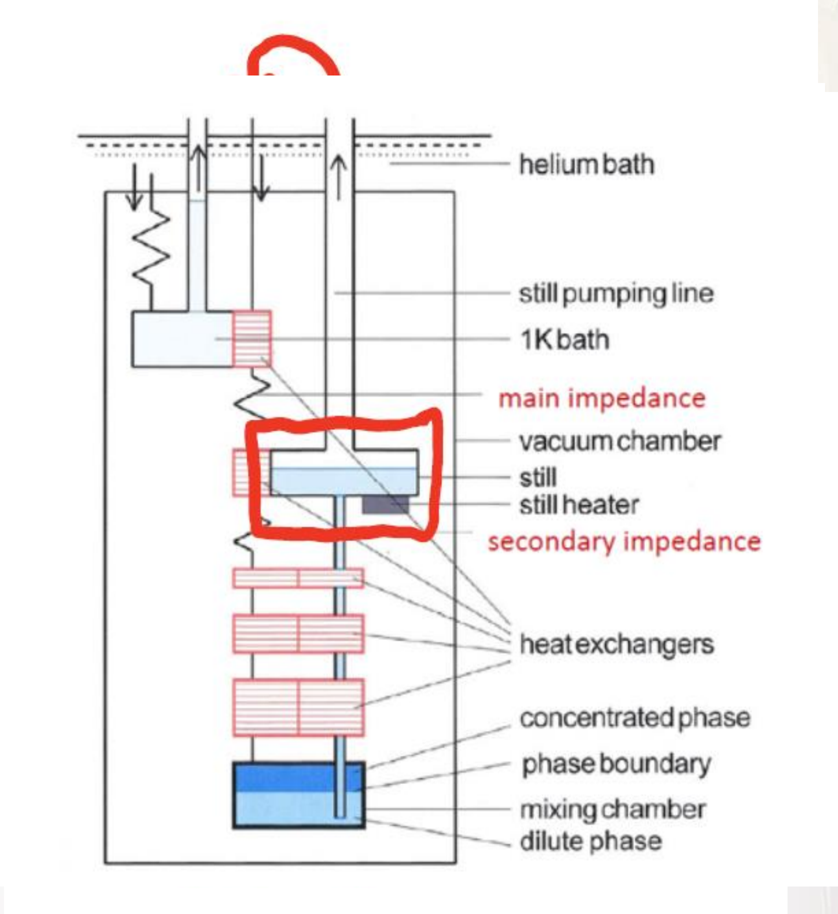
- Use pulse tube refrigerator (PTR)
  - First stage: 70 K, 50 W cooling power
  - Second stage: 4 K, 1-1.5 W cooling power
  - Uses 9 kW compressor…

  - Why PTR? Low vibrations: typically 5-10 um
- No need for costly LHe (bath), no top-ups required!

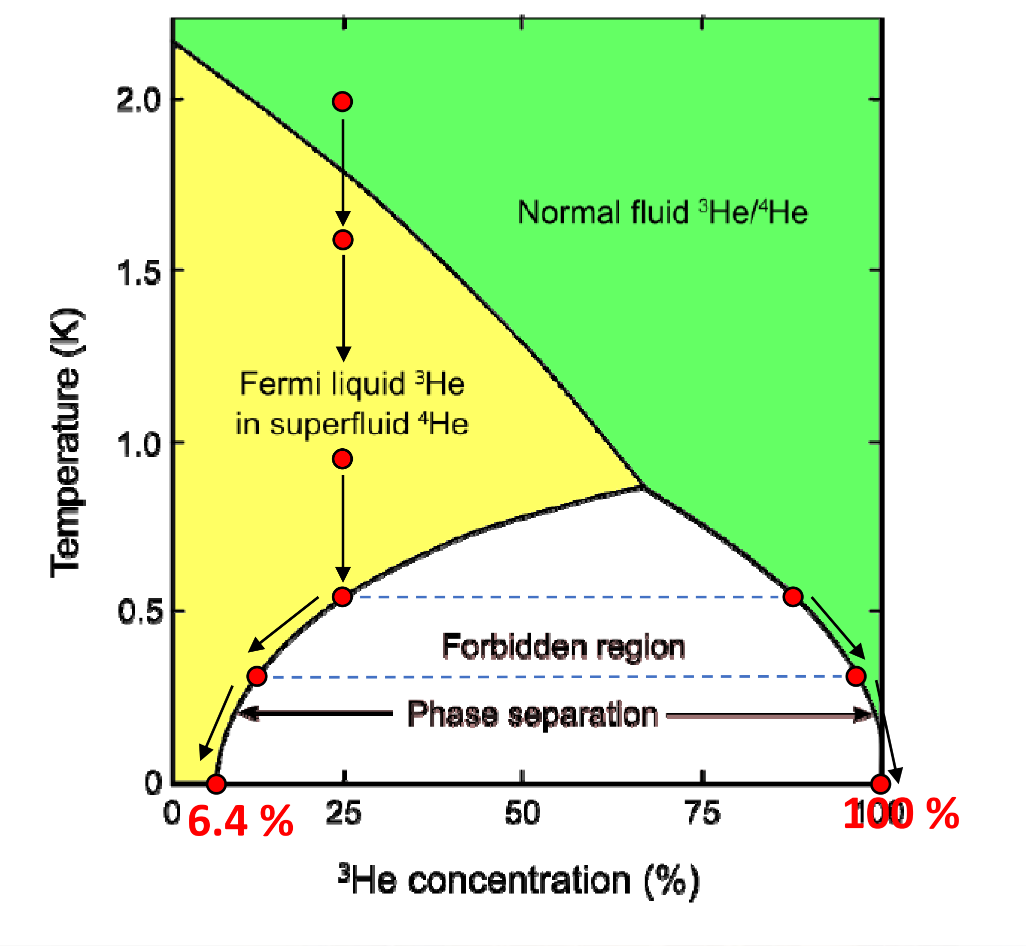


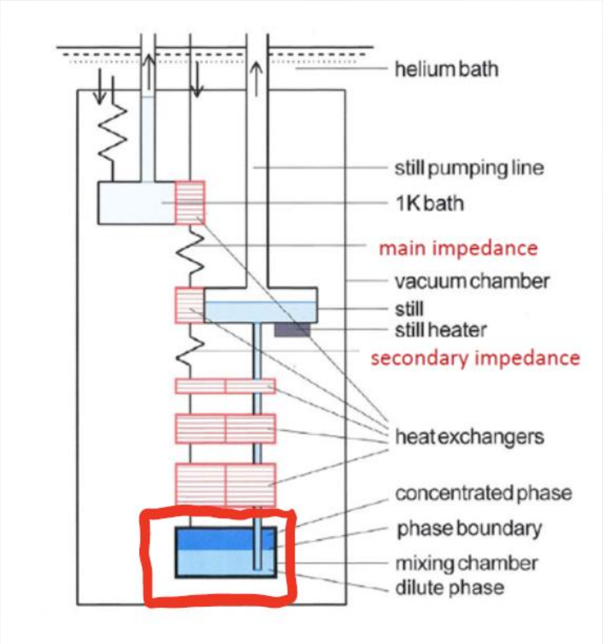
Dilution unit

- Let’s assume we have a working fridge **running at base temperature**



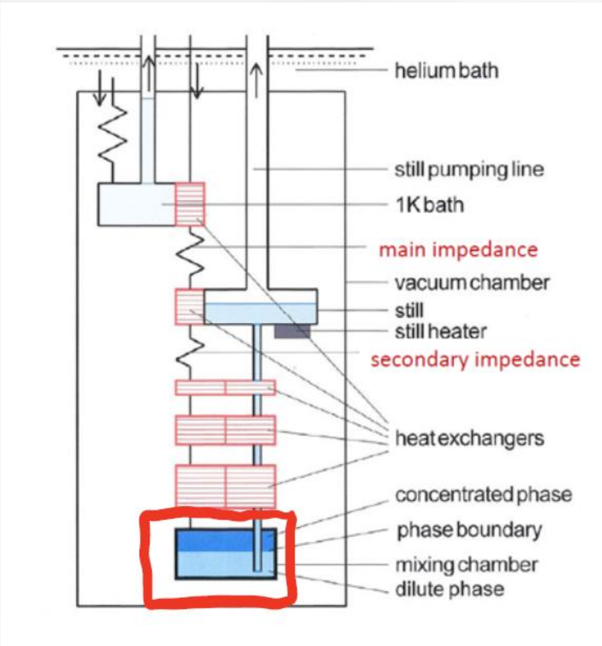
Dilution unit: mixing chamber

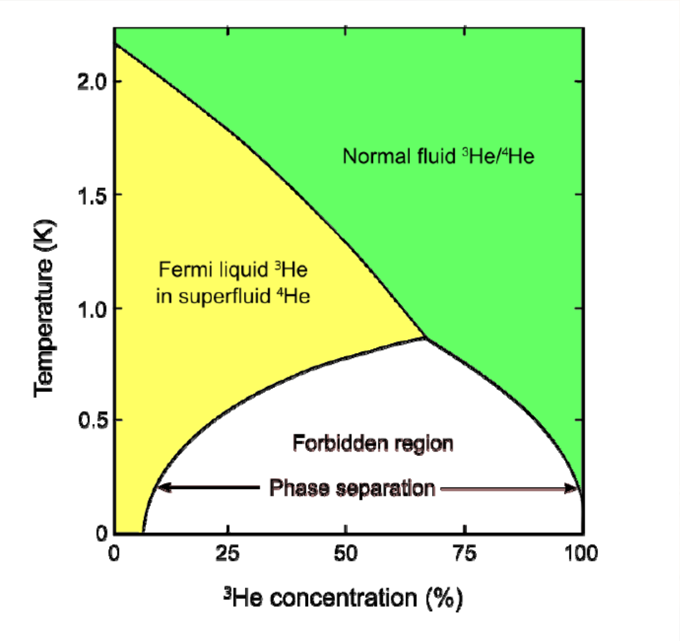


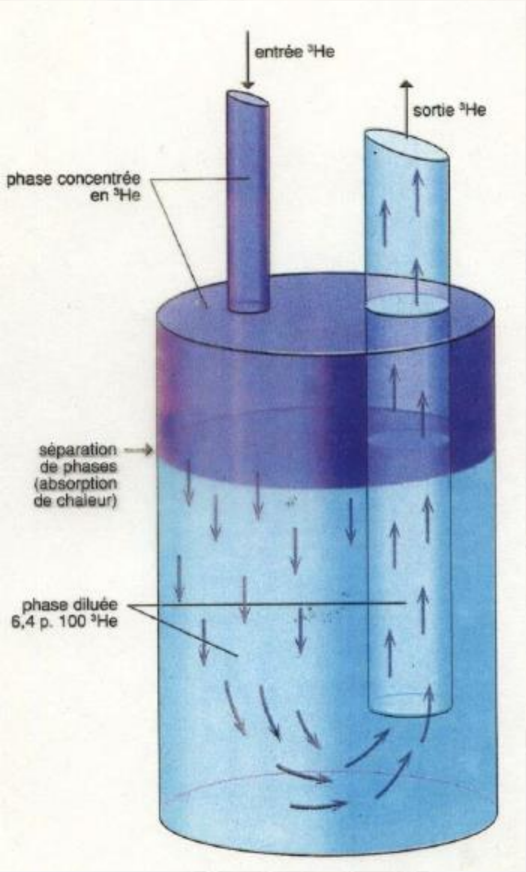


Dilution unit: mixing chamber

- Recall phase separation diagram
  - Phase separation at low temperatures
  - ^{3}He-rich phase is lighter than diluted phase, so it floats on top







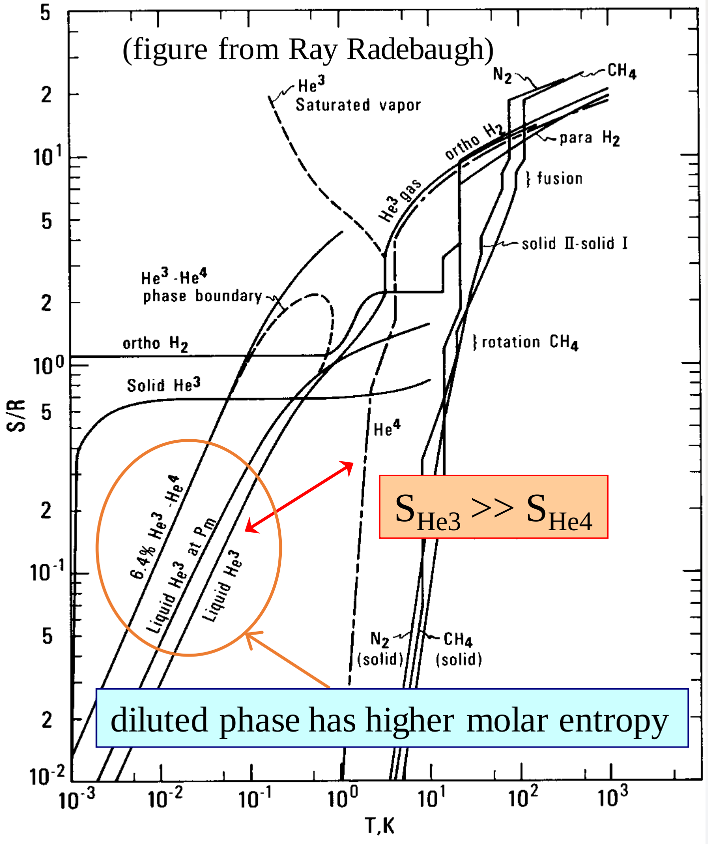
Dilution unit: mixing chamber

- What happens when ^{3}He crosses the phase boundary?
  - If ^{3}He goes from rich phase into diluted phase, its entropy increases → cooling

$$\dot{Q} = \dot{n}_3(95T_{\mathrm{mc}}^2 - 11T_{\mathrm{ex}}^2)$$

where

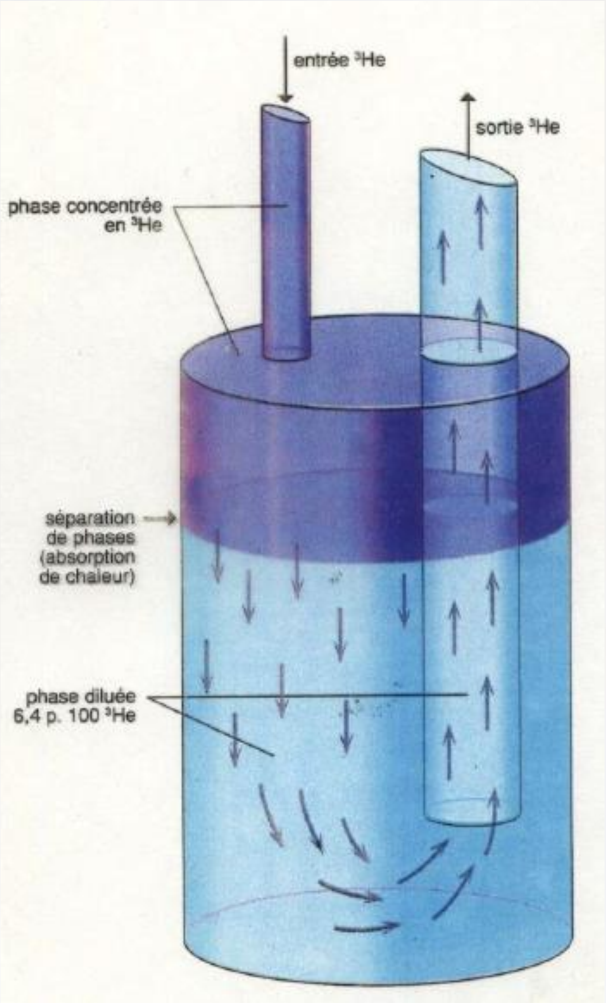
$\dot{Q}$ = cooling power
$\dot{n}_3$ = flow rate of ^{3}He
T_{mc} = mixing chamber temperature
T_{ex} = last heat exchanger temperature



(figure from Ray Radebaugh)

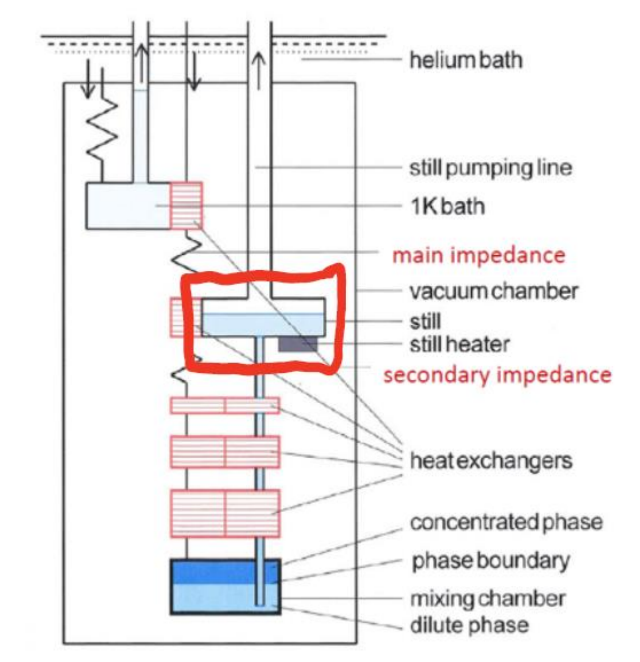
$S_{He3} >> S_{He4}$

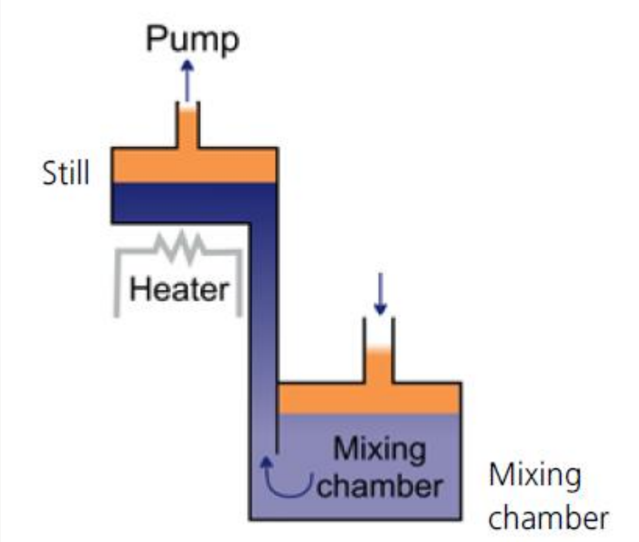
diluted phase has higher molar entropy

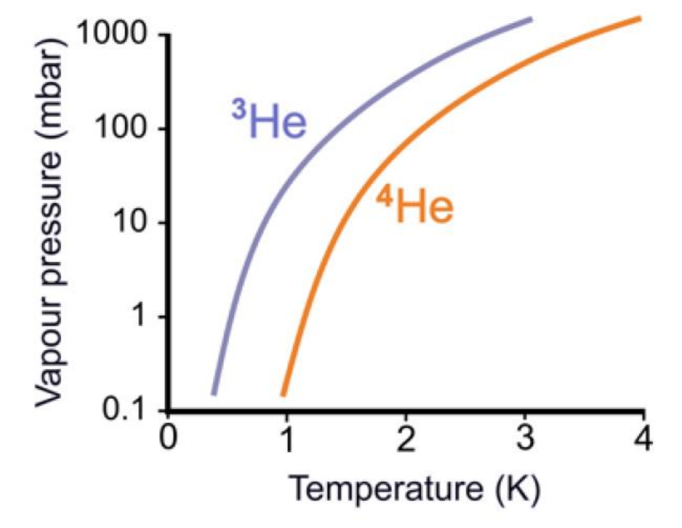


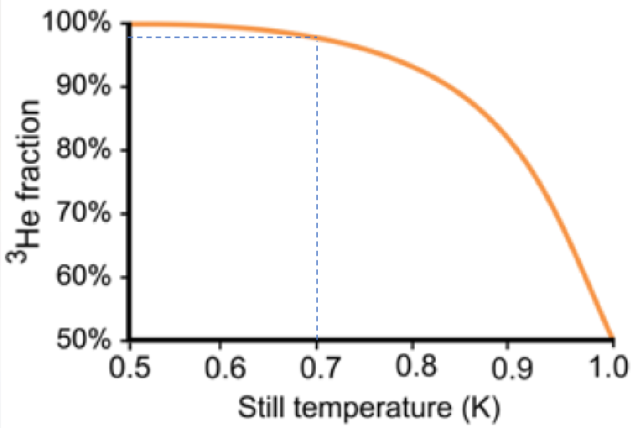
Dilution unit: still

- How to circulate 3He?
  - Use a distiller
    - Pump on exhaust of MC
    - Difference in vapor pressure will lead to evaporation of 3He only
  - Still too low: no vapor → no circulation
  - Still too high: vapor contains more 4He → less efficient DR



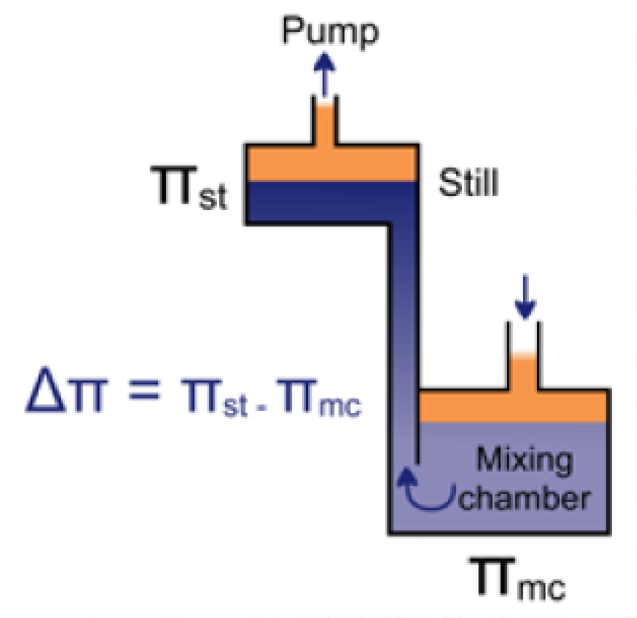






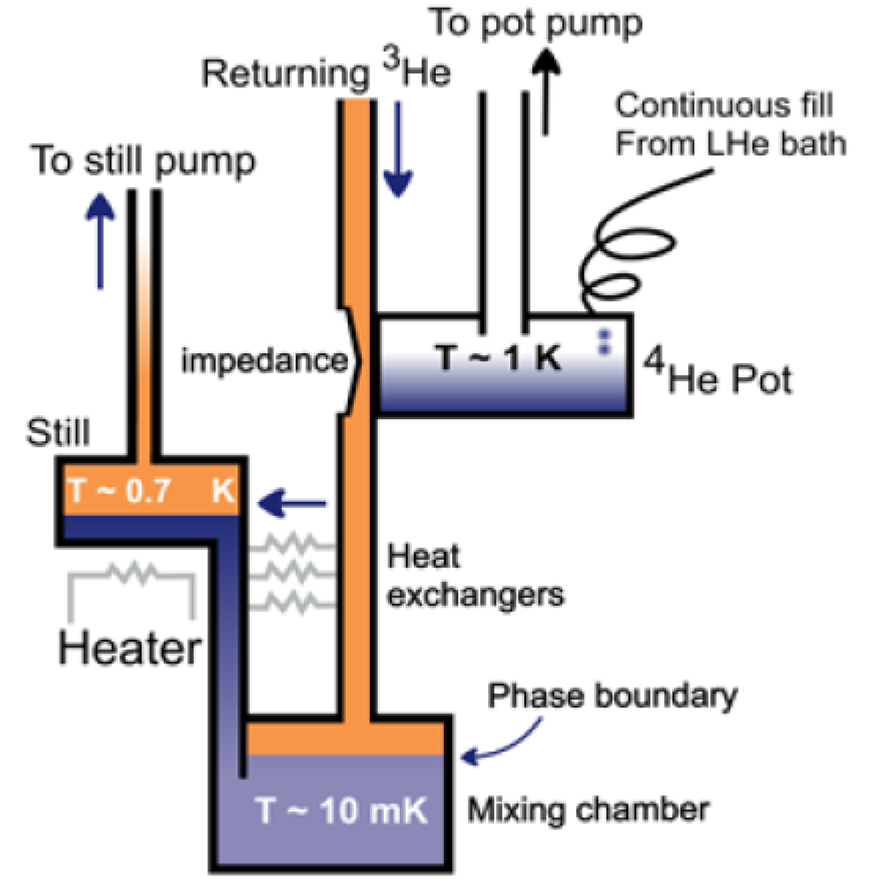
Dilution unit: still

- How to get 3He through phase boundary?
  - Make use of osmotic pressure gradient

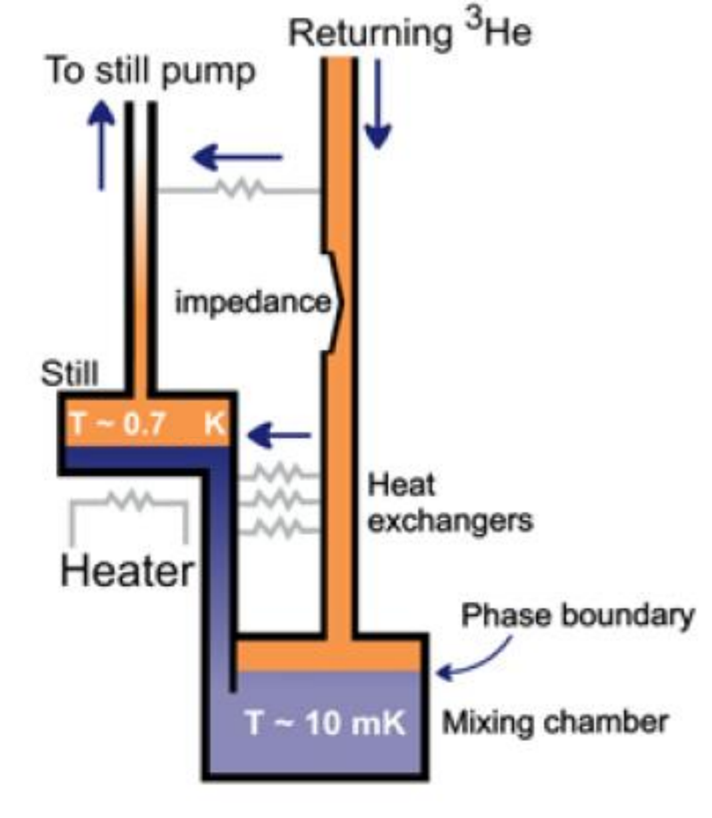


Dilution unit: wet vs dry

Wet fridge



Dry fridge



Wet fridge:

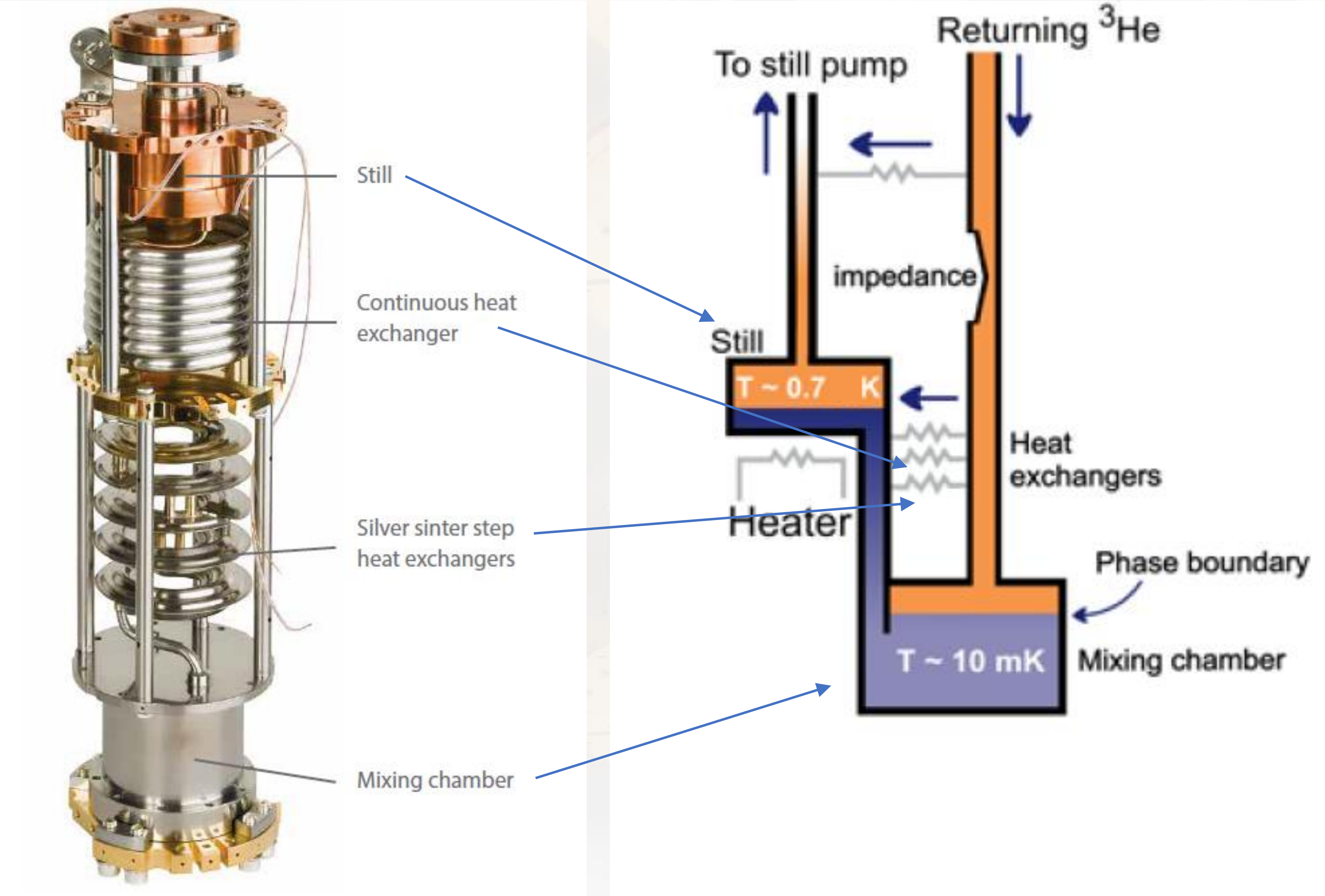
- Incoming 3He with 0.3 bar liquefies at 1 K pot

Dry fridge:

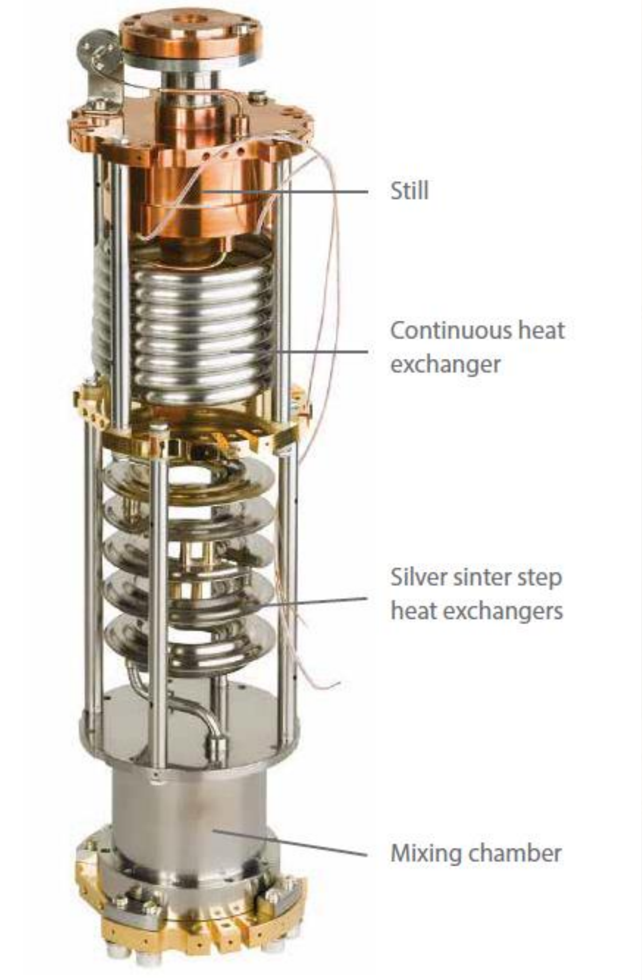
- No 1 K pot, so extra heat exchanger
- Use high pressure (2.5 bar) to liquefy mixture

Dilution unit: dry unit (Oxford Instruments)

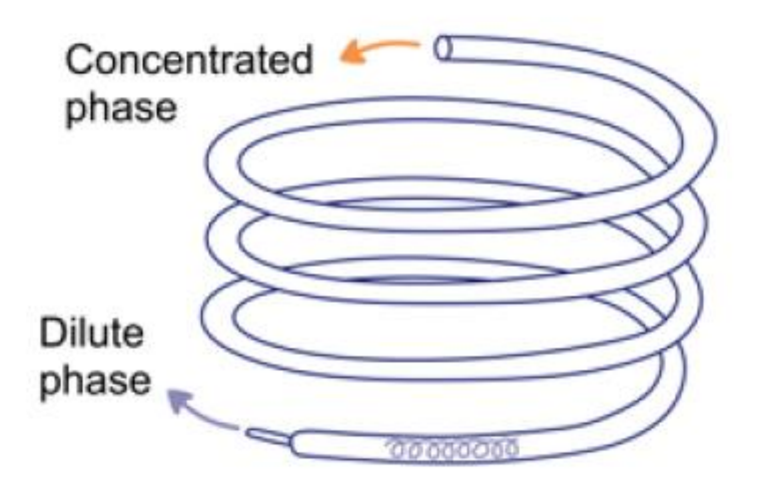
Dry fridge



Dilution unit: dry

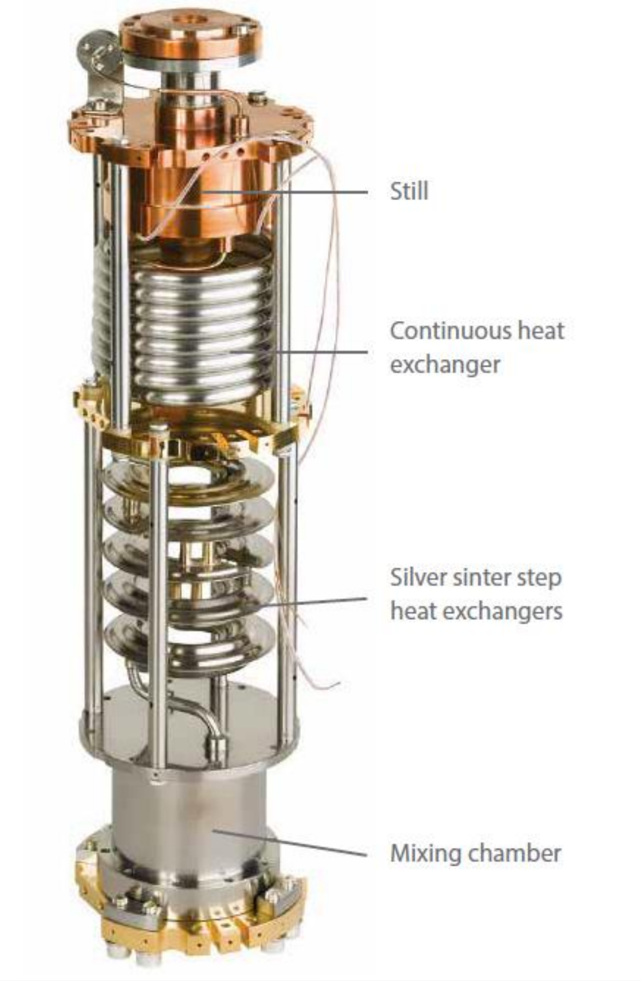


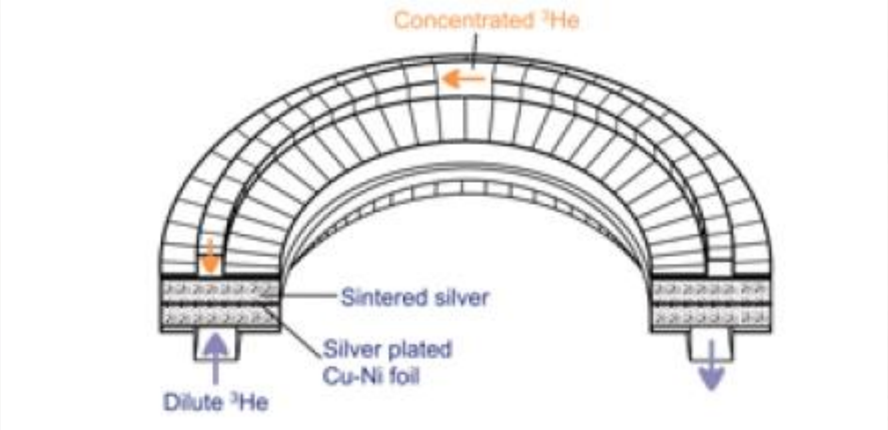
‘High’ temperature part (> 30 mK) : continuous tube-in-tube heat exchanger



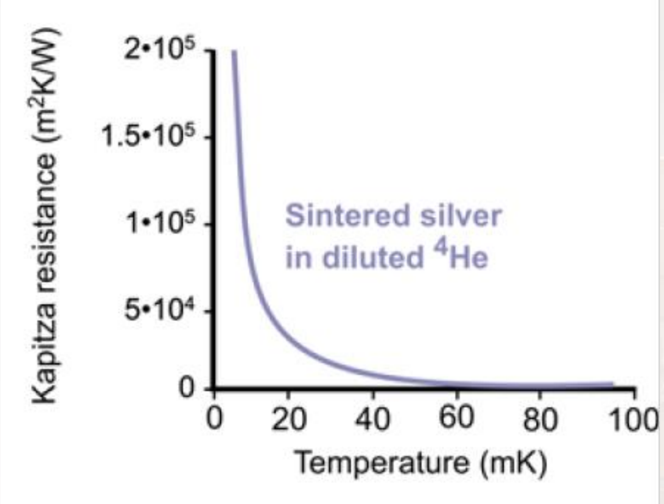
Dilution unit: dry

‘Low’ temperature part : sintered silver heat exchanger. Why?

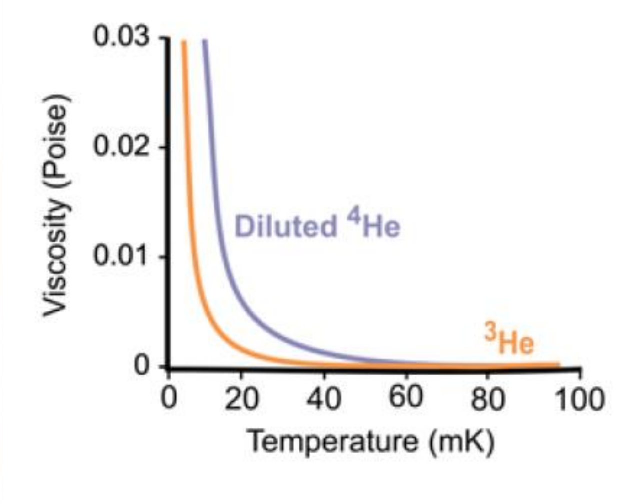




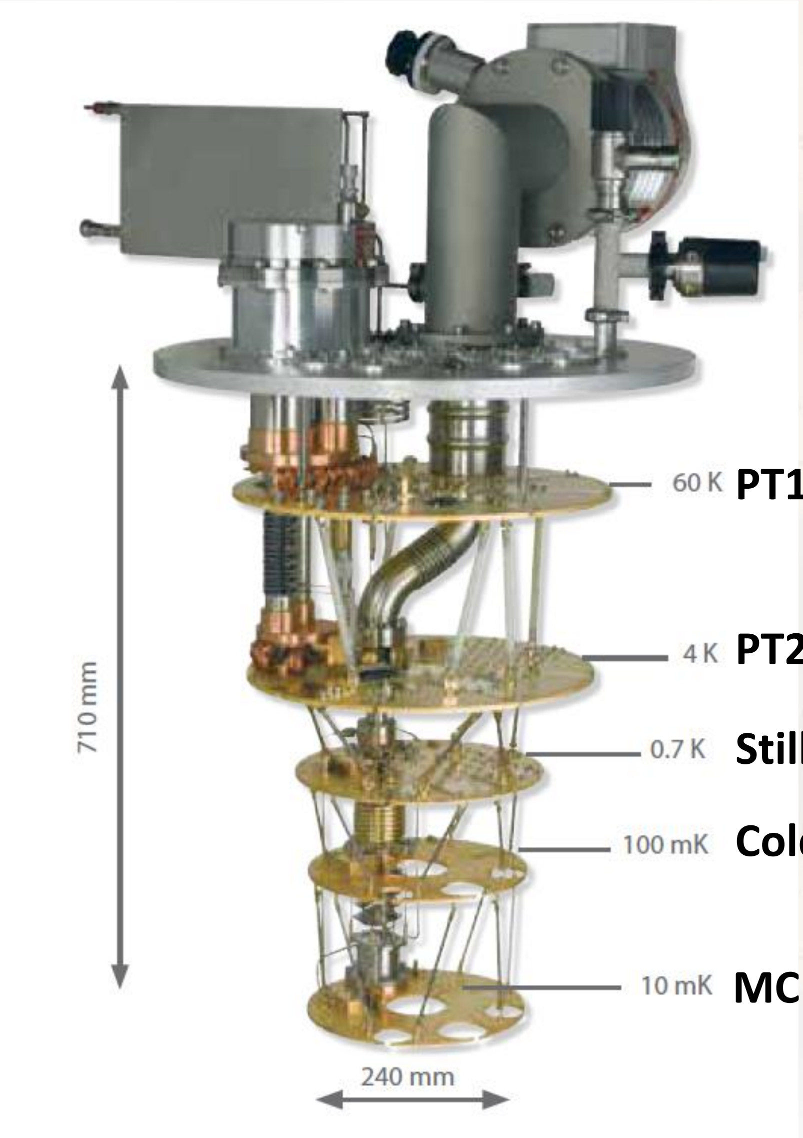
1. Thermal conductivity drops, so need larger surface area



2. Viscosity increased, so friction should be minimized: larger channels



Dry dilution fridge



PT1 plate

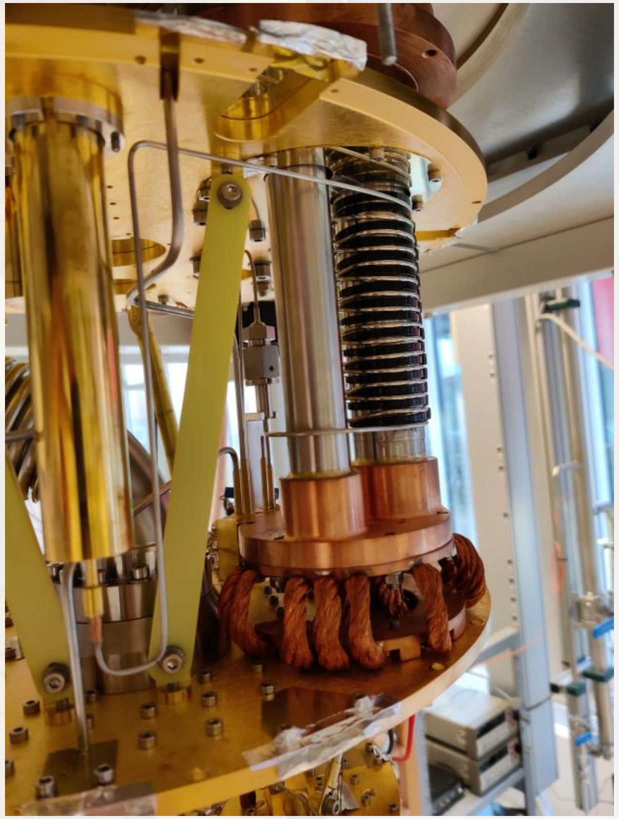
PT2 plate

Still plate

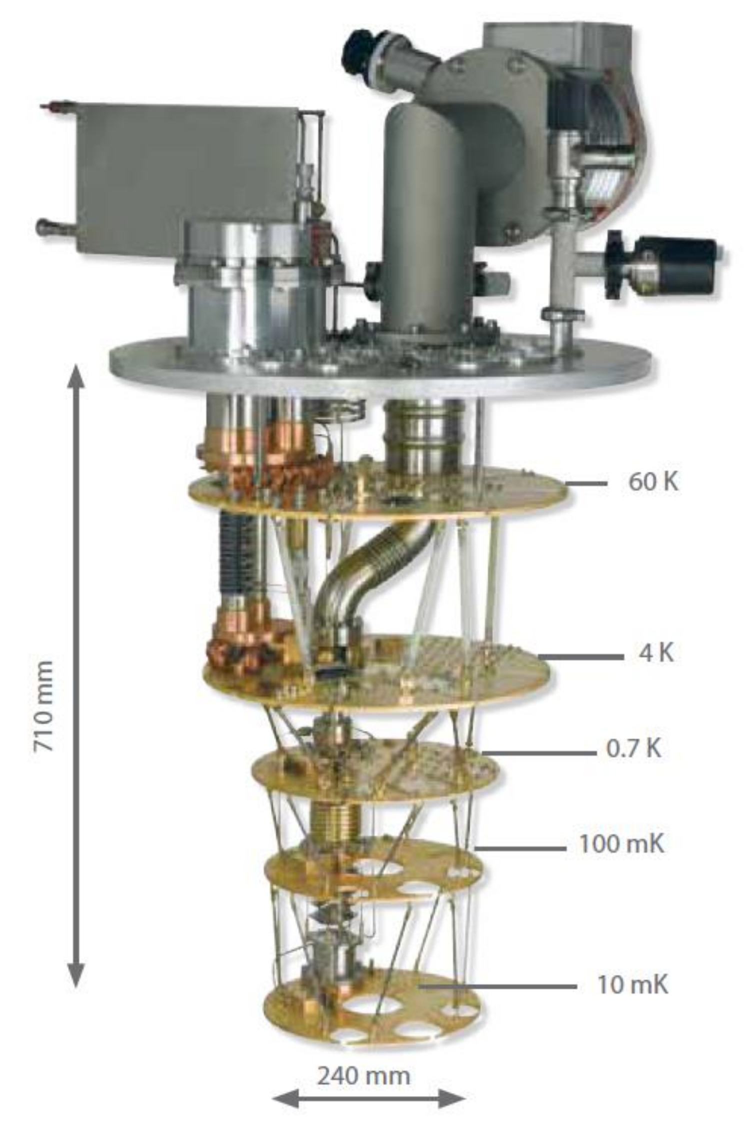
Cold plate

MC plate

- PTR cools first and second plates (PT1 at 60 K, PT2 at 4 K)
  - PTR is connected via copper braids to reduce vibrations to < 100 nm

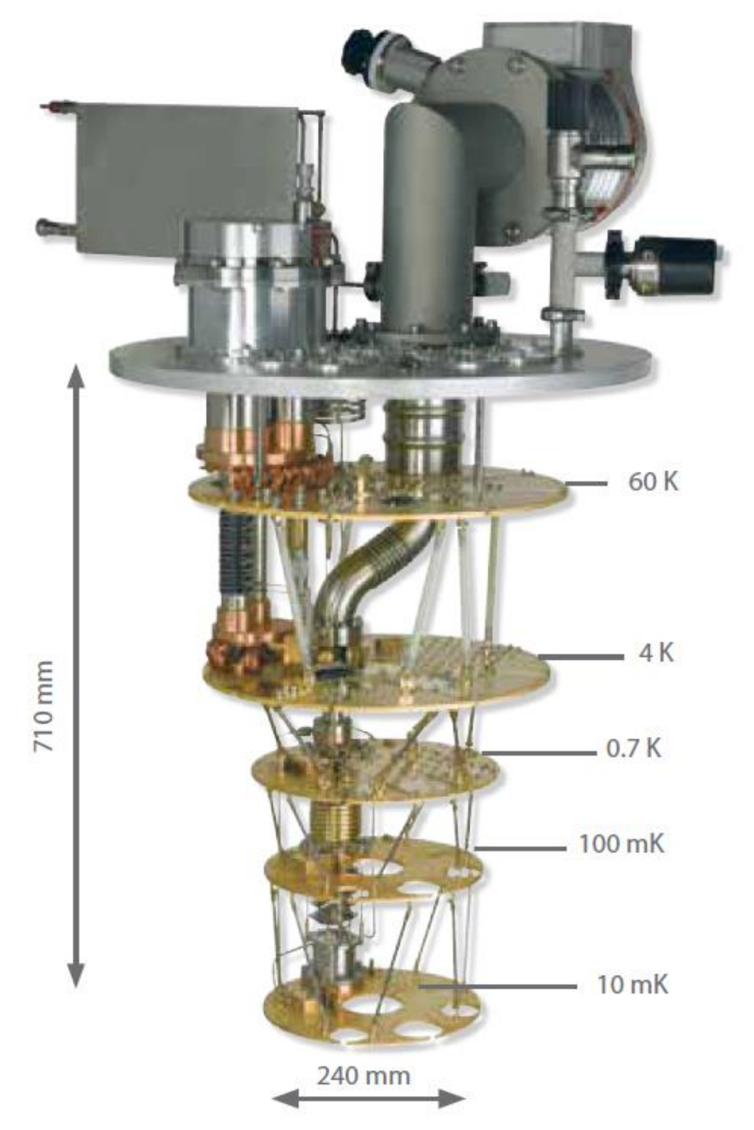


Dry dilution fridge: how to go from RT to mK?

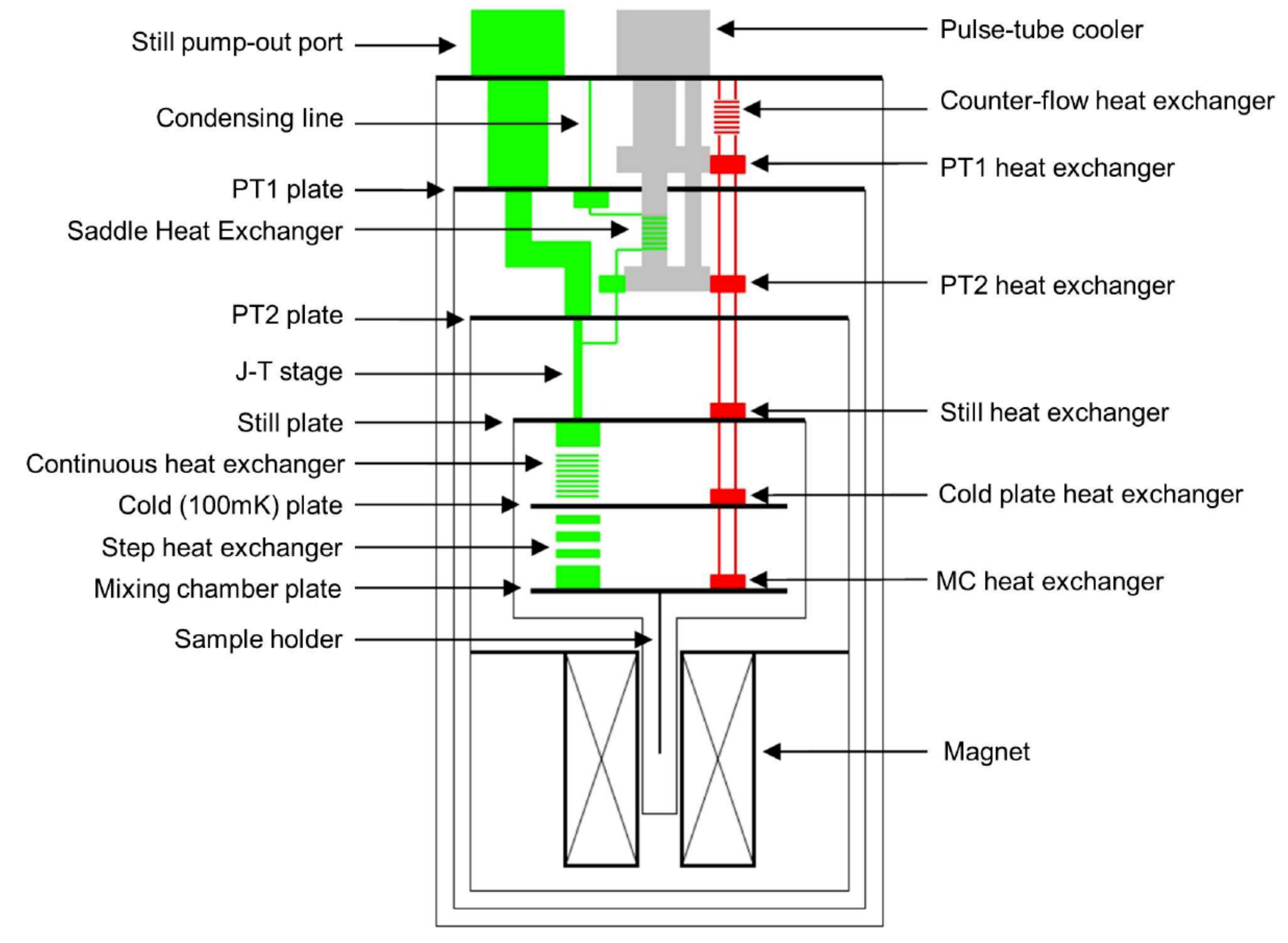


- PTR only cools PT1 and PT2 plates…
- Thermal link from these plates to other plates creates massive heat leak to 10 mK plate…

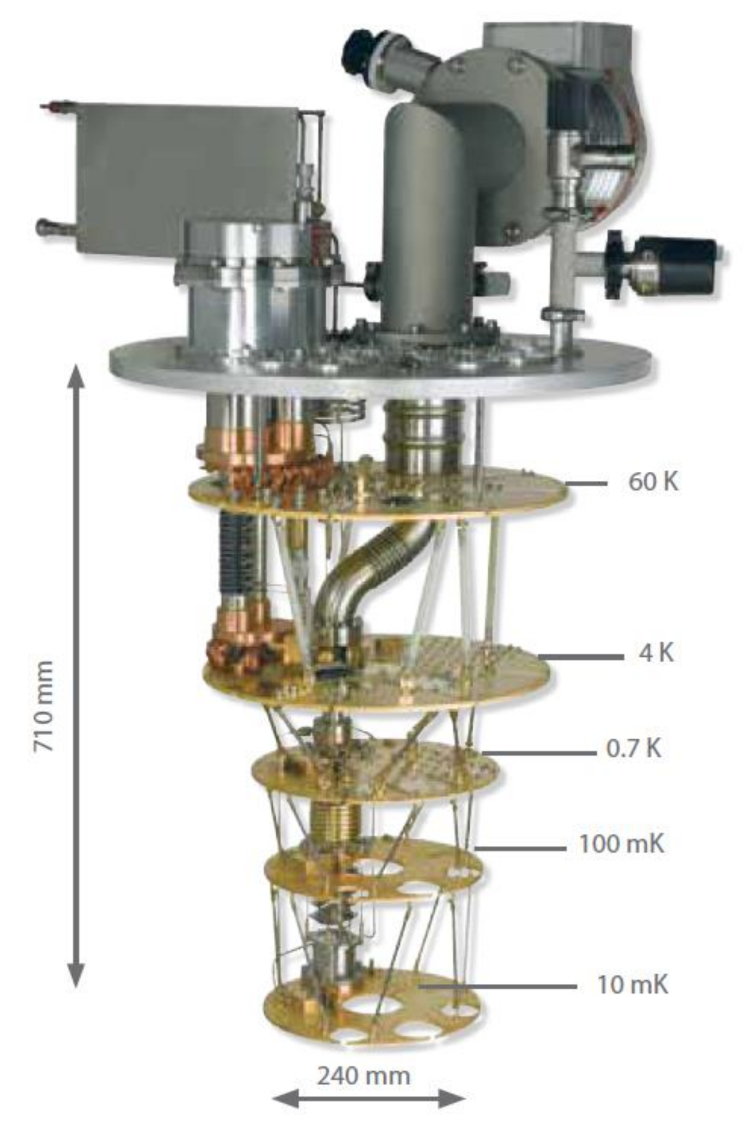
Dry dilution fridge: how to go from RT to mK?



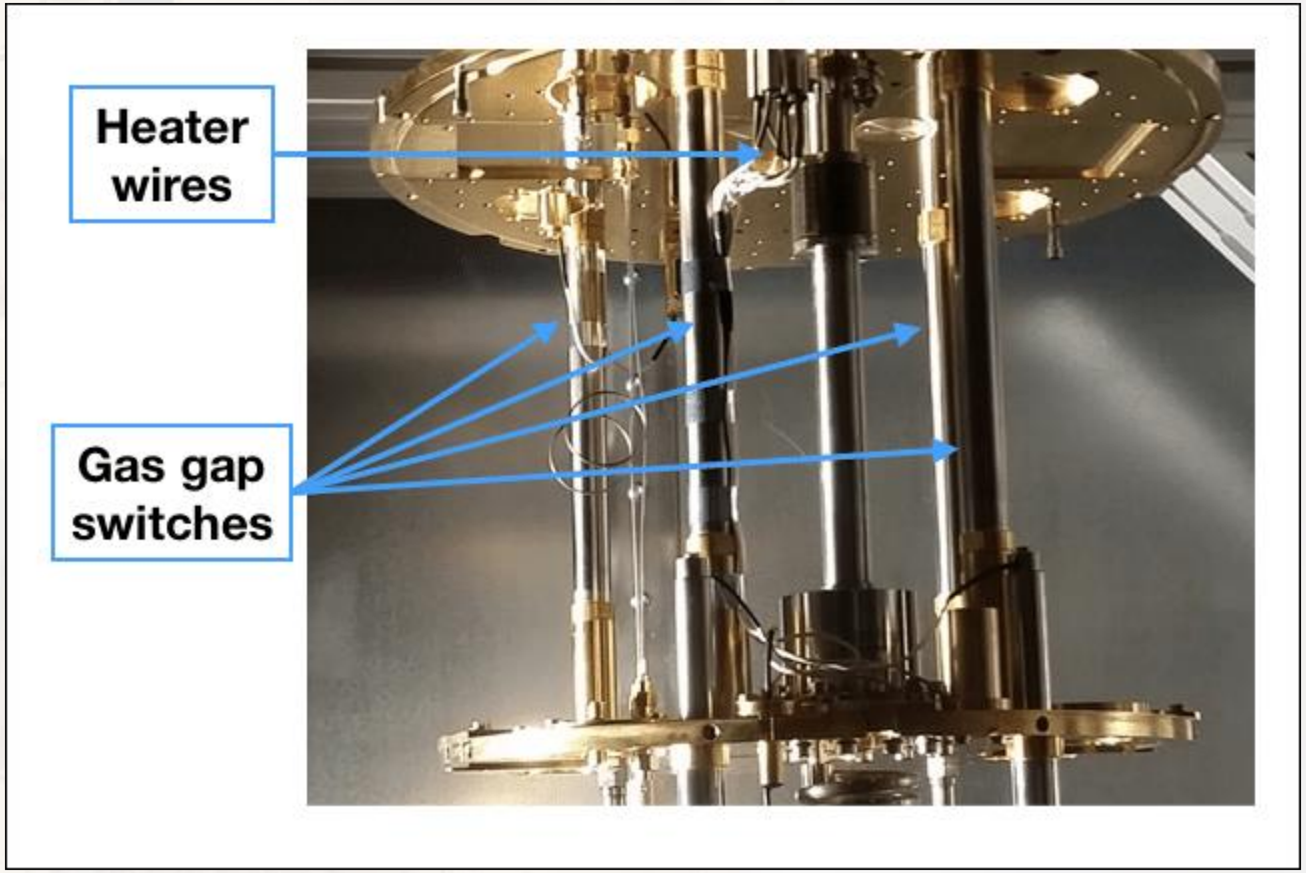
Oxford Instruments:



Dry dilution fridge: how to go from RT to mK?



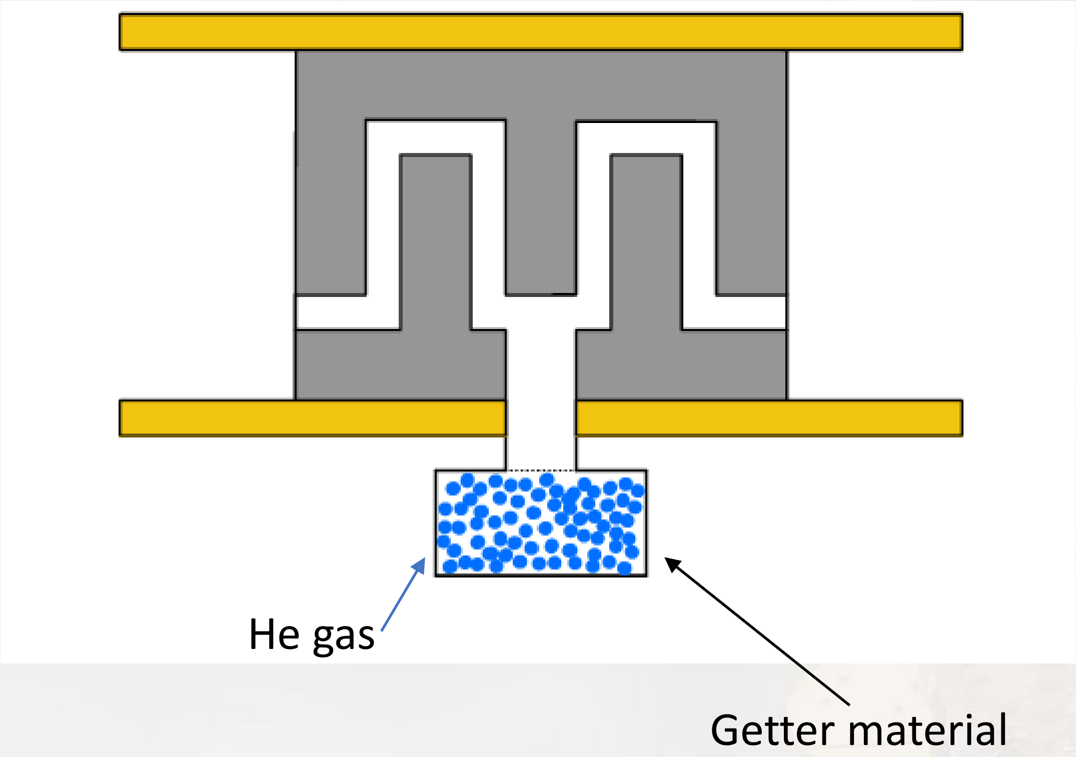
Bluefors:



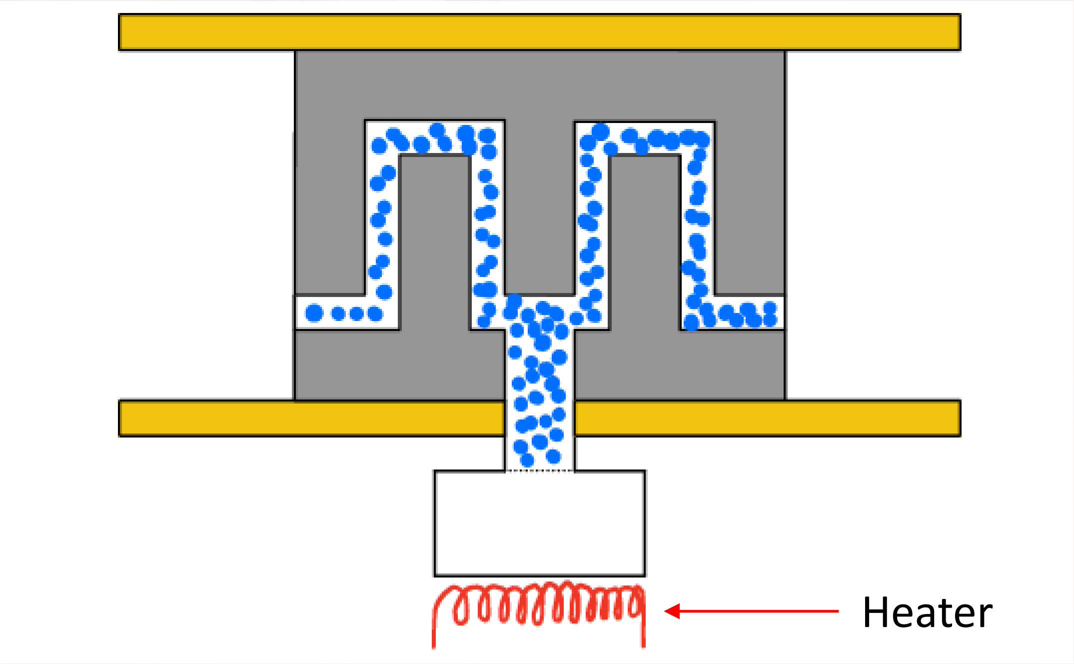
Dry dilution fridge: how to go from RT to mK?

Bluefors: gas gap switch

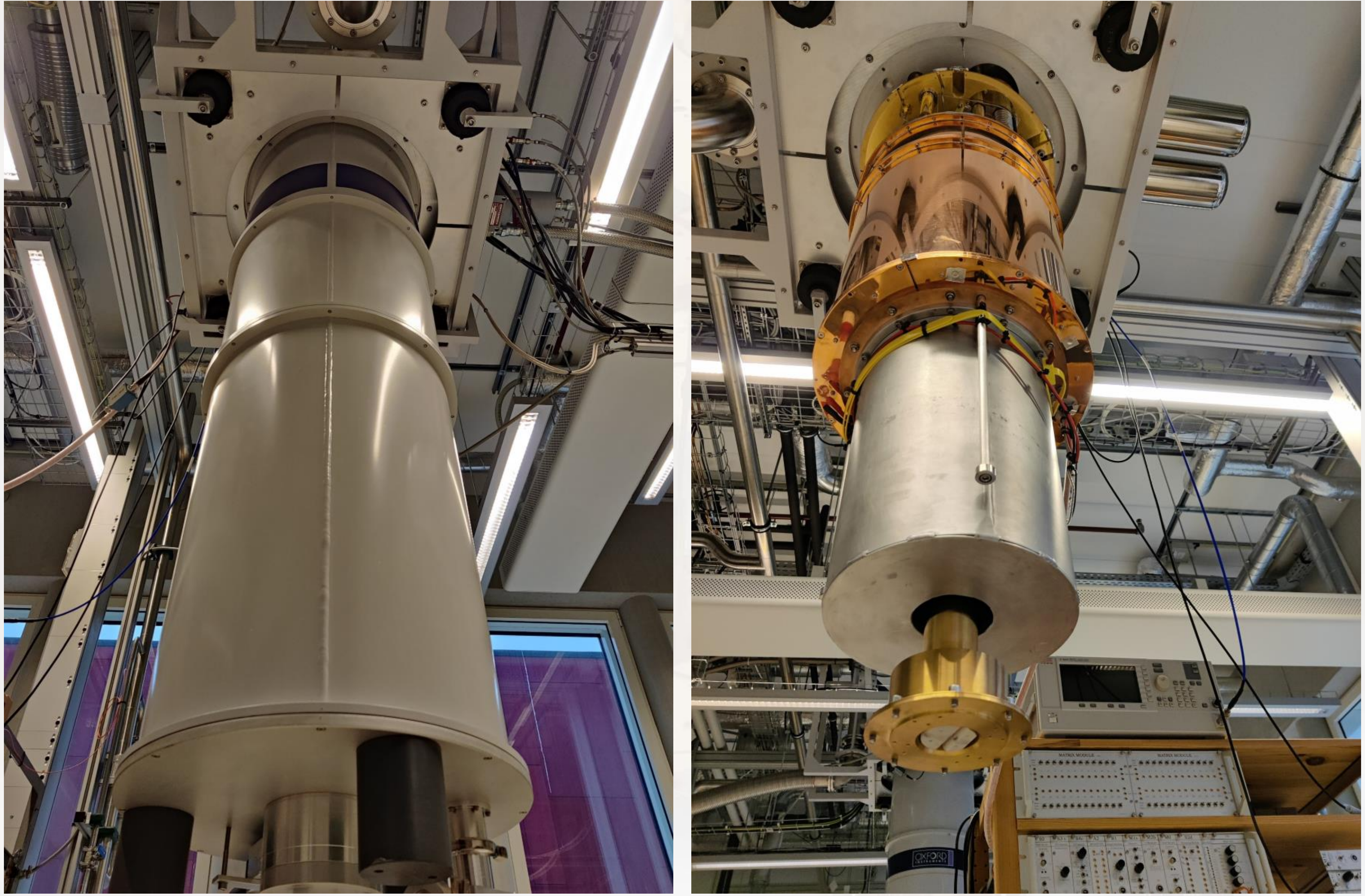
Heater off: getter pumps gas from switch → no thermal link



Heater on: getter releases gas into switch → thermal link



Dry dilution fridge (our lab):



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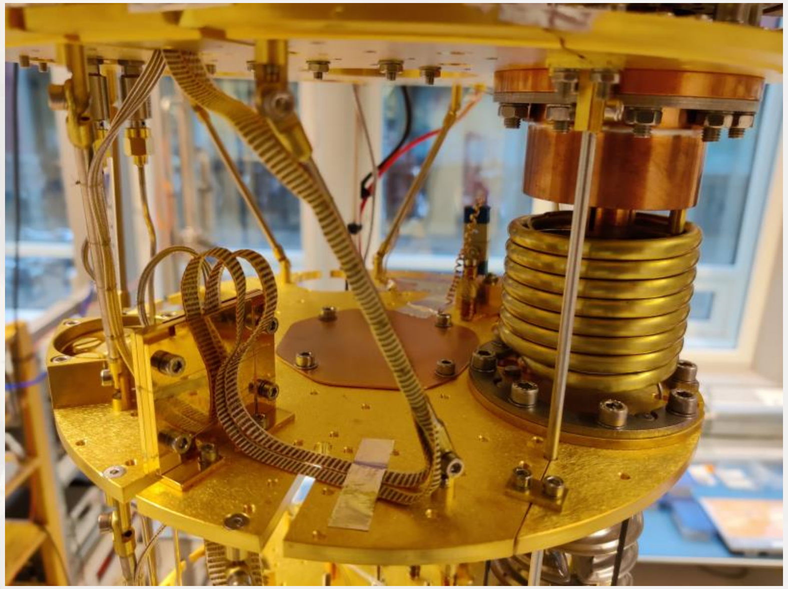
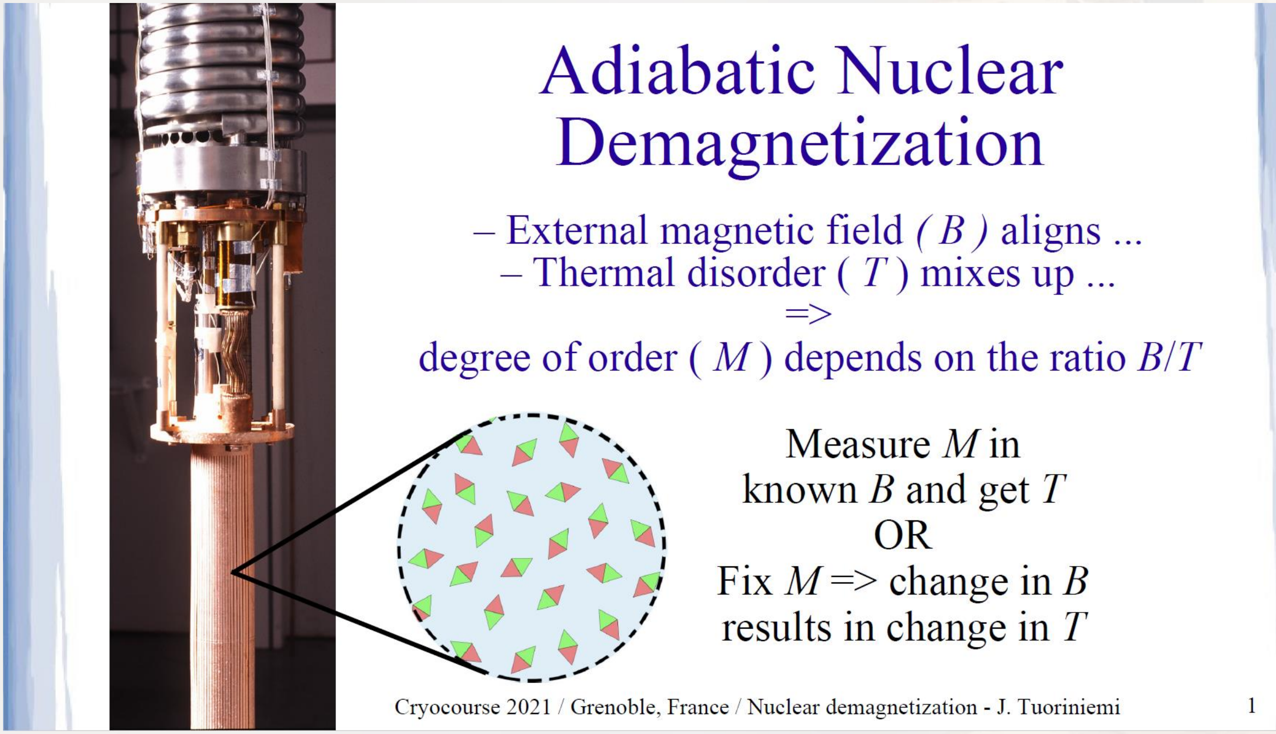


Can we go lower?



Adiabatic Nuclear Demagnetization

– External magnetic field (B) aligns ...
– Thermal disorder (T) mixes up ...
=>
degree of order (M) depends on the ratio B/T

Measure M in known B and get T
OR
Fix M => change in B results in change in T

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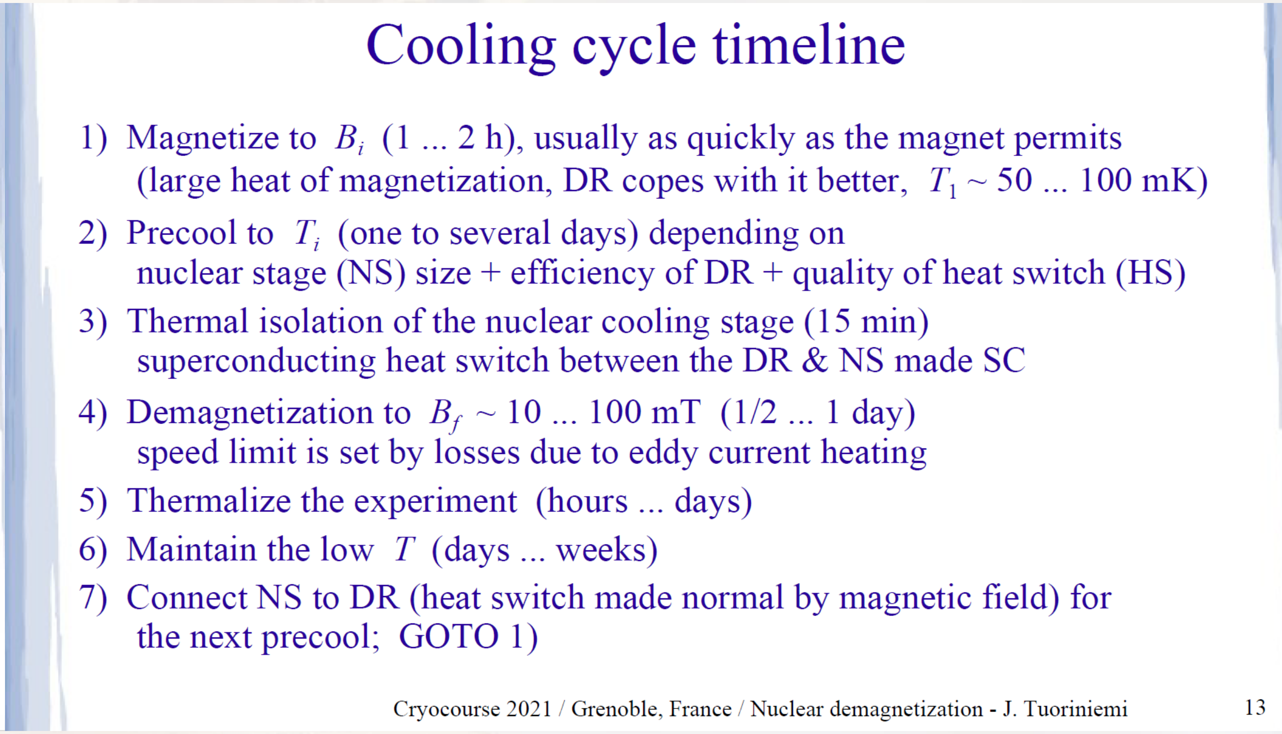

Cooling cycle timeline

1) Magnetize to B_i (1 ... 2 h), usually as quickly as the magnet permits (large heat of magnetization, DR copes with it better, $T_1 \sim 50 \ldots 100$ mK)
2) Precool to T_i (one to several days) depending on nuclear stage (NS) size + efficiency of DR + quality of heat switch (HS)
3) Thermal isolation of the nuclear cooling stage (15 min) superconducting heat switch between the DR & NS made SC
4) Demagnetization to $B_f \sim 10 \ldots 100$ mT (1/2 ... 1 day) speed limit is set by losses due to eddy current heating
5) Thermalize the experiment (hours ... days)
6) Maintain the low T (days ... weeks)
7) Connect NS to DR (heat switch made normal by magnetic field) for the next precool; GOTO 1)

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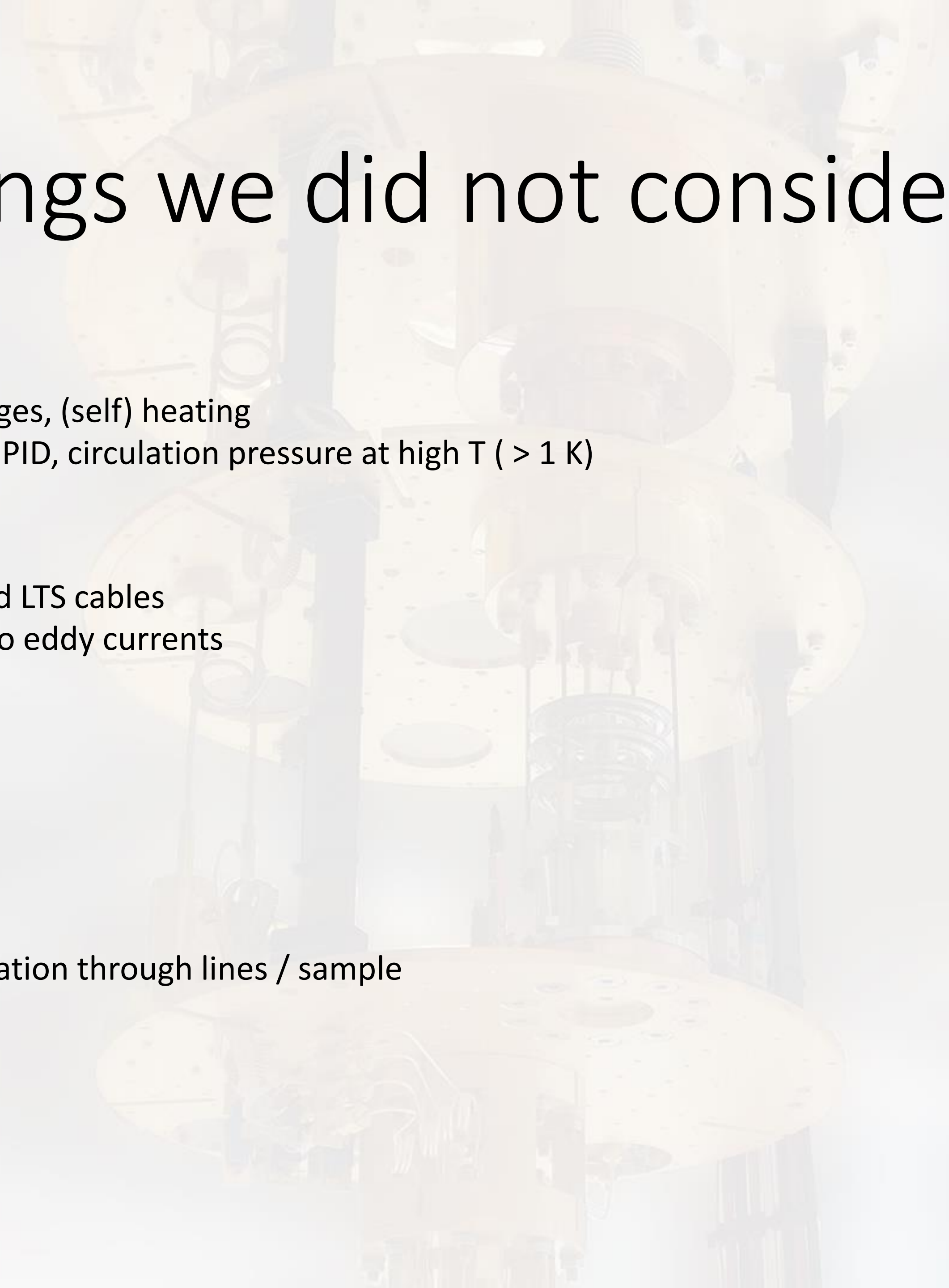
5 μK for Cu electron temperature
2 μK for Pt electron temperature
100 pK for Rh nuclear spin temperature

(Practical) things we did not consider today…



- Thermometry
    - Cernox, RuOx, excitation voltages, (self) heating
    - Temperature control: heaters, PID, circulation pressure at high T (> 1 K)
- Integrated magnets
    - Solenoid or vector magnets
    - Magnet feedthroughs: HTS and LTS cables
    - Mixing chamber heating due to eddy currents
- Keeping the mixture clean
    - External LN_2 trap
    - Internal charcoal traps
- Experimental lines
    - DC looms, RF lines, …
    - Heat load from wiring
    - Heat load due to power dissipation through lines / sample
    - Filter stage(s)
- ….