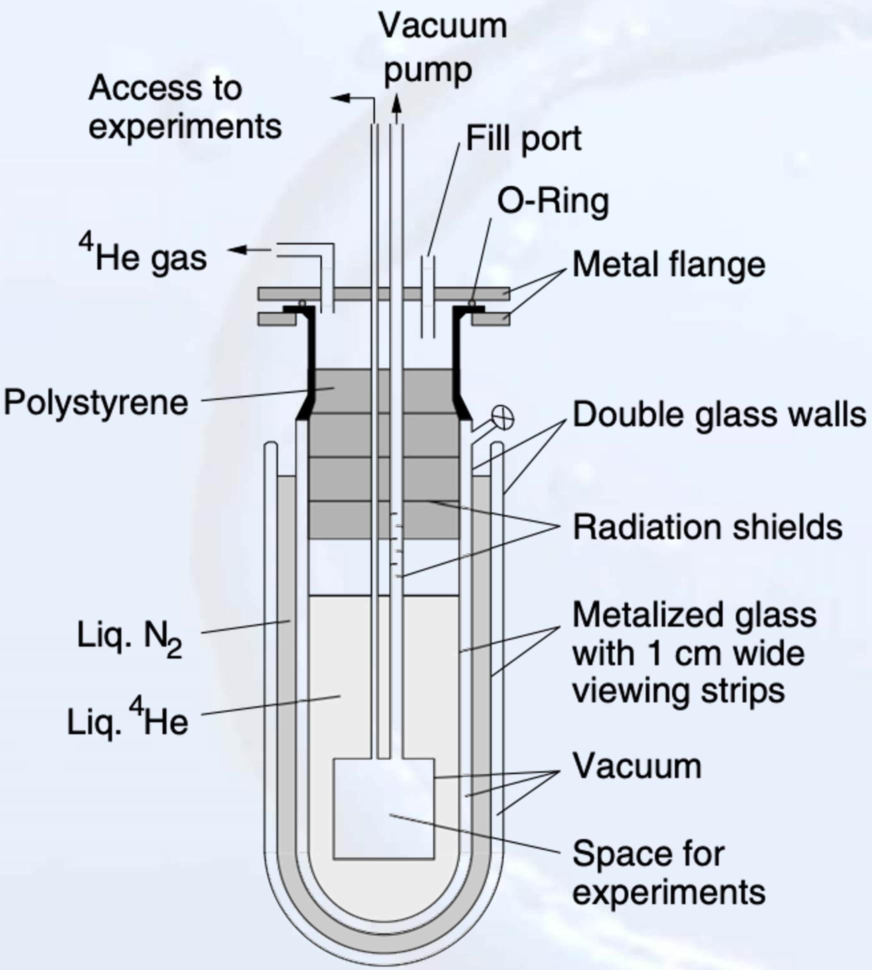
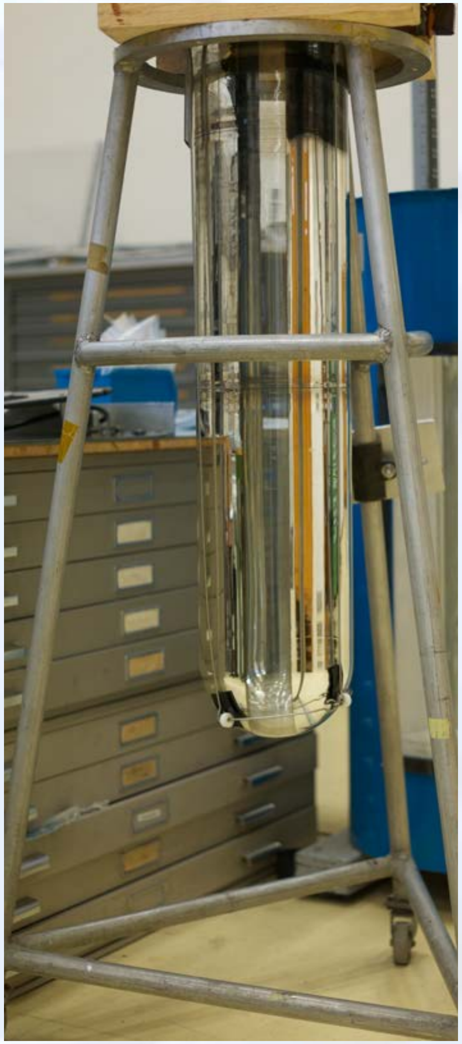
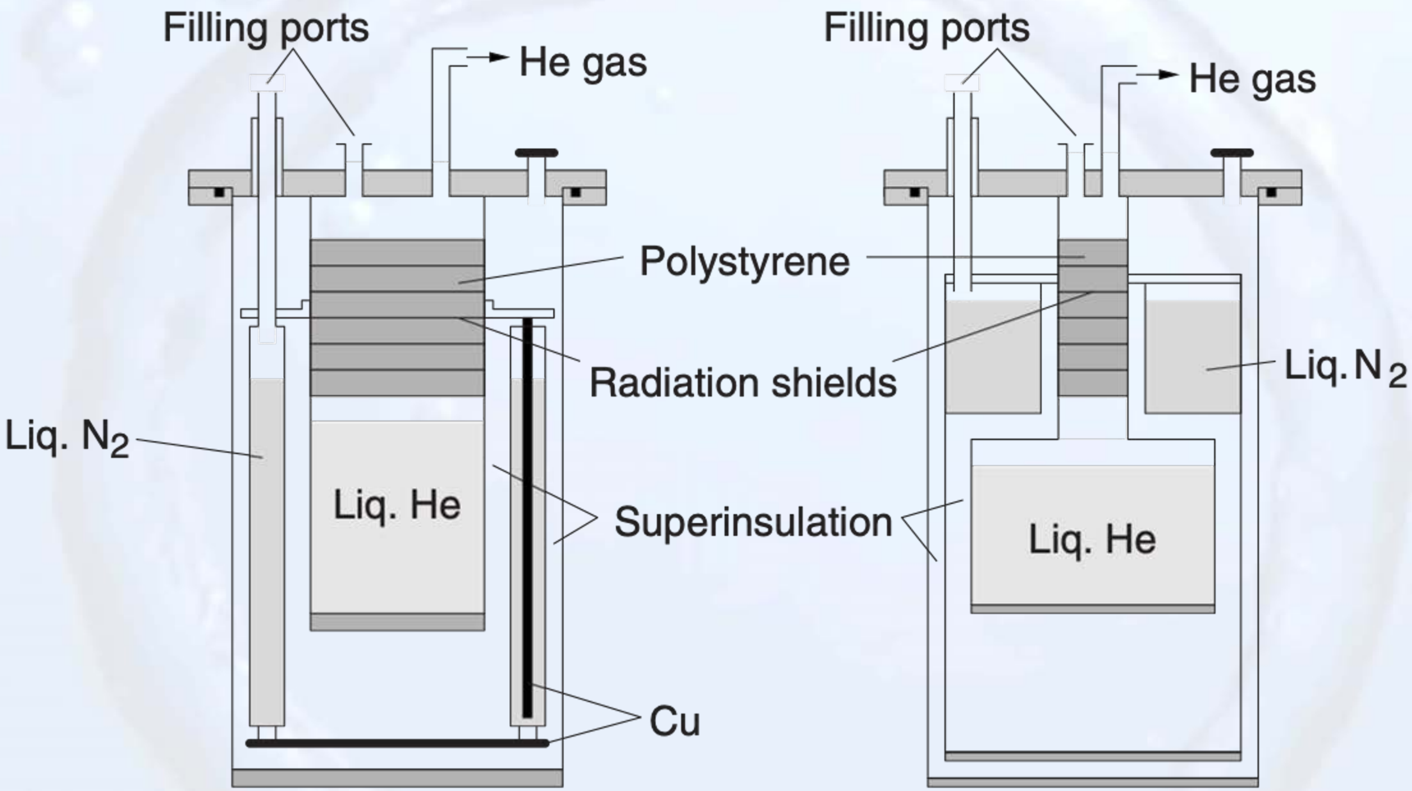
## $^4$He bath cryostat: glass dewar



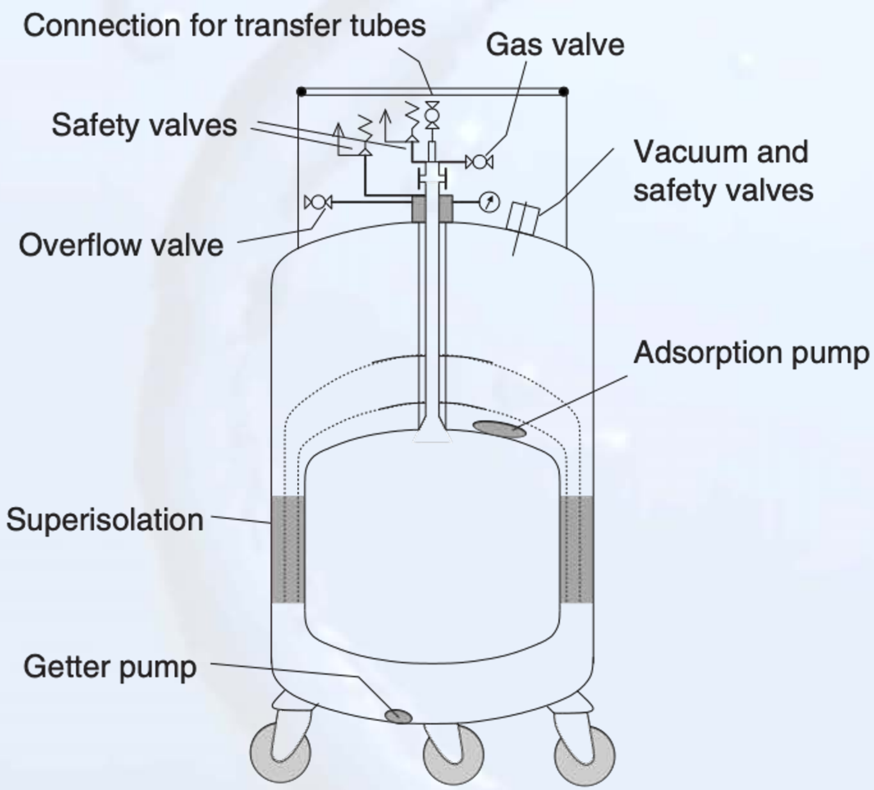
# 11.1 Bath Cryostats

$^4$He Bath cryostat: metal dewar

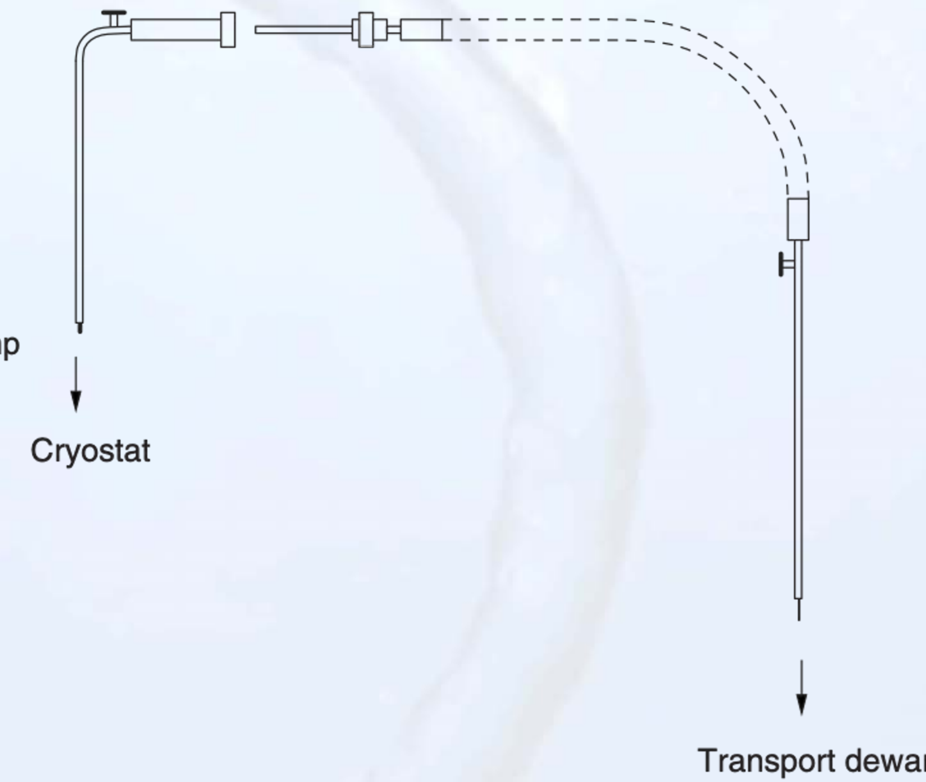


# 11.1 Bath Cryostats

helium transport vessel




helium transfer tube



# 11.1 Bath Cryostats

## Radiation shields – super insulation

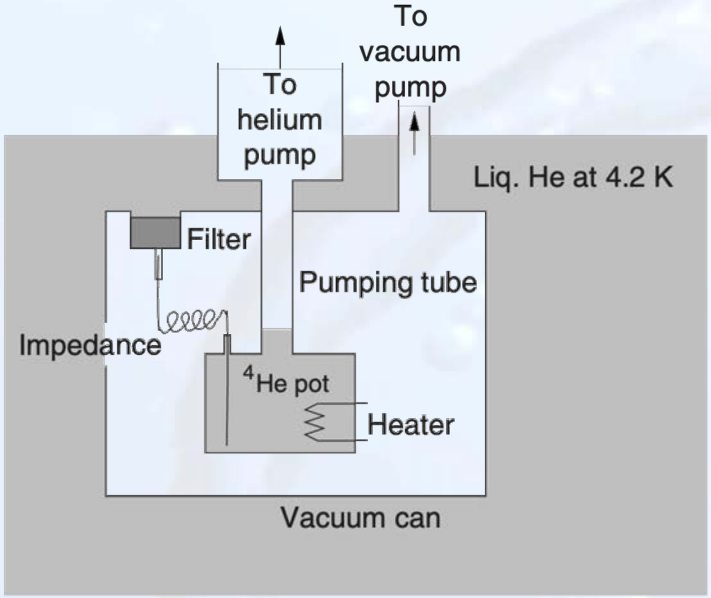

multiple radiation shields → smaller steps → reduction of heat flow

30 to 80 layers of low conductivity
high reflection material → aluminized Mylar

apparent thermal conductivity
$\sim 10^{-4}$ to $10^{-5}$ W/(m K)

# 11.1 Bath Cryostats

## Cryostats with 1-K-Pot




$$^4\mathrm{He} \quad L = 90\,\mathrm{J\,mol^{-1}}$$

$$^3\mathrm{He} \quad L = 40\,\mathrm{J\,mol^{-1}}$$

## Vapor pressure curve of various cryogenic liquids

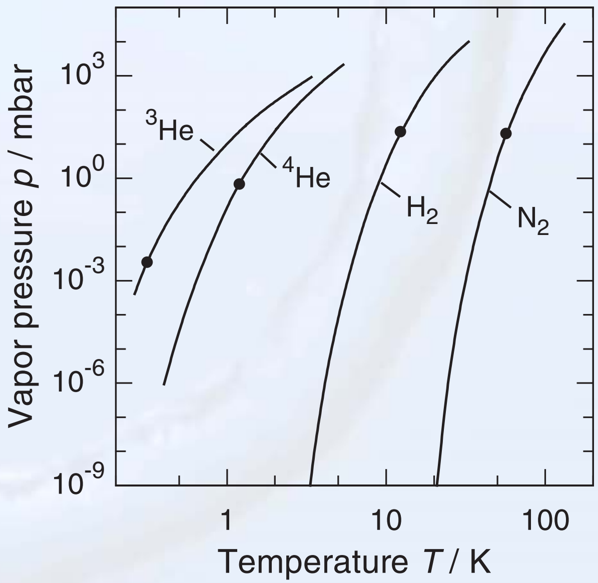
Clausius-Clapeyron equation

$$\frac{\mathrm{d}p}{\mathrm{d}T} = \frac{L}{\Delta V\, T}$$

$$\Delta V = V_g - V_\ell \approx V_g \qquad pV_g = RT$$

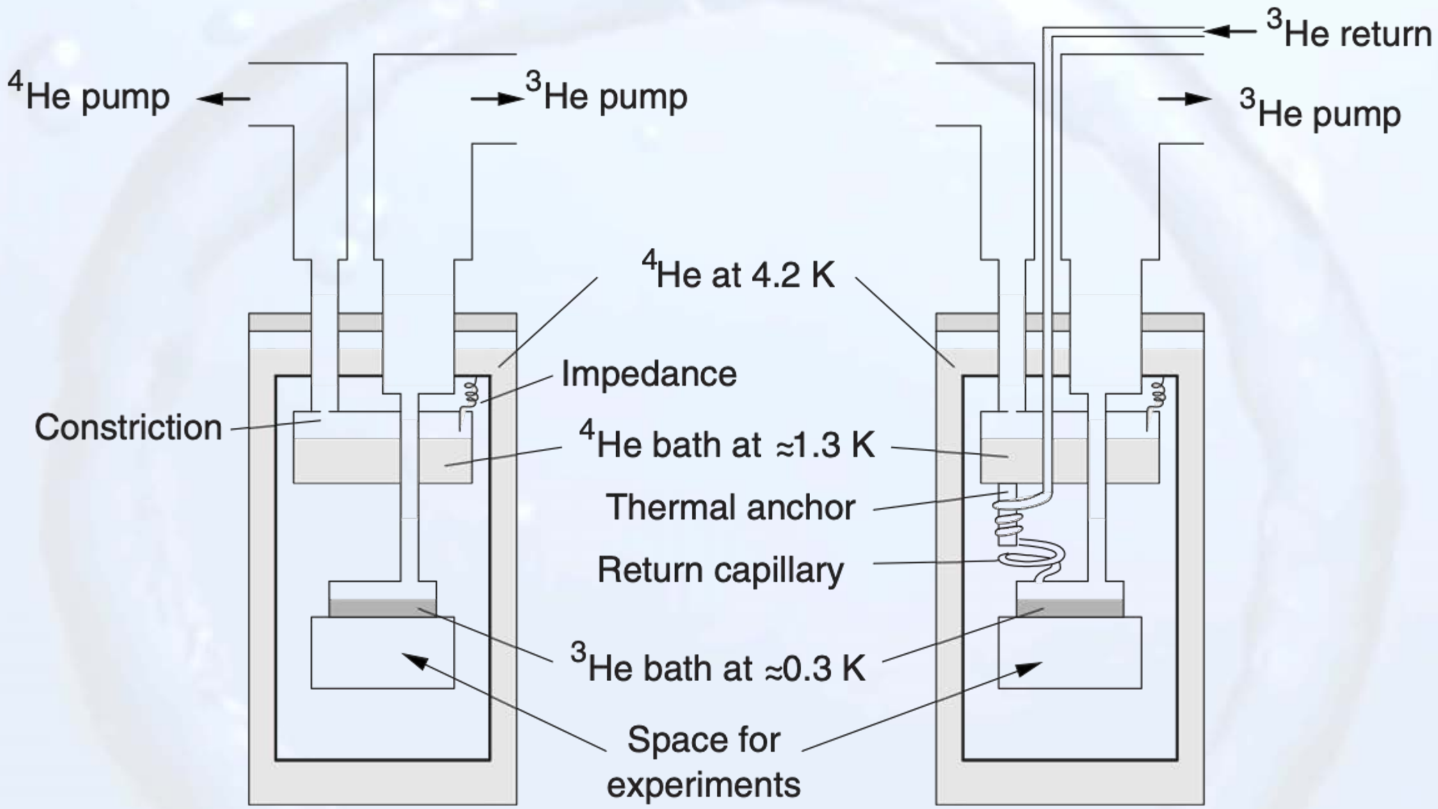
$$\frac{\mathrm{d}p}{\mathrm{d}T} = \frac{L}{RT^2}\, p \quad \rightarrow \quad p(T) = p_0\, \mathrm{e}^{-L/RT}$$

vapor pressure curve



# 11.1 Bath Cryostats

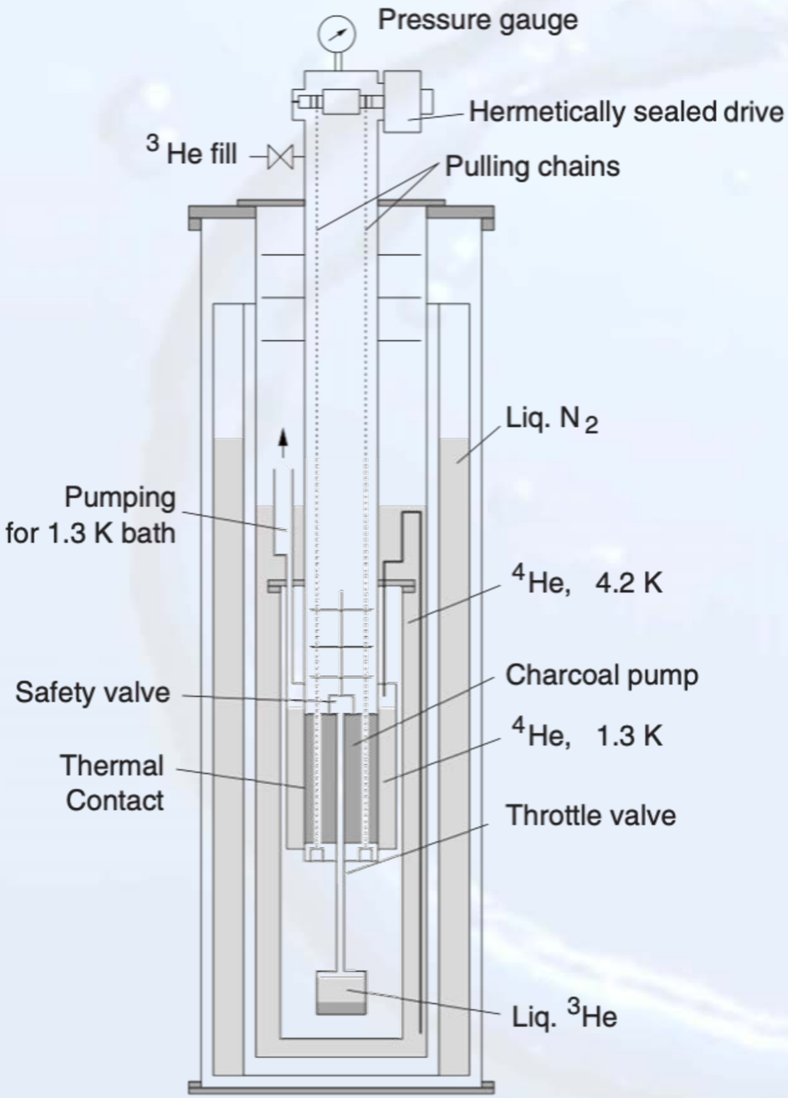
## $^3$He cryostats

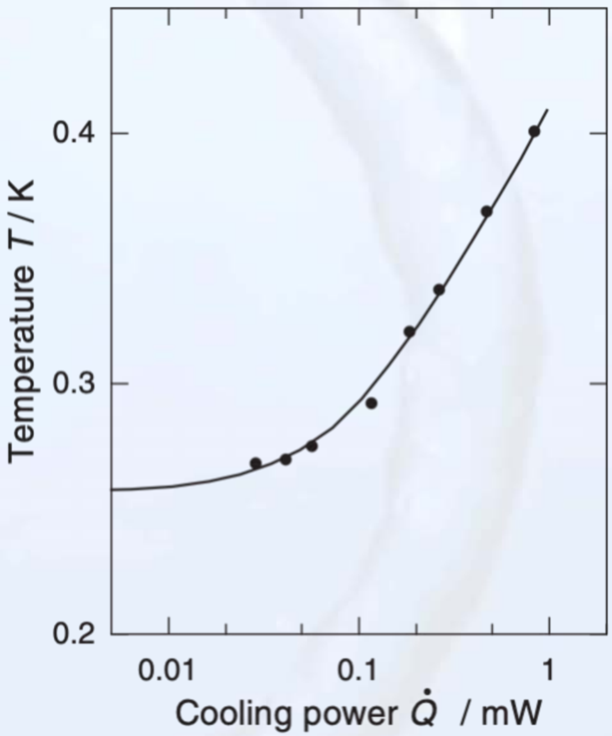



cooling power $\dot{Q} = \dot{n}_{\mathrm{g}}\, L \propto p \propto \mathrm{e}^{-L/RT}$

# 11.1 Bath Cryostats

## Cooling power of a $^3$He cryostat with charcoal absorption pump



# 11.2 Dilution Refrigerators

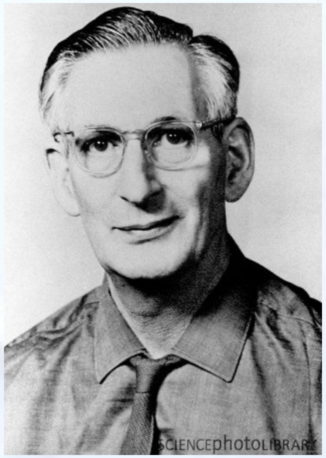
History

1951 basic idea suggested by Heinz London

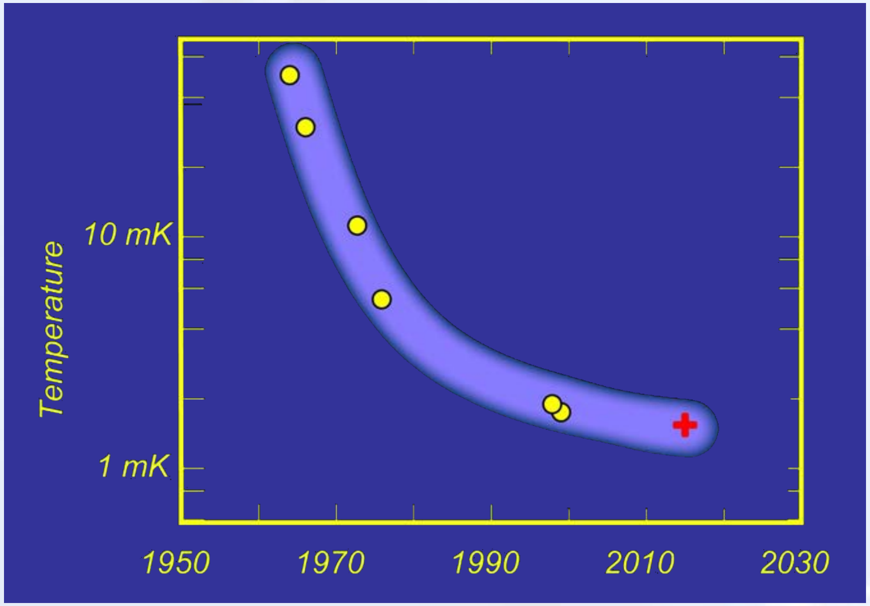
1962 detailed concept worked out by London, Clark, Mendoza

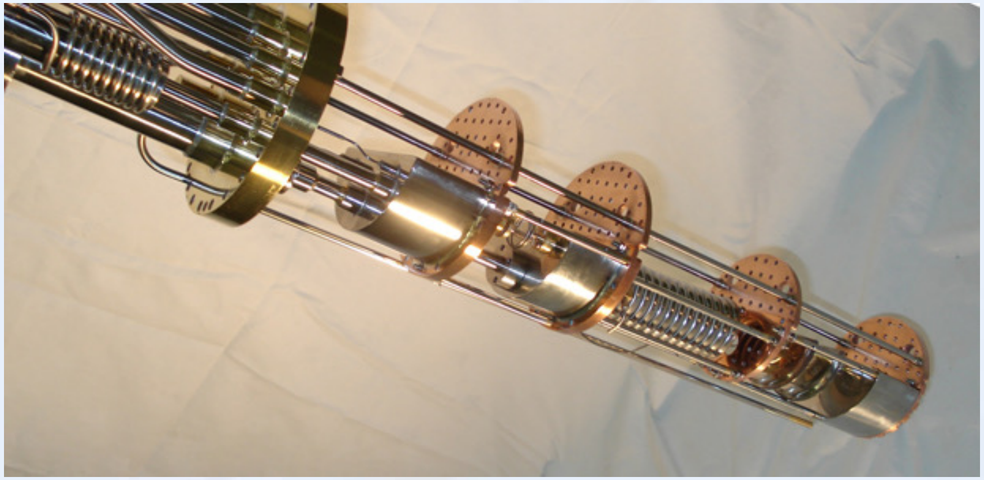
1965 first realization Das, De Bruyn Ouboter, Taconis $T_{min}$ = 220 mK

1999 lowest temperature obtained , J.C. Cousins *et al.* $T_{min}$ = 1.75 mK

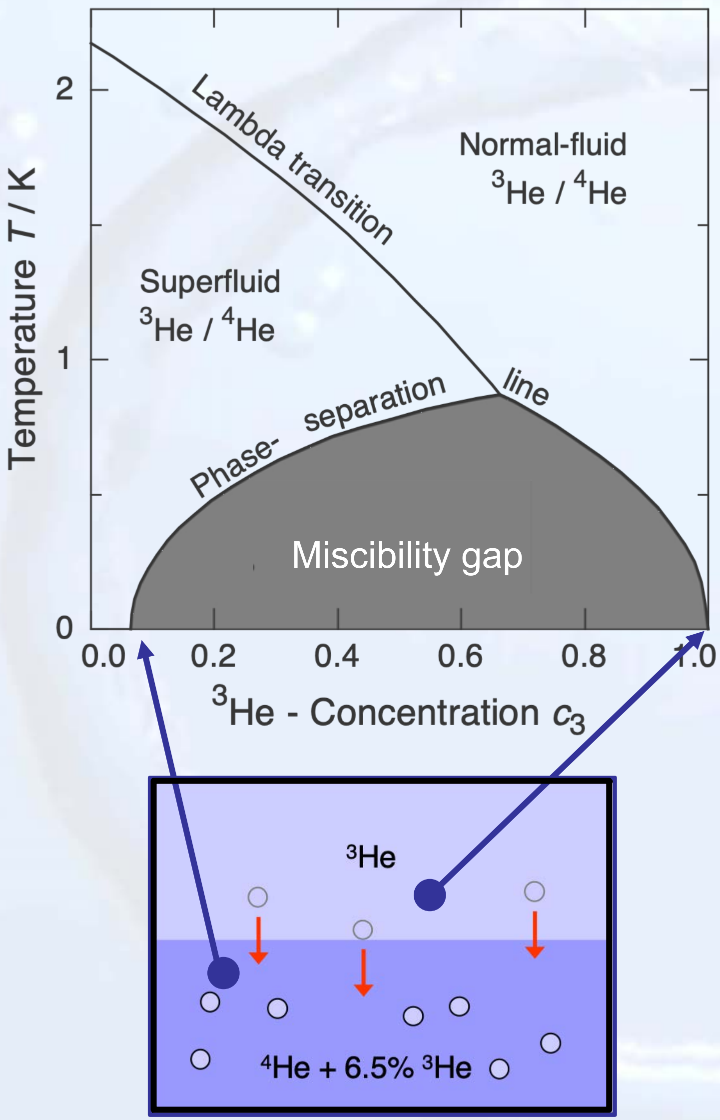


Heinz London



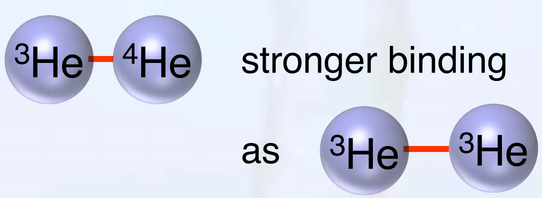
# 11.2 Dilution Refrigerators




occurrence of miscibility gap

but 6.5 % $^3$He in $^4$He at $T = 0$ K

reason:
zero-point motion weakens binding



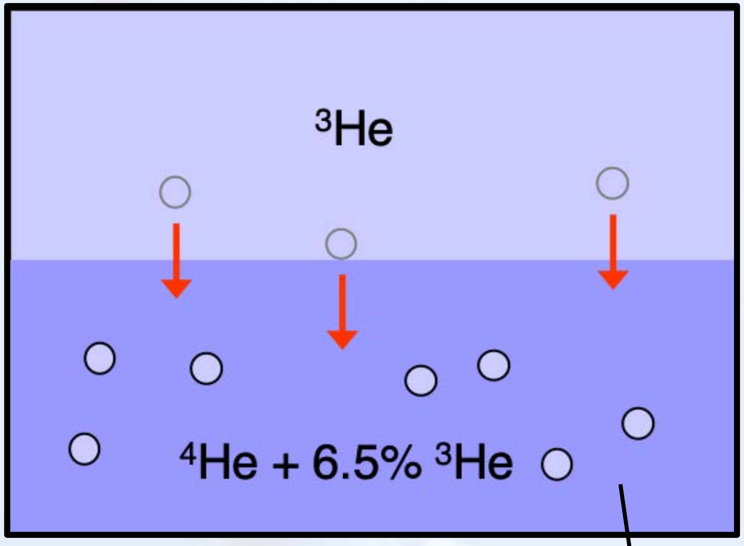

but: Fermi energy

max. 6.5% $^3$He in $^4$He at $T = 0$ K

# 11.2 Dilution Refrigerators

principal of cooling by mixing $^3He/^4He$

- transition of $^3He$ into the $^4He$ rich phase
- cooling by „evaporation“ of $^3He$ into $^4He$ quasi vacuum



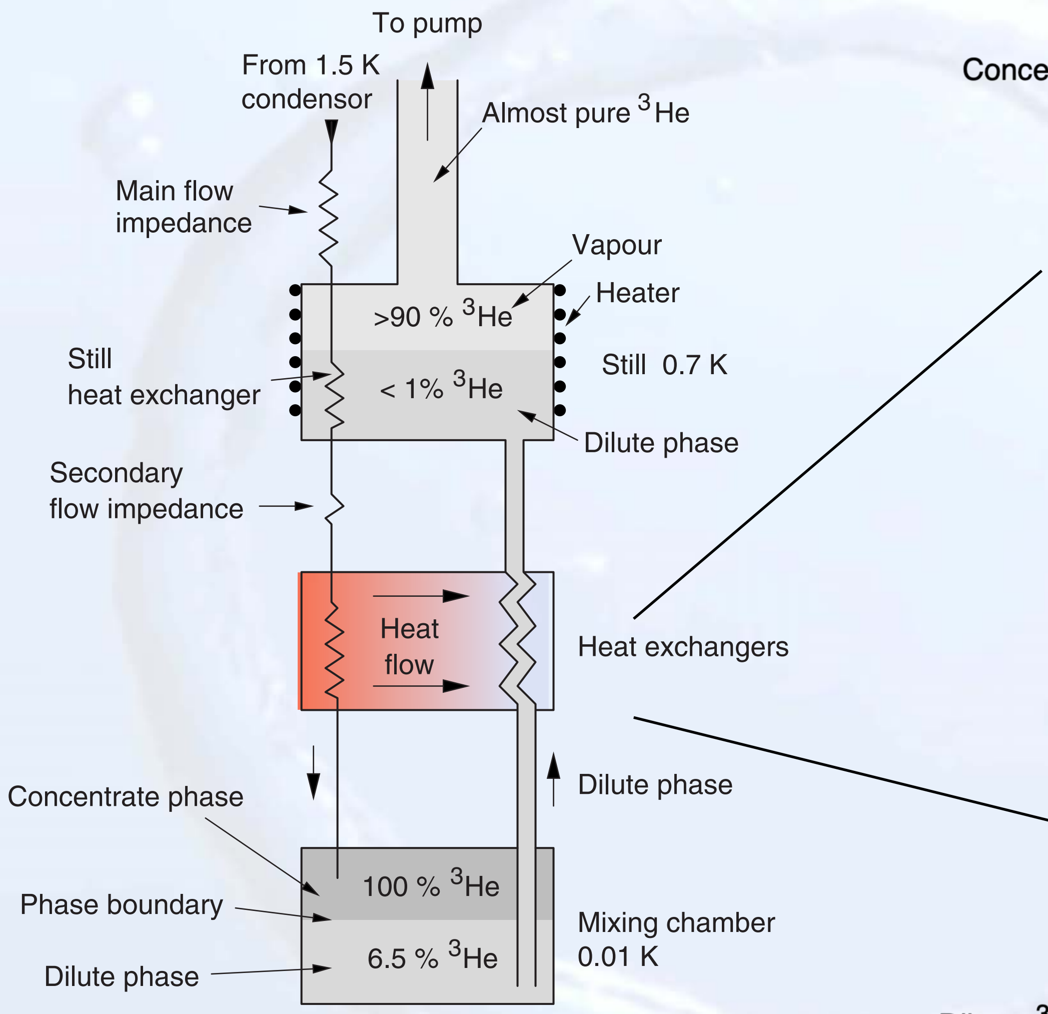

heat of solubility per Mol:

$$\Delta Q = T \Delta S = aT^2$$

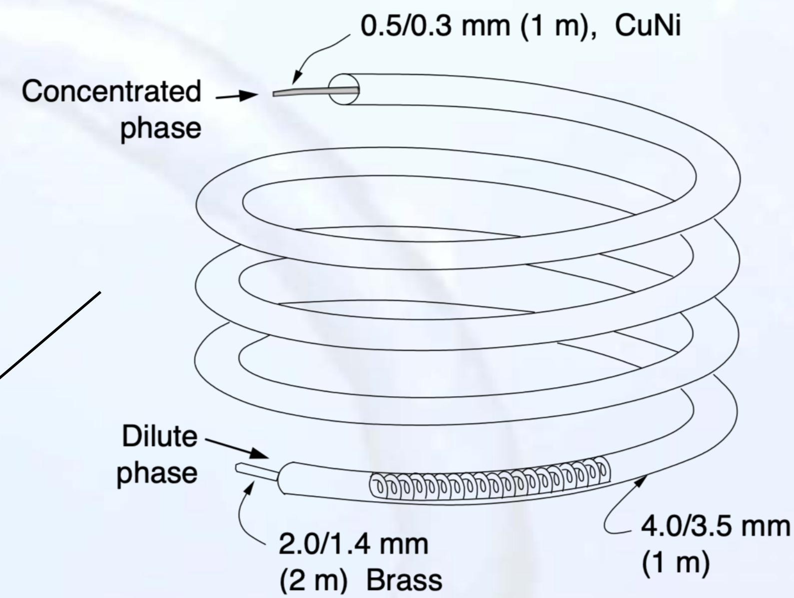
$$a = -84\,\mathrm{J/K^2}$$

# 11.2 Dilution Refrigerators

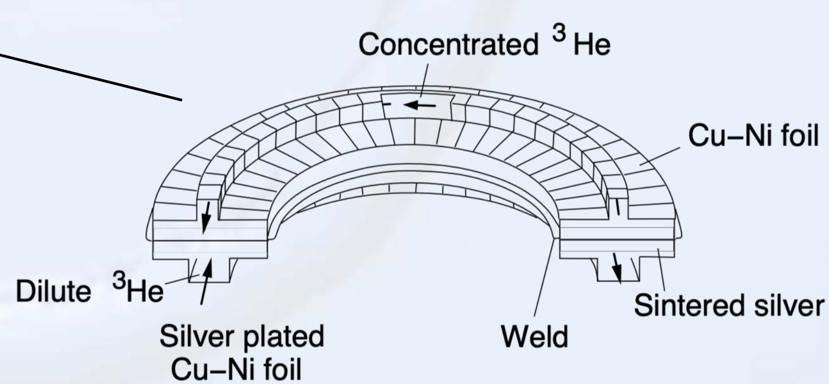
## Realisation of $^3He/^4He$ cooling cycle




## continuous heat exchanger




## step heat exchanger



# 11.2 Dilution Refrigerators

Kapitza Resistance — thermal boundary resistance

Snell's law of refraction $$\frac{\sin\alpha_\ell}{\sin\alpha_s} = \frac{v_\ell}{v_s}$$

critical angle of total reflection $$\alpha_\ell^c = \arcsin\left(\frac{v_\ell}{v_s}\right)$$

for liquid helium and copper $\alpha_\ell^c \approx 4°$

fraction of phonons incident within critical angle

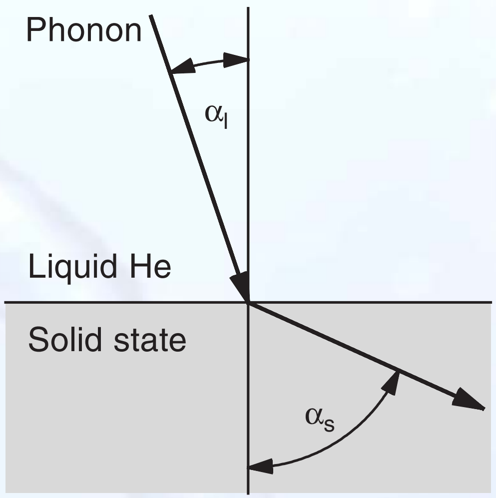
$$f = \frac{1}{2}\sin^2\alpha_\ell^c = \frac{1}{2}\left(\frac{v_\ell}{v_s}\right)^2 < 10^{-2}$$

transmission coefficient

$$t = \frac{4Z_\ell Z_s}{(Z_\ell + Z_s)^2} \approx \frac{4Z_\ell}{Z_s} = \frac{4\varrho_\ell v_\ell}{\varrho_s v_s}$$

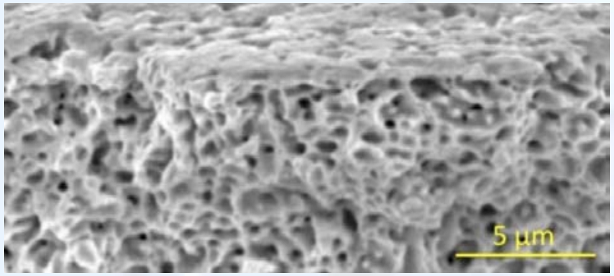
$Z_\ell = \varrho_\ell v_\ell$ $Z_s = \varrho_s v_s$ acoustic impedances

fraction of phonons crossing the interface $$ft = \frac{2\varrho_\ell v_\ell^3}{\varrho_s v_s^3}$$



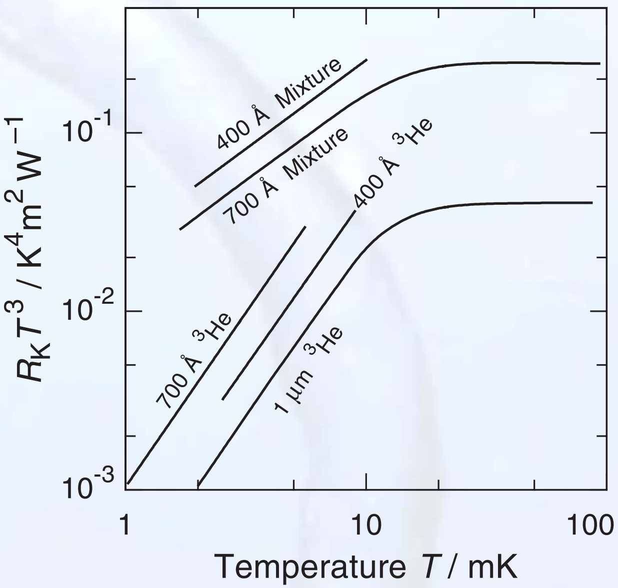

- Kaptiza resistance occurs at any solid-solid, liquid-solid interface
- particular problematic for liquid helium because of the low sound velocity
- helium-copper $ft < 10^{-5}$

# 11.2 Dilution Refrigerators




silver sinter
SEM image

Kapitza resistance between pure $^3$He and $^3$He/$^4$He mixtures and silver sinters of different grain sizes




heat flow from liquid to solid (using Debye model)

$$\dot{Q} = \frac{1}{2} f t u v_\ell A = \frac{\pi^2 k_B^4 \varrho_\ell v_\ell}{30\hbar^3 \varrho_s v_s^3} A T^4$$

$$u = U/V = \pi^2 k_B^4 T^4 / (30\hbar^3 v_\ell^3)$$

in equilibrium identical heat flow from solid to liquid

net flow in non-equilibrium ($\Delta T$)

$$\dot{Q} = \frac{d\dot{Q}}{dT} \Delta T = \frac{2\pi^2 k_B^4 \varrho_\ell v_\ell}{15\hbar^3 \varrho_s v_s^3} A T^3 \Delta T$$

Kapitza resistance

$$R_K = \frac{A \Delta T}{\dot{Q}} = \frac{15\hbar^3 \varrho_s v_s^3}{2\pi^2 k_B{}^4 \varrho_\ell v_\ell} \frac{1}{T^3}$$

- $20\,\mathrm{mK} < T < 100\,\mathrm{mK}$ good agreement with Debye model $R_K \propto T^{-3}$
- below 20 mK $R_K \propto T^{-2}$ or $R_K \propto T^{-1}$

⟶ anomalous Kapitza resistance

origin: TLS, coupling to zero and second sound modes, phonon wavelength larger than sinter grains

Cooling power

assuming 100% $^3$He circulation one finds in equilibrium:

$$\dot{Q}_{\mathrm{mc}} + \dot{N}_3\,[H_3(T_{\mathrm{ex}}) - H_3(T_{\mathrm{mc}})] = \dot{N}_3\,[H_{3,\mathrm{d}}(T_{\mathrm{mc}}) - H_3(T_{\mathrm{mc}})]$$

- $\dot{Q}_{\mathrm{mc}}$: heat leak and/or available cooling power
- $\dot{N}_3$: circulation rate
- $T_{\mathrm{ex}}$: temperature after last heat exchanger
- $H_3$: enthalpy of $^3$He-rich phase
- $H_{3,\mathrm{d}}$: enthalpy of $^3$He-dilute phase
- $T_{\mathrm{mc}}$: mixing chamber temperature

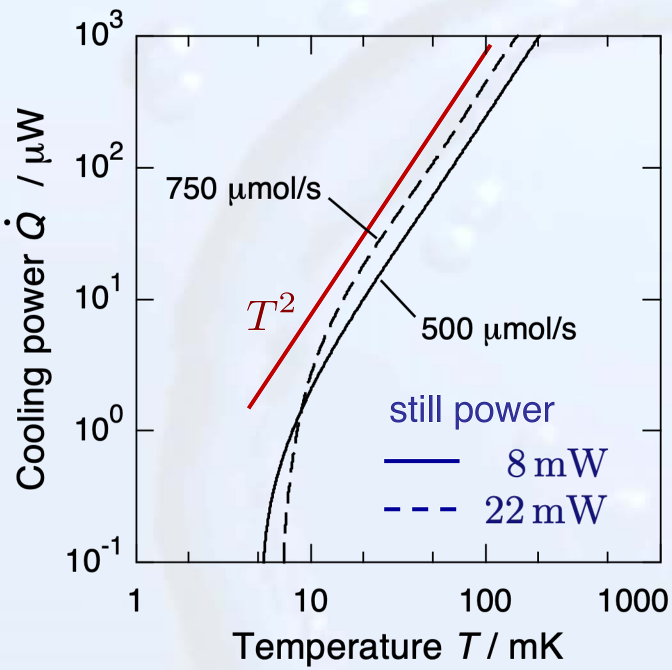
enthalpy

$$H = U + pV$$

inserting the enthalpies

$$\begin{aligned}\dot{Q}_{\mathrm{mc}} &= \dot{N}_3\,[H_{3,\mathrm{d}}(T_{\mathrm{mc}}) - H_3(T_{\mathrm{ex}})] \\ &= \dot{N}_3\,(95\,T_{\mathrm{mc}}^2 - 11\,T_{\mathrm{ex}}^2)\left(\frac{\mathrm{J}}{\mathrm{mol\ K^2}}\right)\end{aligned}$$

## Temperature and circulation rate dependence of the cooling power




limiting case of vanishing cooling power: $\dot{Q}_{\mathrm{mc}} = 0$

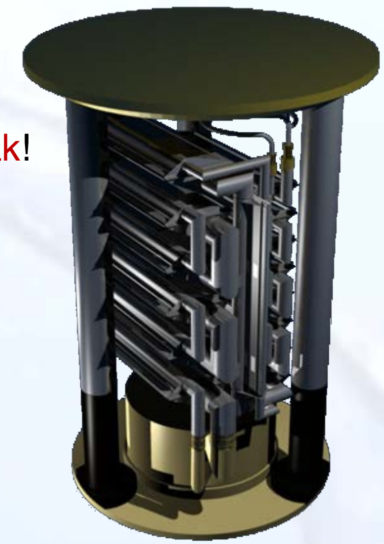
$$95\,T_{\mathrm{mc}}^2 - 11\,T_{\mathrm{ex}}^2 = 0$$

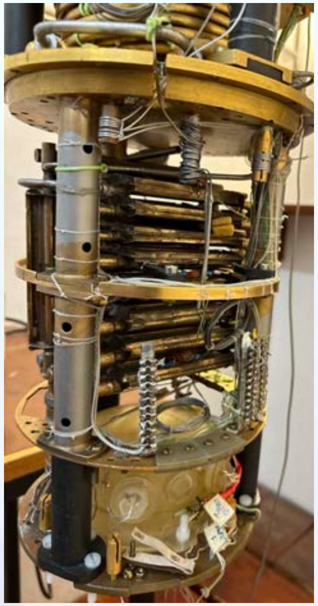
$$\frac{T_{\mathrm{ex}}}{T_{\mathrm{mc}}} = 2.8$$

→ this underlines the importance of the heat exchanger quality

- for $\dot{Q} \gg \dot{Q}_{\mathrm{heat\ leak}}$ ⟶ $\dot{Q} \propto T^2$ , $\dot{Q} \propto \dot{N}_3$ (circulation rate)
- heat leak determines lowest temperature

# 11.2 Dilution Refrigerators





## Minimum temperature

- there is no principle limit … it is determined by the heat leak!
- unavoidable heat leak: viscous friction of $^3$He

pressure difference along the heat exchanger:

$$\Delta p = G\eta\dot{V}$$ Hagen-Poiseuille law

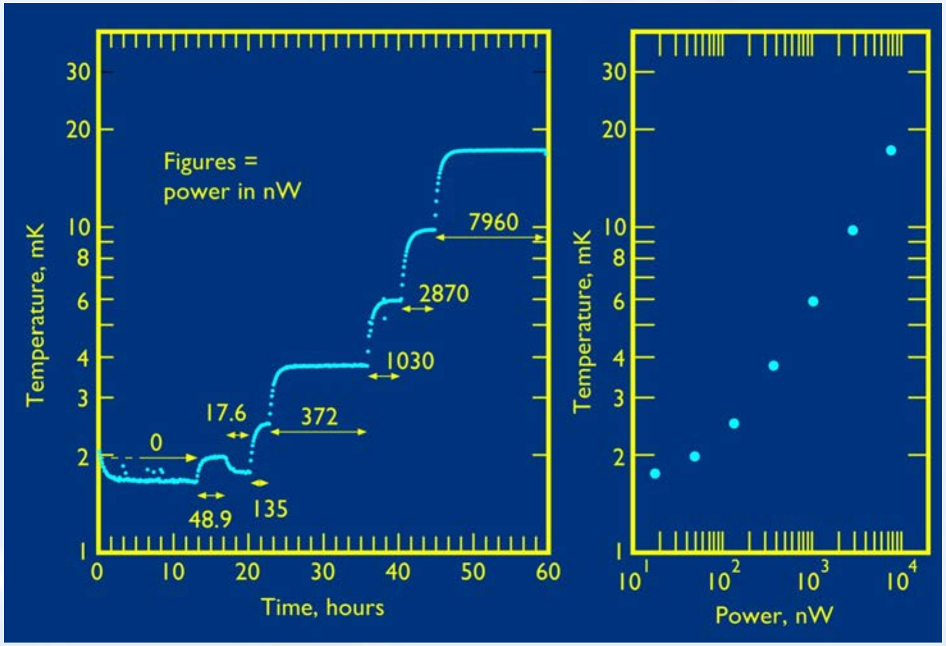
$$G = 8L/(\pi r^4)$$

heat leak due to viscous friction

$$\dot{Q}_{\text{visc}} = \dot{V}\,\Delta p \; = \; G\eta\dot{V}^2$$

single shot minimum temperature

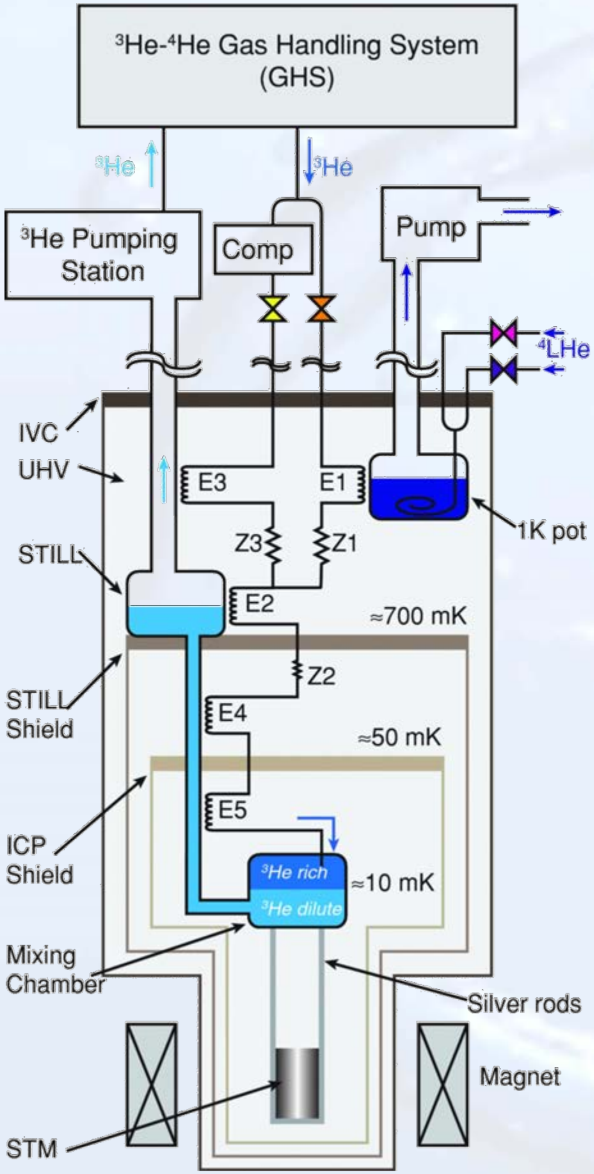
$$T_{\text{min.}} = \frac{4}{\sqrt[3]{d}}\,\text{mK}(\text{mm})^{1/3}$$



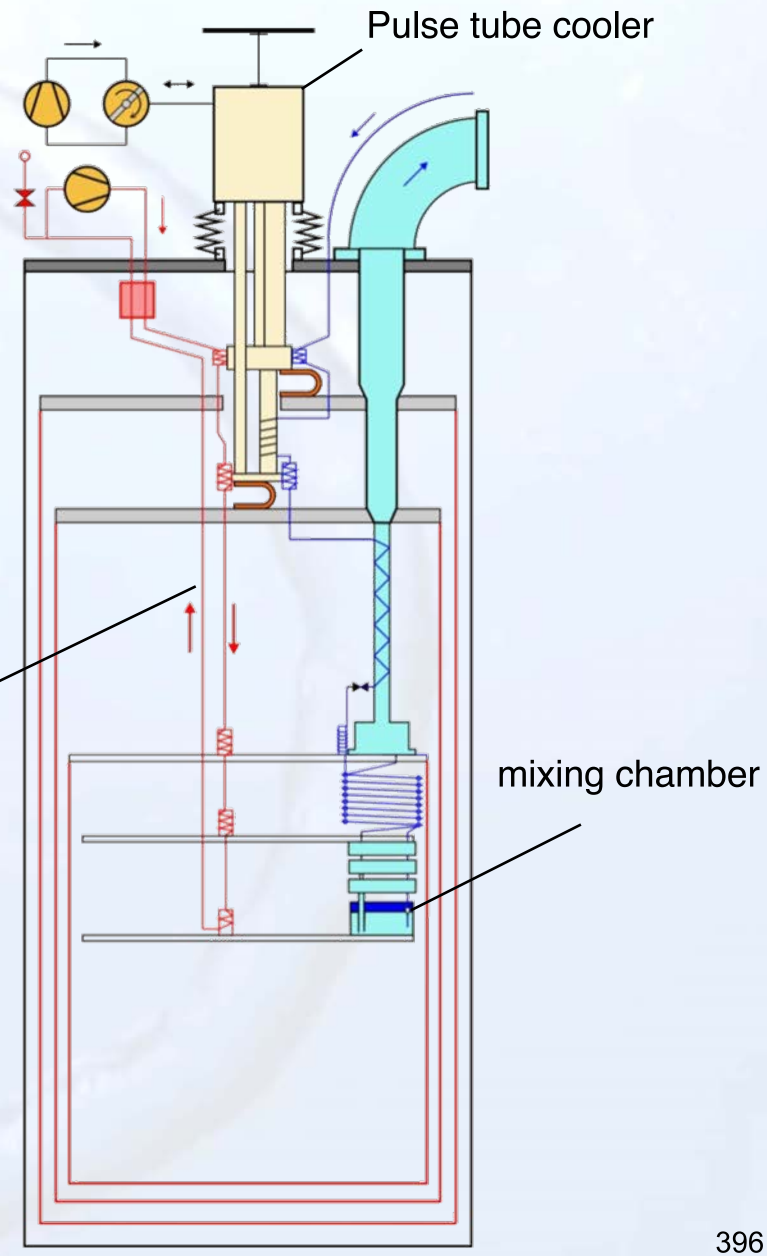
# 11.2 Dilution Refrigerators

## Realizations

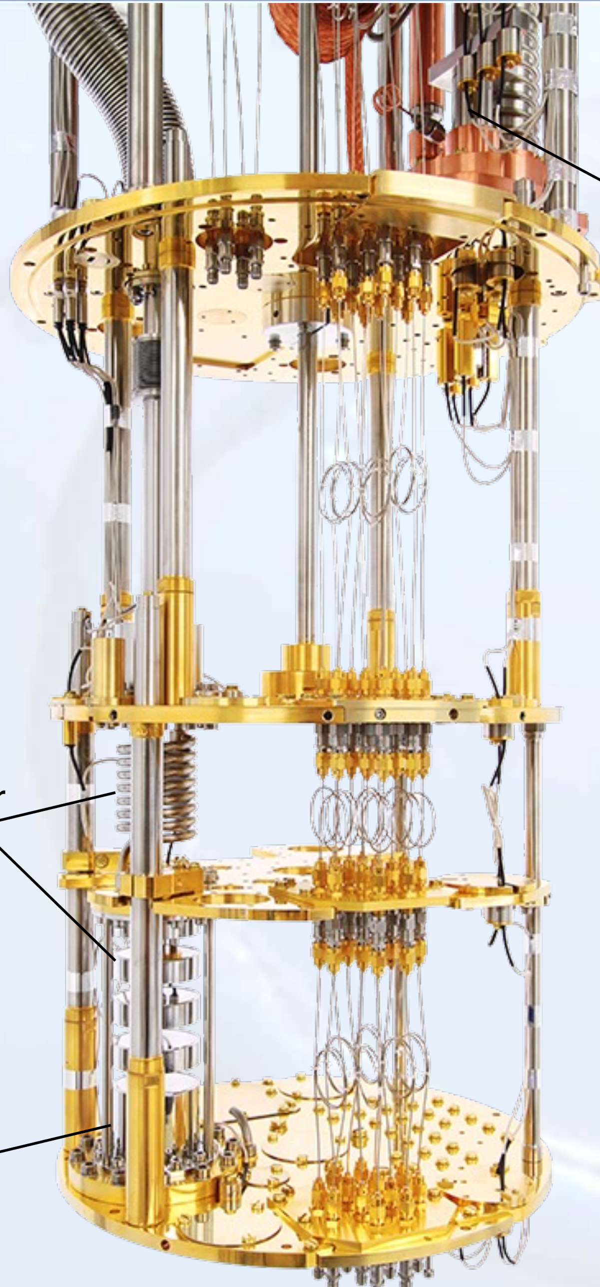
Wet

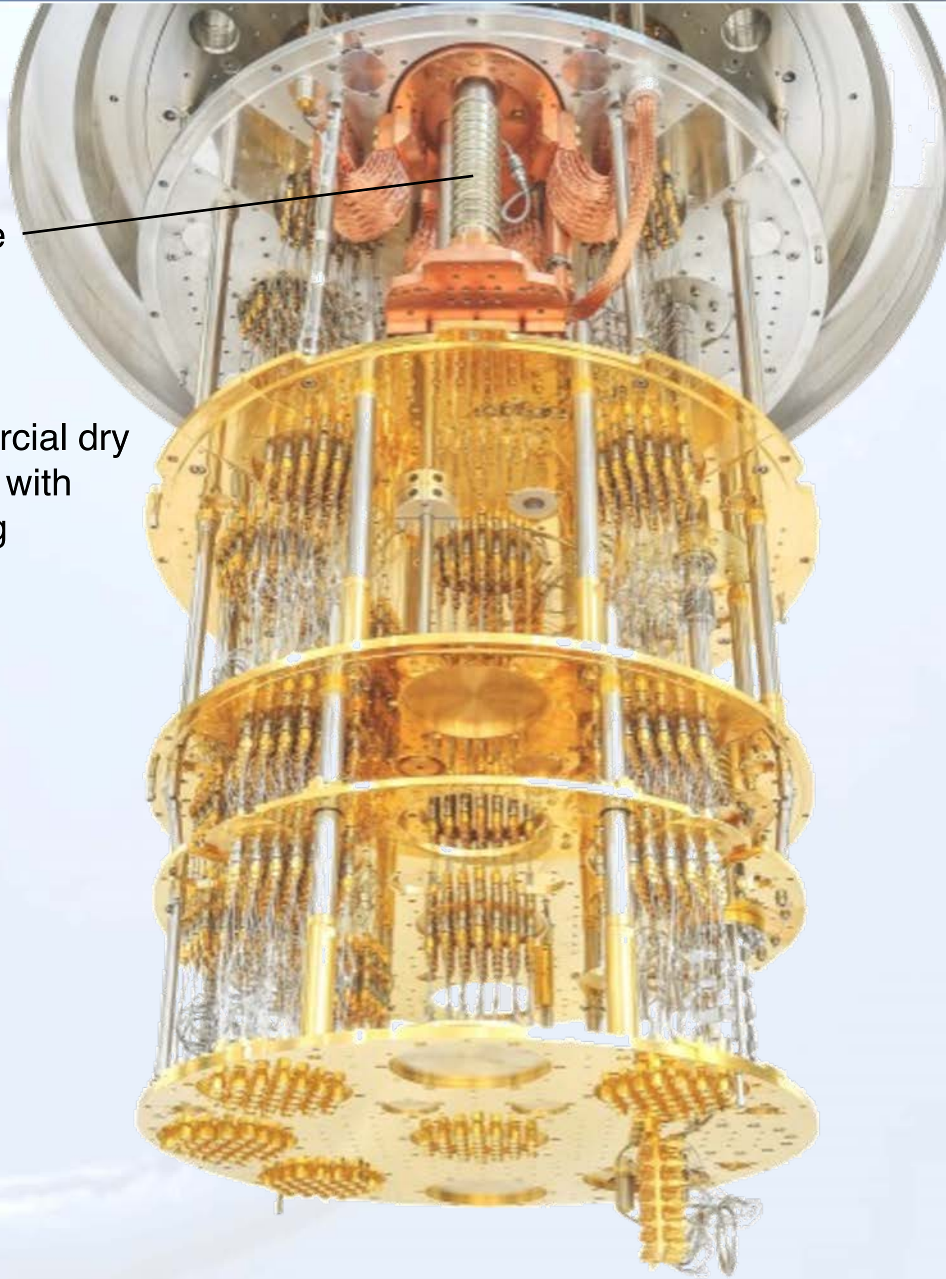



Dry



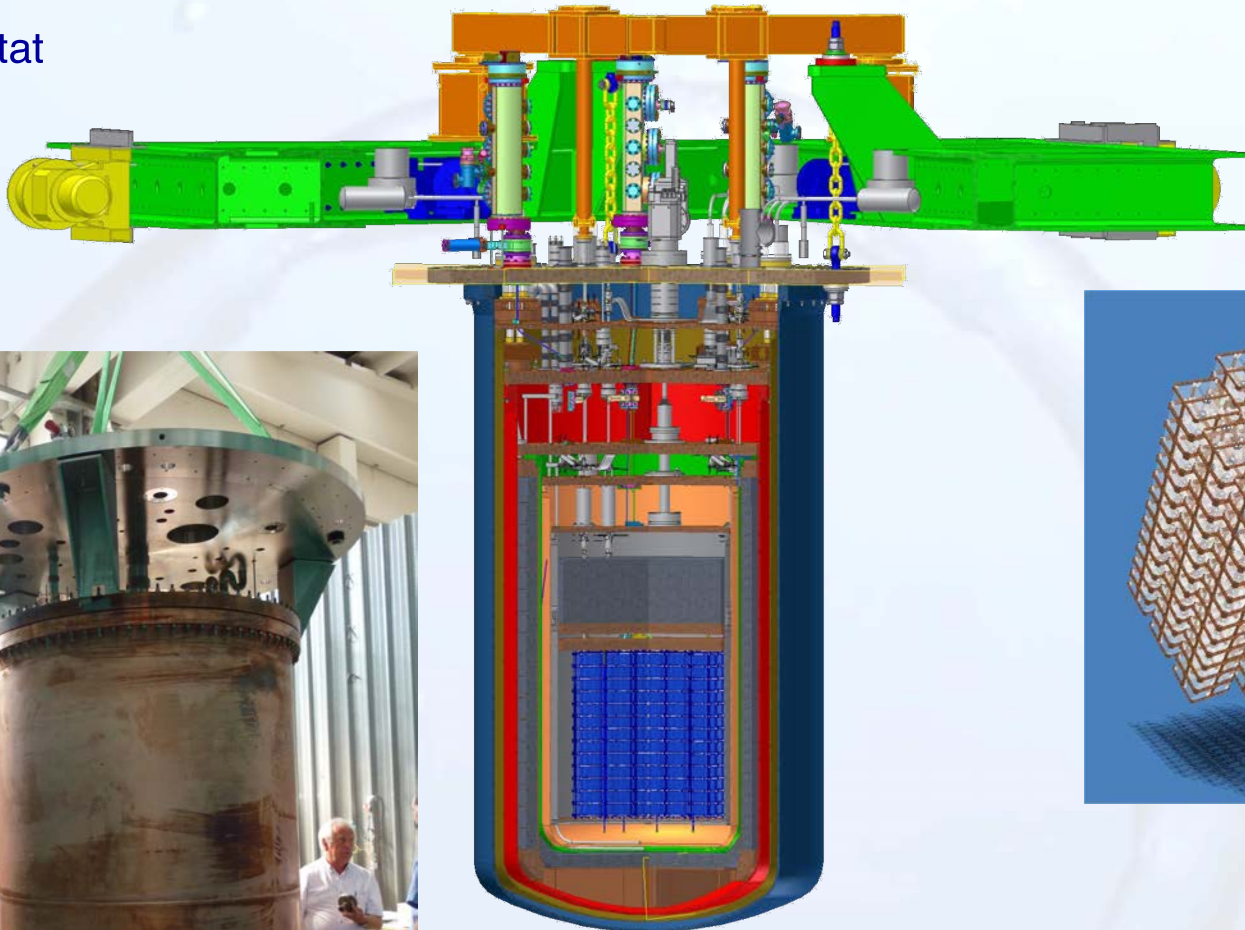
# 11.2 Dilution Refrigerators

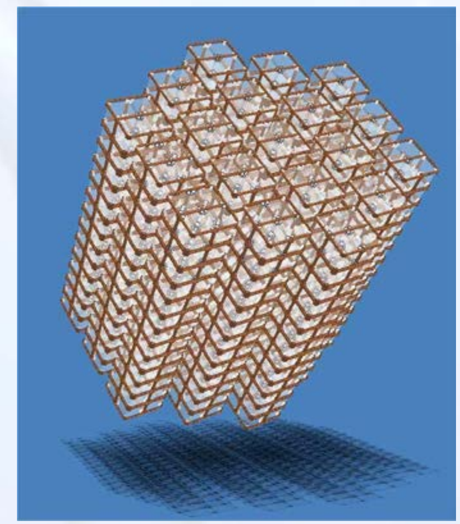


# 11.2 Dilution Refrigerators

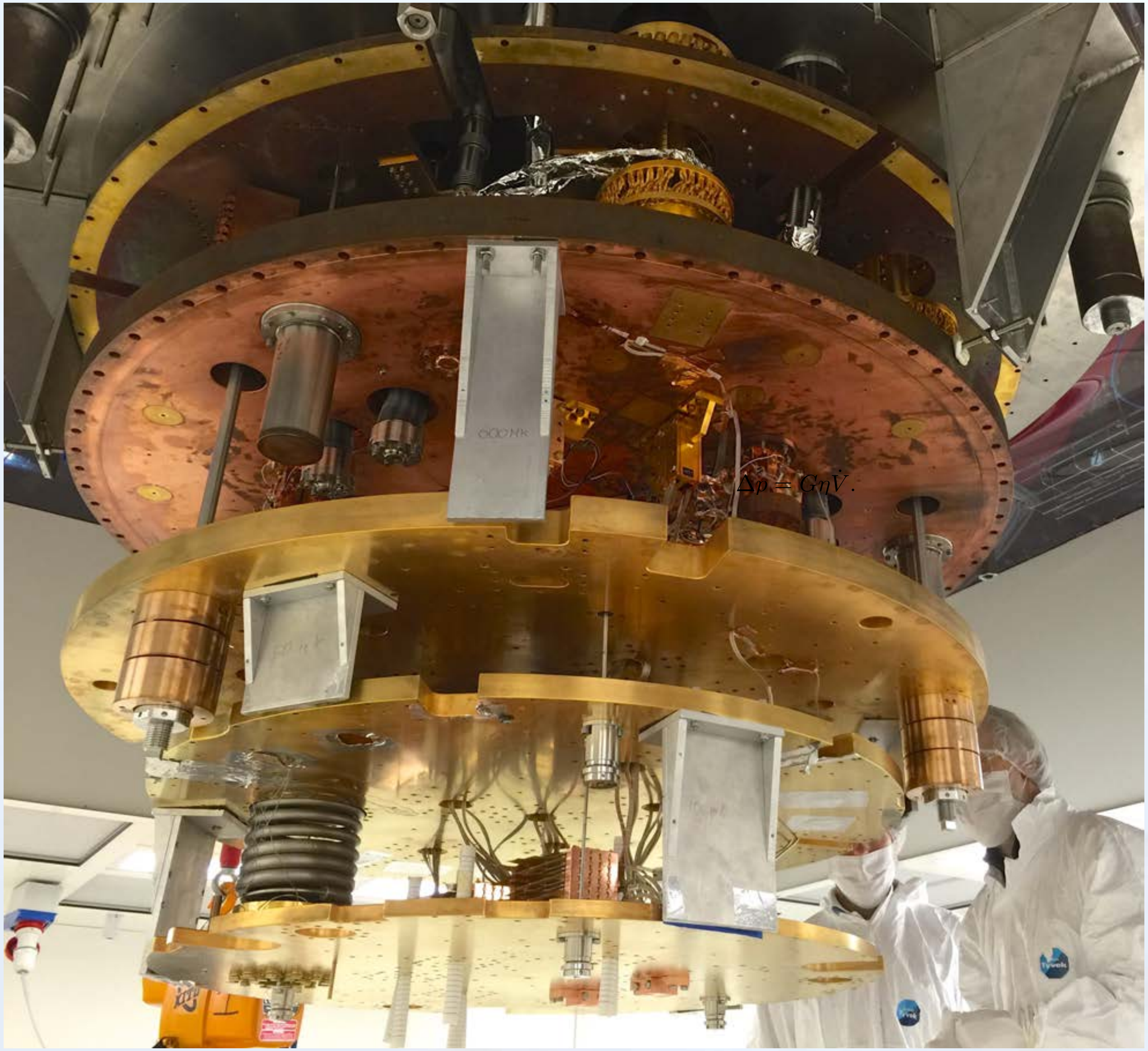
## Cuore Cryostat





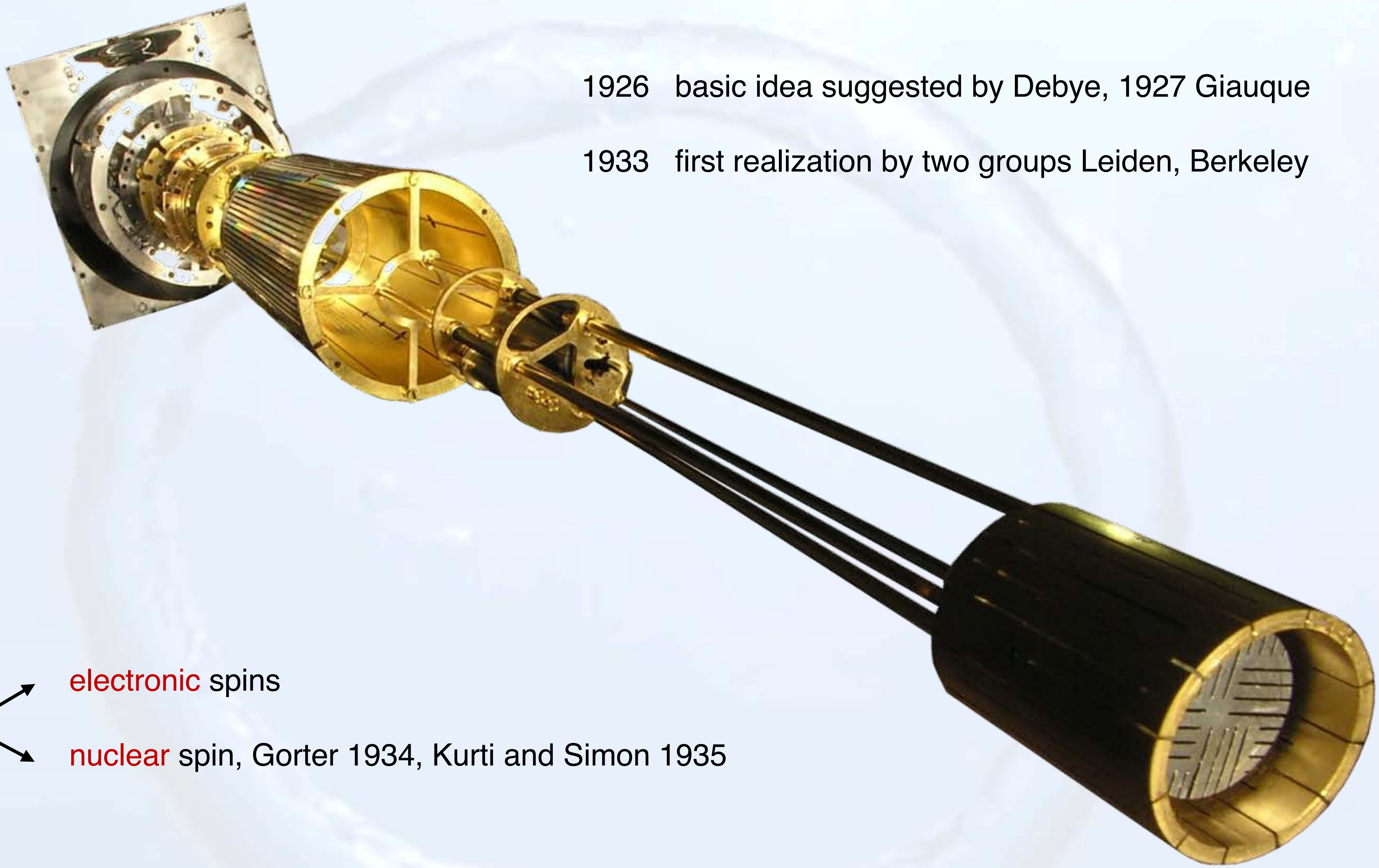
- $750$ kg of $TeO_2$ cooled by the mixing chamber to 10 mK
- made of materials with low level radioactivity materials
- two cold shields made of ancient roman lead 10 cm thick
- 5 Pulse Tube Coolers

# 11.2 Dilution Refrigerators




Cuore Cryostat

# 11.3 Adiabatic Demagnetization Refrigerators



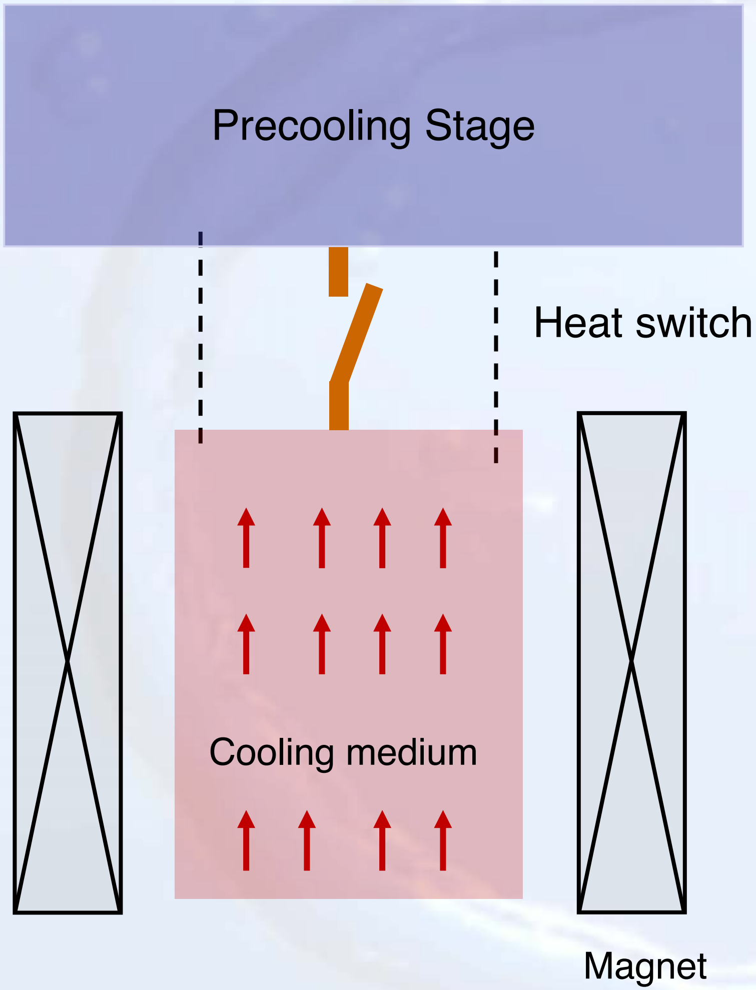
1926 basic idea suggested by Debye, 1927 Giauque

1933 first realization by two groups Leiden, Berkeley

- electronic spins
- nuclear spin, Gorter 1934, Kurti and Simon 1935

# 11.3 Adiabatic Demagnetization Refrigerators

## General cooling principle




- precooling
- isothermal magnetisation

$$\Delta Q_{\rm mag} = -T_{\rm i}[\,S(B_{\rm i},T_{\rm i}) - S(0,T_{\rm i})\,]$$

- thermal isolation
- heat switch opened
- adiabatic demagnetisation

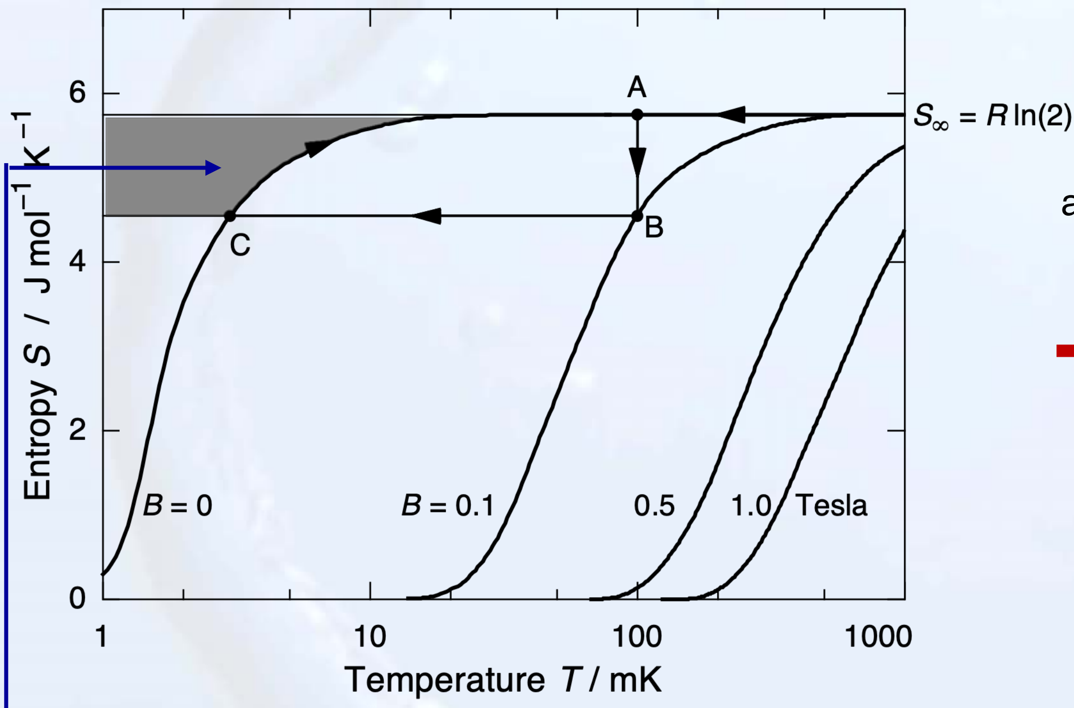
$$S = S\left(\frac{B}{T}\right) = \text{const.}$$

# 11.3 Adiabatic Demagnetization Refrigerators

Entropy: ideal system, no heat leaks

internal field caused by spin-spin interaction

$$S = N k_B \left\{ \ln(2J+1) - \frac{g^2 J(J+1)\mu_B^2}{6\,k_B{}^2} \frac{B^2 + B_{int}^2}{T^2} \right\}$$




adiabatic demagnetization $S_B = S_c$

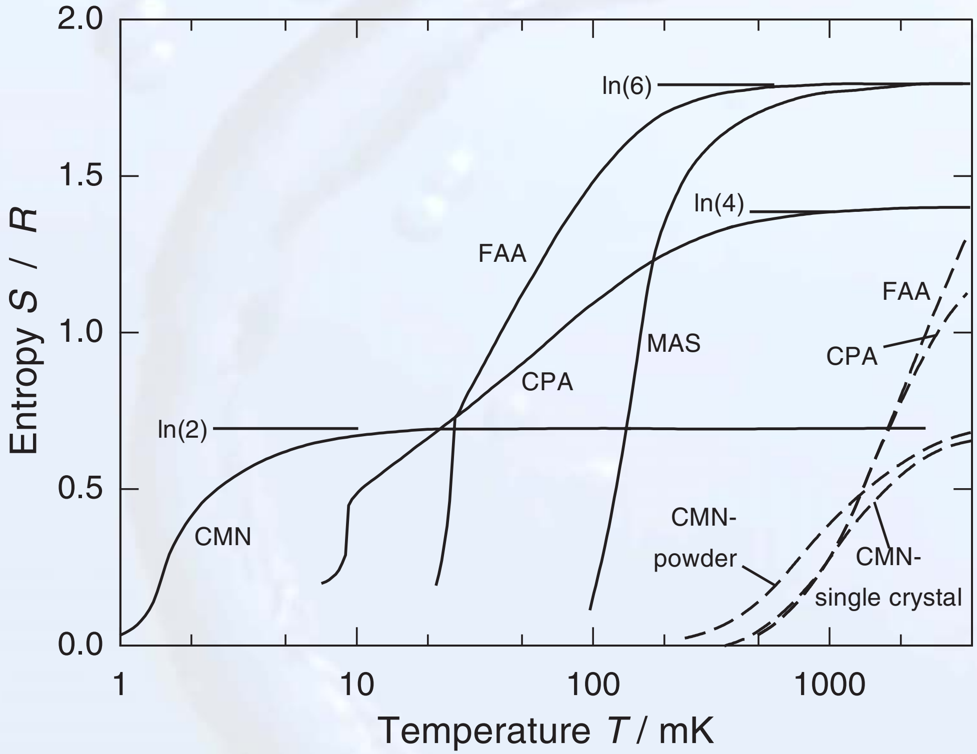
$$T_f = T_i \sqrt{\left( \frac{B_f^2 + B_{int}^2}{B_i^2 + B_{int}^2} \right)}$$

$$\Delta Q_{spin}(B_C) = \int_{T_C}^{T_A} C_{spin}\, dT = \int_{T_C}^{T_A} T \left( \frac{\partial S}{\partial T} \right)_{B_C} dT$$

# 11.3 Adiabatic Demagnetization Refrigerators

## a) Electronic spins

entropy of different paramagnetic salts




MAS for $MnSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$

FAA for $Fe_2(SO_4)_3 \cdot (NH_4)_2SO_4 \cdot 24H_2O$

CPA for $Cr_2(SO_4)_3 \cdot K_2SO_4 \cdot 24H_2O$

CMN for $2Ce(NO_3)_3 \cdot 3Mg(NO_3)_2 \cdot 24H_2O$

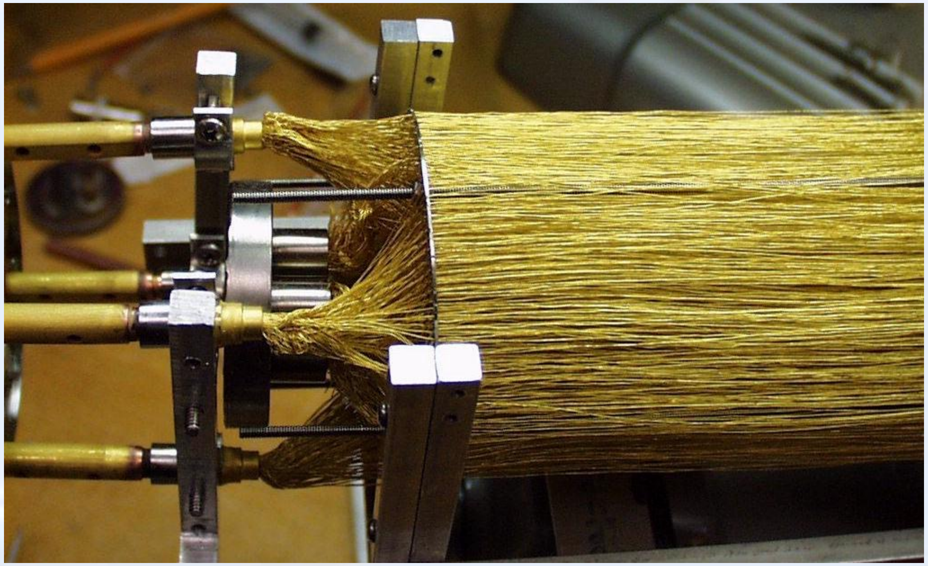
problems with paramagnetic salts → $T_c$ relatively high
→ low thermal conductivity

# 11.3 Adiabatic Demagnetization Refrigerators

high conductive wires to improve low thermal conductivity of salt pills

NASA GSFC

- FAA salt pill for space application
- 15.000 gold wires
- salt pill grown around the wires



# 11.3 Adiabatic Demagnetization Refrigerators

## b) Nuclear spins

$$\frac{\mathrm{d}T_\mathrm{n}^{-1}}{\mathrm{d}t} = -\frac{\left(T_\mathrm{n}^{-1} - T_\mathrm{e}^{-1}\right)}{\tau_1}$$

$\tau = \kappa / T_\mathrm{e}$ Korringa relation

- metals with fast relaxation time
- nuclei with large magnetic moment
- isotopes with large natural abundance
- cubic structure to avoid quadropole contributions
- no superconductor
- pure material, easy to machine

| | Structure | $I$ | $\mu/\mu_\mathrm{N}$ | $\kappa$ (K s) | Abundance (%) |
|---|---|---|---|---|---|
| $^{63}$Cu | fcc | 3/2 | 2.22 | 1.27 | 69.1 |
| $^{65}$Cu | fcc | 3/2 | 2.38 | 1.09 | 30.9 |
| $^{195}$Pt | fcc | 1/2 | 0.597 | 0.03 | 33.8 |
| $^{141}$PrNi$_5$ | fcc | 5/2 | 4.28 | <0.001 | 100 |

van Vleck paramagnet

# 11.3 Adiabatic Demagnetization Refrigerators

Gas gap heat switch

exchange gas
→ pumping to open switch

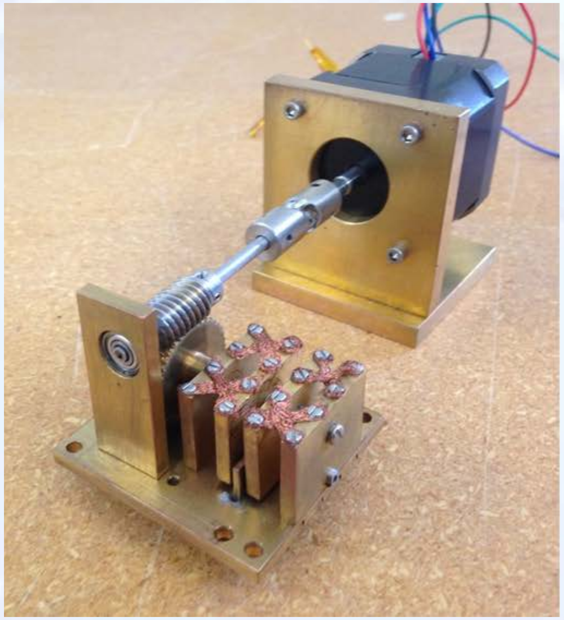
$^4$He: superfluid layer → creep

$H_2$: ortho-para conversion

$^3$He: no exothermic reaction
no creep
high vapor pressure

→ ideal exchange gas

Mechanical heat switch



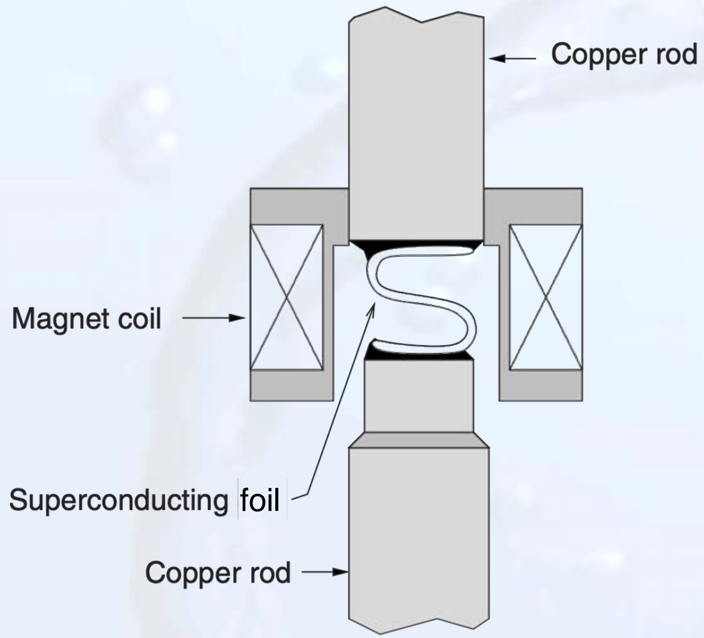
- large force needed ~ 100 N
- closed: mW/K … 1 W/K @ 15K
- problem: heating on opening

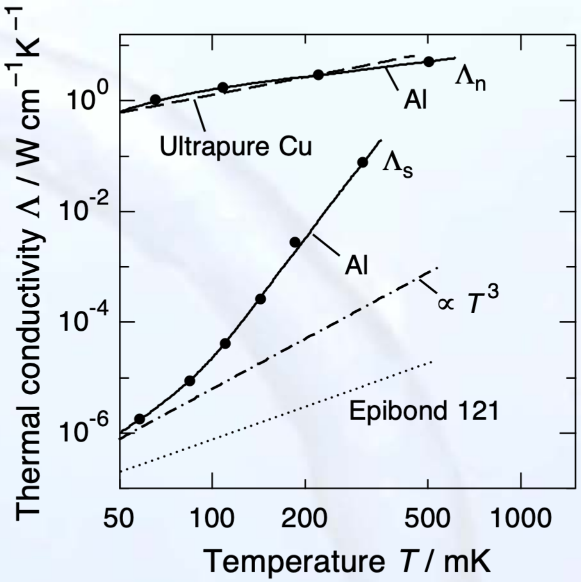
Superconducting heat switch

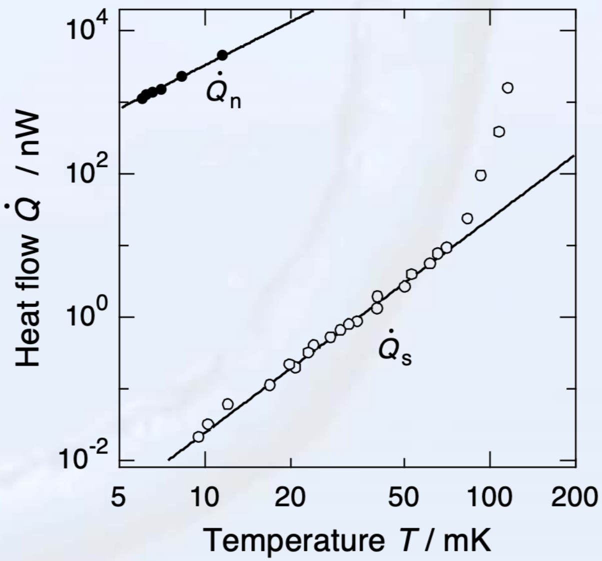
- only good well below $T_c$
- open means low conductivity
- problems: eddy currents
  flux trapping

# 11.3 Adiabatic Demagnetization Refrigerators

## Performance of superconducting heat switch




- switching ratio $10^6$ at 10 mK
- heat leak in open state 10 pW

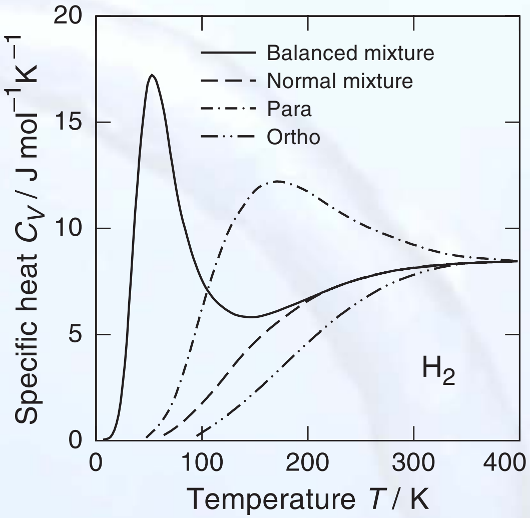
# 11.3 Adiabatic Demagnetization Refrigerators

## Heat leaks

- eddy current heating $\dot{Q}_{\rm eddy} = f\frac{V\dot{B}^2}{\varrho}$
- em fields and vibrations
- ortho-para conversion
- radioactive impurities
- tunneling systems

} time dependent heat leaks (ortho-para conversion, radioactive impurities, tunneling systems)

### specific heat of $H_2$




singlet state

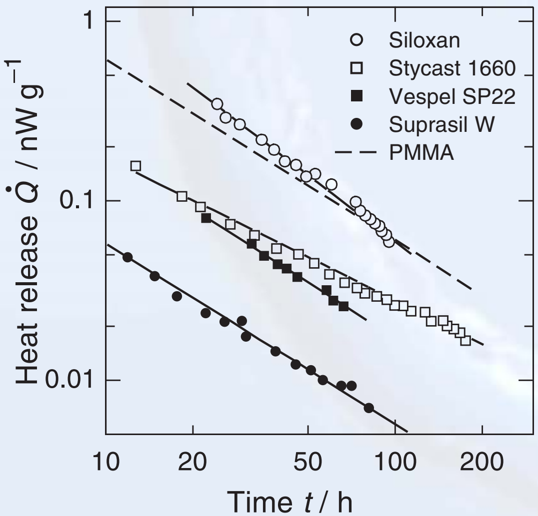
para $H_2$ $j = 0$

triplet state

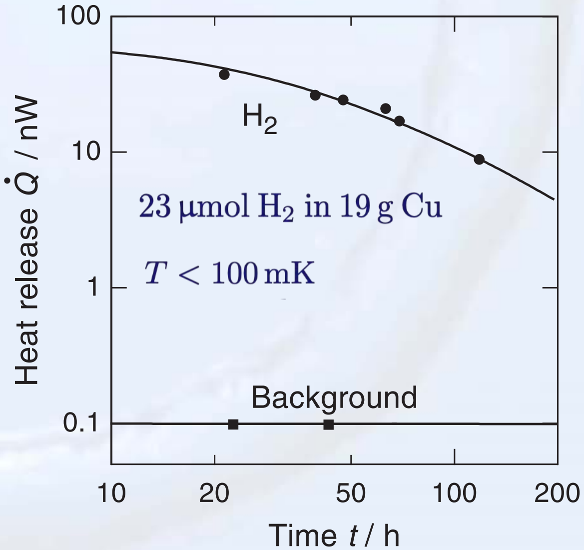
ortho $H_2$ $j = 1$

### atomic tunneling systems

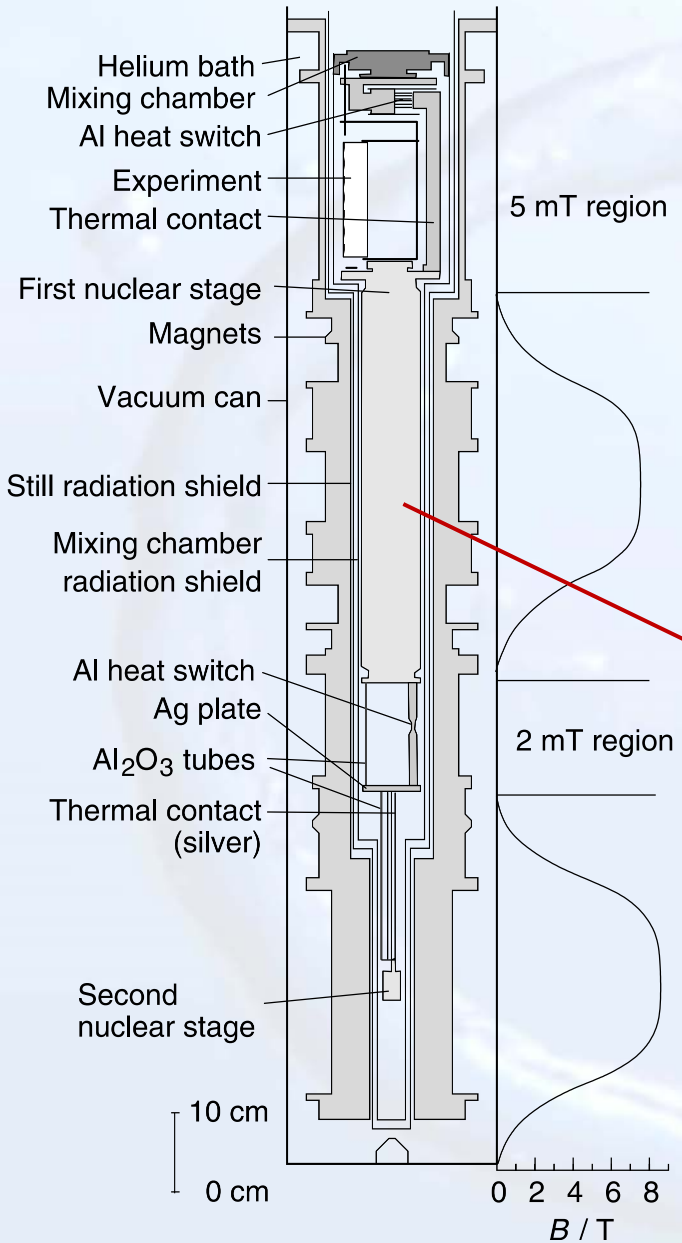
$$\dot{Q} = \frac{\pi^2 k_{\rm B}^2}{24} P_0 \left(T_1^2 - T_0^2\right) \frac{1}{t}$$

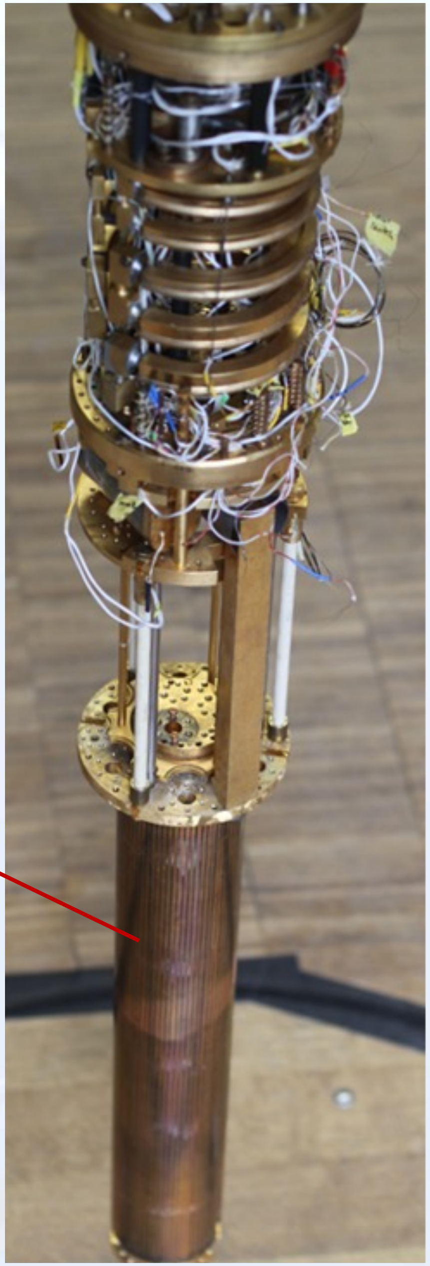



### $H_2$ ortho-para conversion



# 11.3 Adiabatic Demagnetization Refrigerators



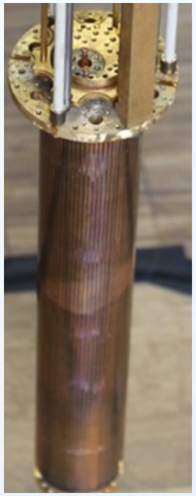
# 11.3 Adiabatic Demagnetization Refrigerators

## Cooling process

- precooling to $T_A$ and isothermal magnetization

nuclear Curie constant $\lambda_n = \dfrac{nI(I+1)\mu_0\mu_n^2 g_n^2}{3k_B}$

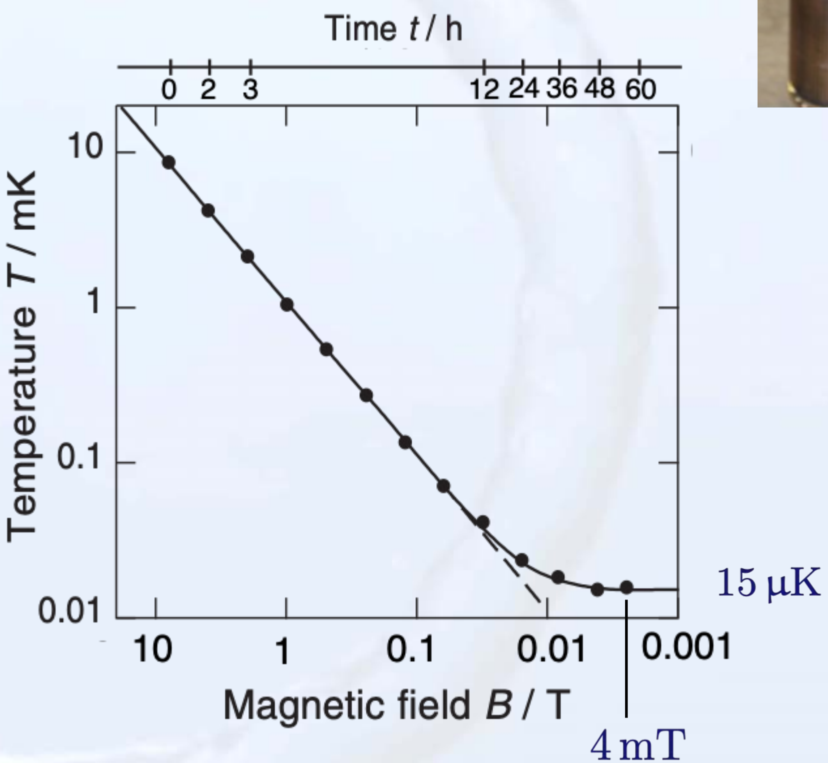
$$\longrightarrow \quad Q = nT_A\Delta S = -\frac{\lambda_n B_i^2}{2\mu_0 T_A}$$



- reducing $B$ in steps to optimal final field

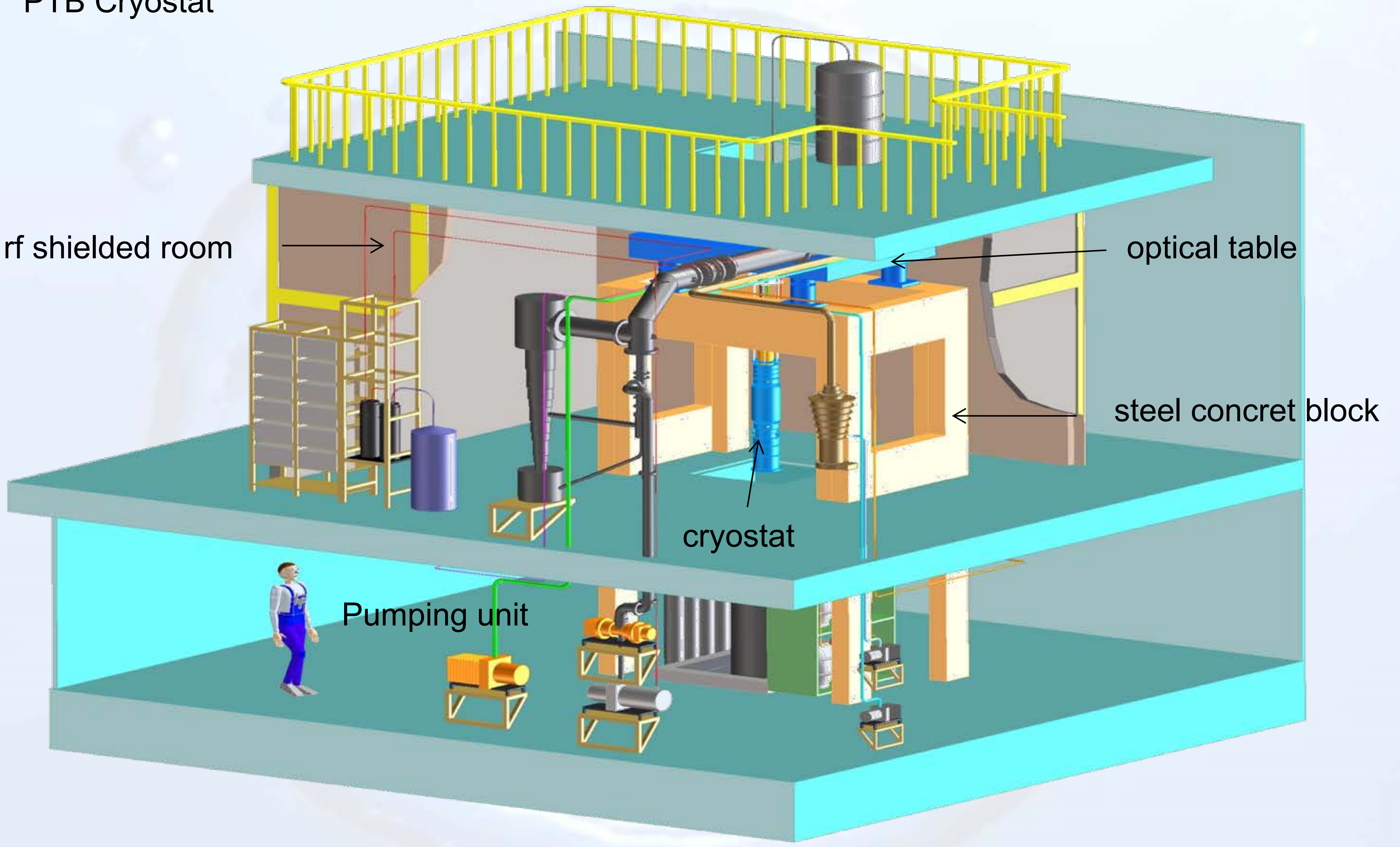
$$B_{f,opt} = \sqrt{\frac{3k_B\kappa\dot{Q}}{n g_n^2 I(I+1)\mu_n^2}}$$

heat leak: $\dot{Q}$



# 11.3 Adiabatic Demagnetization Refrigerators

PTB Cryostat

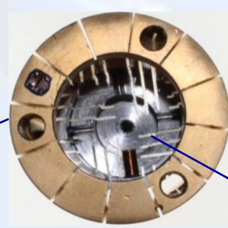


# 11.3 Adiabatic Demagnetization Refrigerators

heat switch

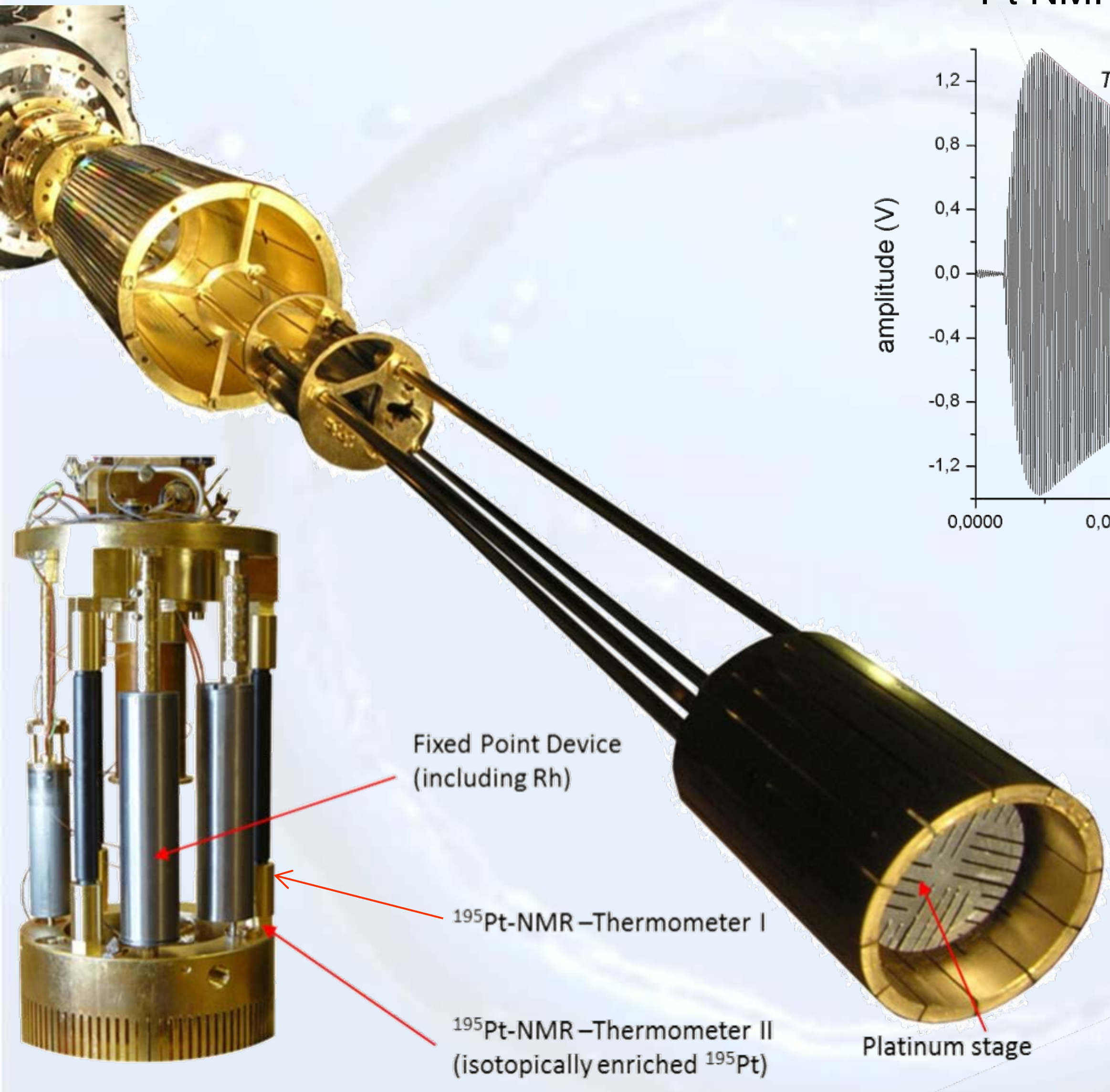
Cu stage

heat switch

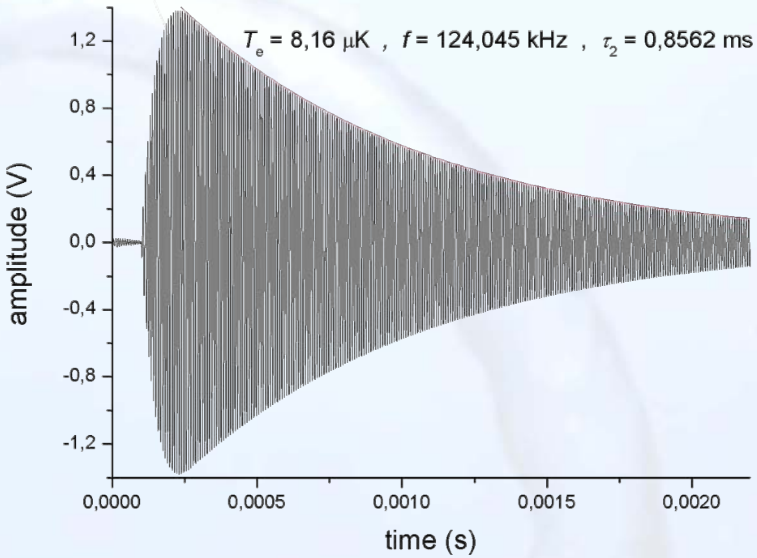


Pt stage

# 11.3 Adiabatic Demagnetization Refrigerators




$^{195}$Pt NMR thermometer signal



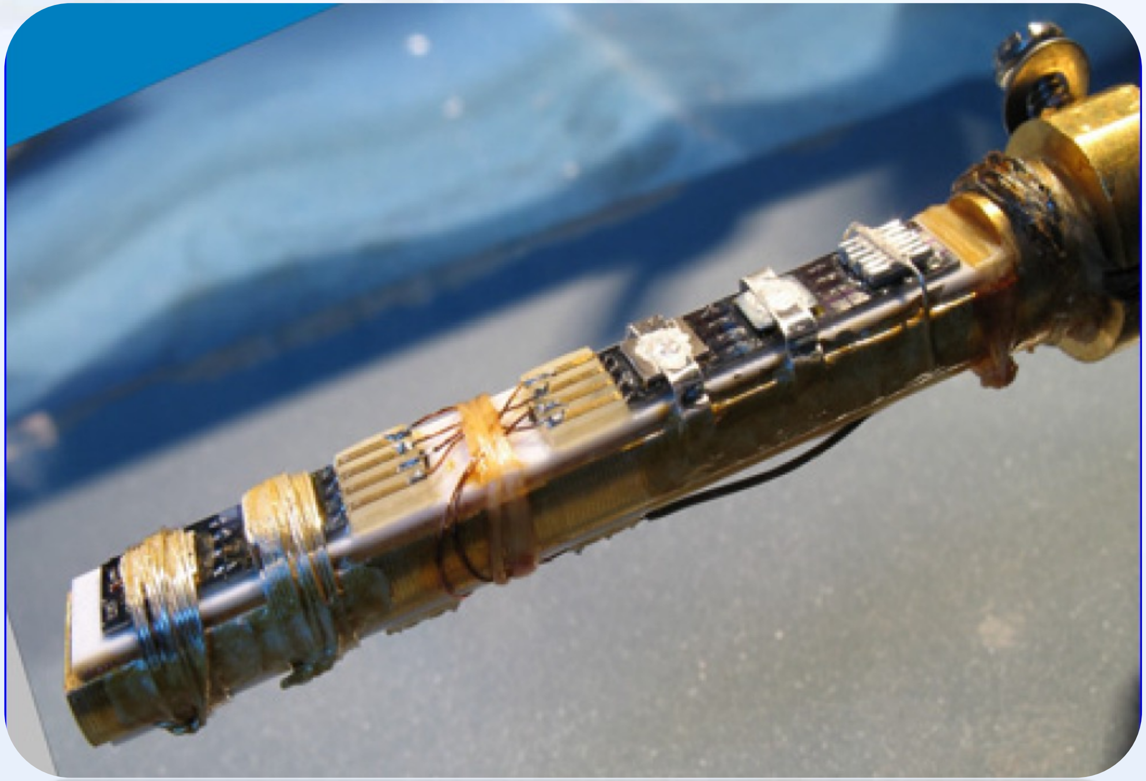

Lowest temperature at Pt stage

$T_{min}$ = 800 nK

# 12. Thermometry at Low Temperature

**Primary thermometers**
Superconducting fixpoints
Current/flux noise
$^{195}$Pt NMR
Coulomb blockade
Nuclear orientation
$^{3}$He melting curve
....

**Secondary thermometers**
Resistance
Capacitance
Magnetic susceptibility
.....



# 12. Thermometry at Low Temperature

Temperature is a thermodynamic property of state

It can be defined by a reversible cycle, like a carnot cycle $\oint T^{-1}\mathrm{d}Q = 0$

not practical

primary thermometers: can be used without any prior calibration

secondary thermometers: must be calibrated against an other thermometer

distinction is often somewhat arbitrary …

## Temperature scales

defined by *Comité International des Poids et Messures*

based on fixpoints like the triple point of water
and interpolation like Pt-100 resistance thermometry
or gas thermometry

| | | | |
|---|---|---|---|
| ITS-90 | 0.65 K | to | 1358 K |
| PLTS-2000 | 0.9 mK | to | 1358 K |