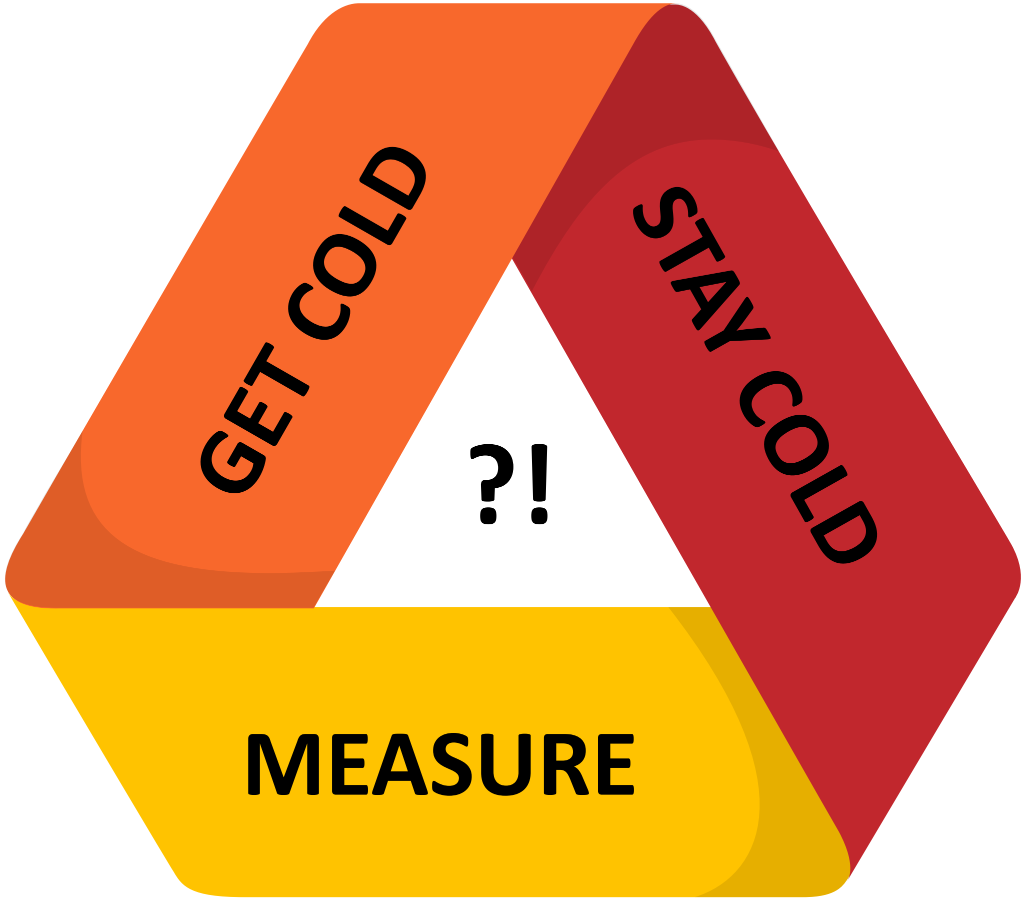
ICEC27-ICMC 2018
27th International Cryogenic Engineering Conference
International Cryogenic Materials Conference 2018
September 3-7 2018 Oxford England

Physics | Lancaster University

EMP

# Cryostat design below 1K

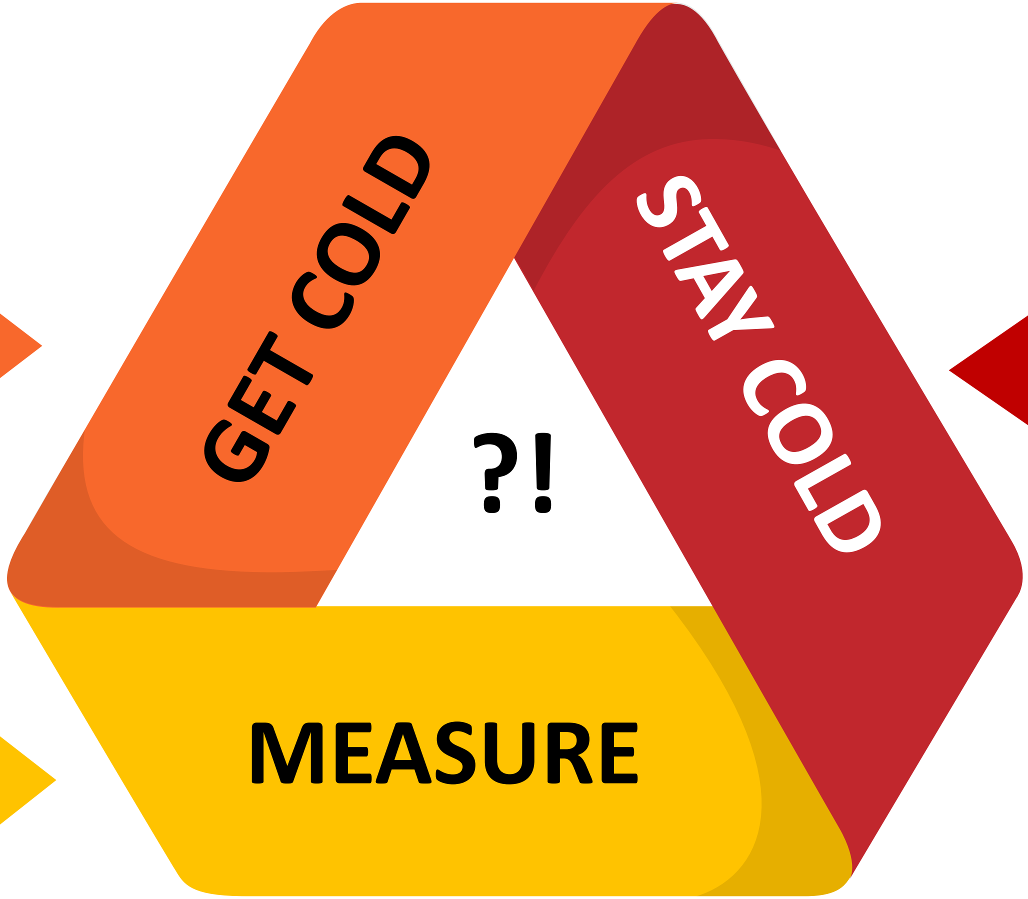



Dr. Viktor Tsepelin (v.tsepelin@lancaster.ac.uk)

Oxford, 3 September 2018

# Initial statement of problem

Choose a refrigerator that can get colder than the temperature needed by your experiment $T_E$




Choose required measurement setup and materials (optics, particle beam, AC, DC wiring, magnetic field, gravity…)

The refrigerator must have sufficient cooling power to absorb the power dissipated by the measurement $Q_E$ and still maintain $T_E$

Cool the sample through some thermal link:

- sample must cool in a reasonable time
- thermal contact must be good enough that $Q_E$ doesn't warm the experiment above $T_E$

Choose appropriate thermometer (resolution, stability, transferability, thermal contact, power dissipation of the thermometer )

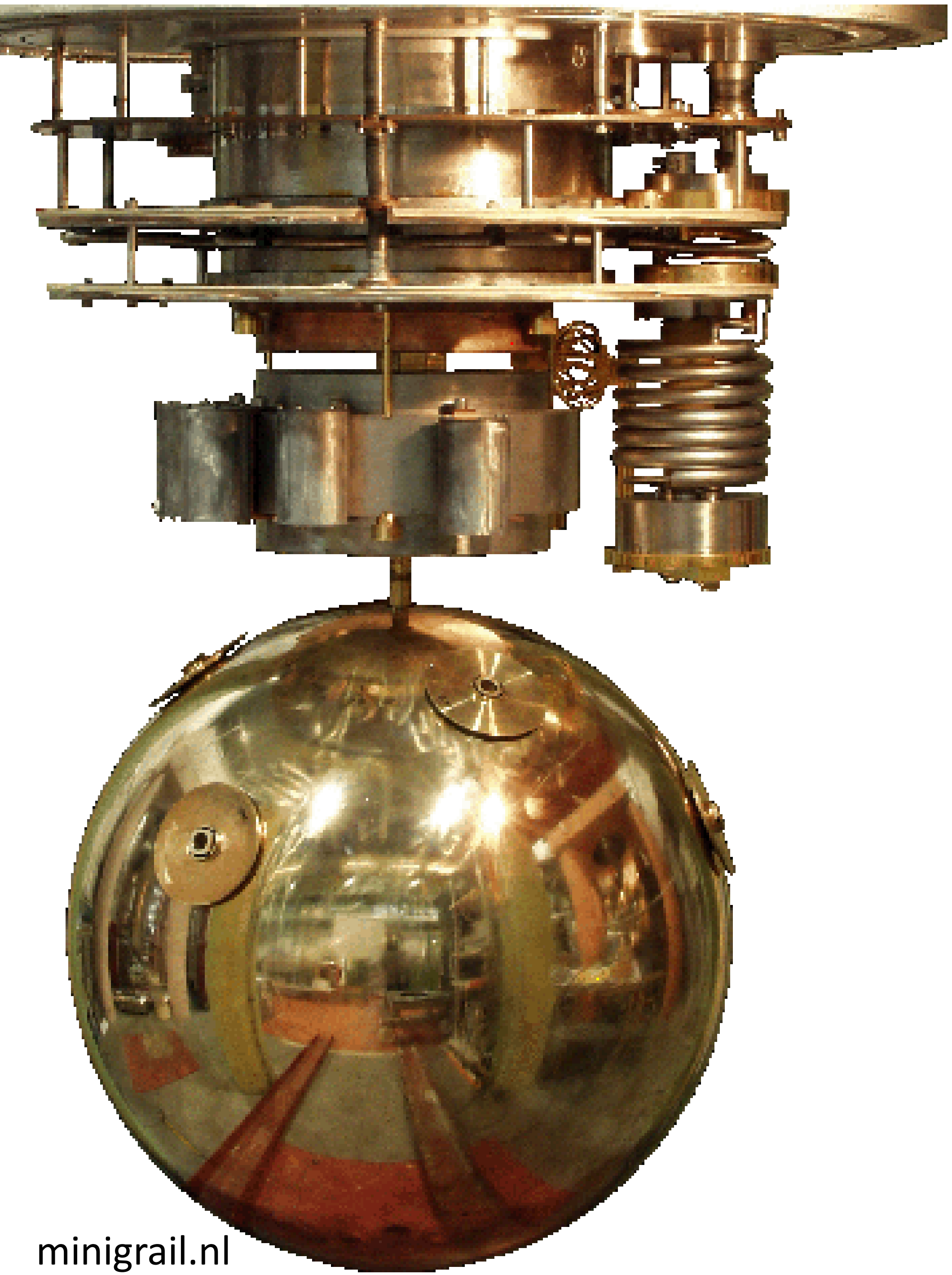
# Reasons to reach low temperatures

selective freezing of degrees of freedom
systems with small energies
low noise measurements
new phenomena, new technologies and modelling inaccessible systems

Superfluidity
Superconductivity
Magnetic ordering
Quantum Turbulence

Quantum Metrology
Quantum Computing
Cryogenic Particle Detectors

Cosmic Strings
The Early Universe
Neutron Stars



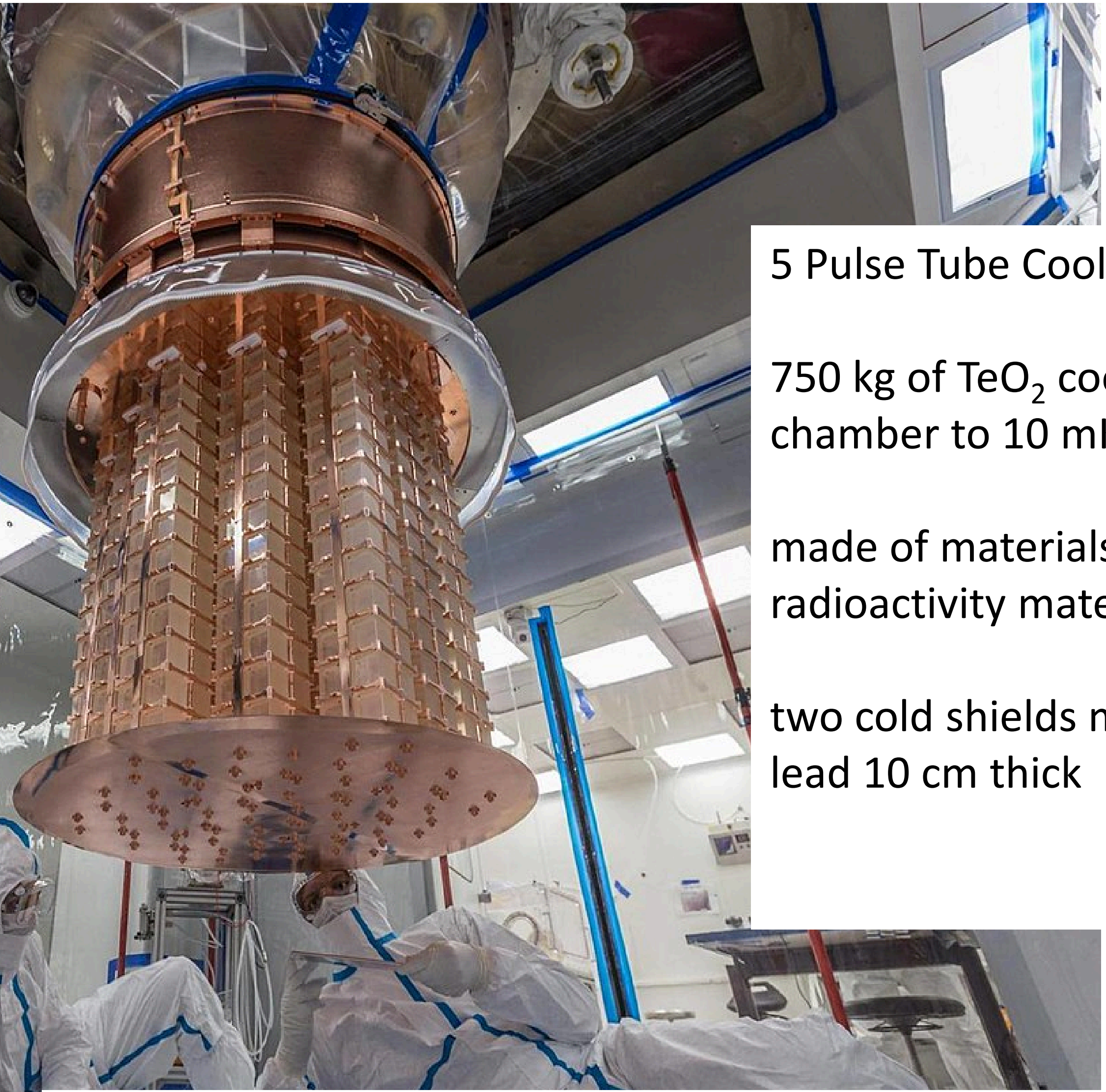
minigrail.nl

# MiniGRAIL - Gravitational Radiation Antenna In Leiden

- The MiniGRAIL detector is a 68 cm diameter spherical gravitational wave antenna made of CuAl (6%) alloy.
- Mass of 1400 Kg.
- The antenna will operate at a temperature of 20 mK.

Leiden Cryogenics
Leader in Low Temperature Techniques

# Cuore - Cryogenic Underground Observatory for Rare Events

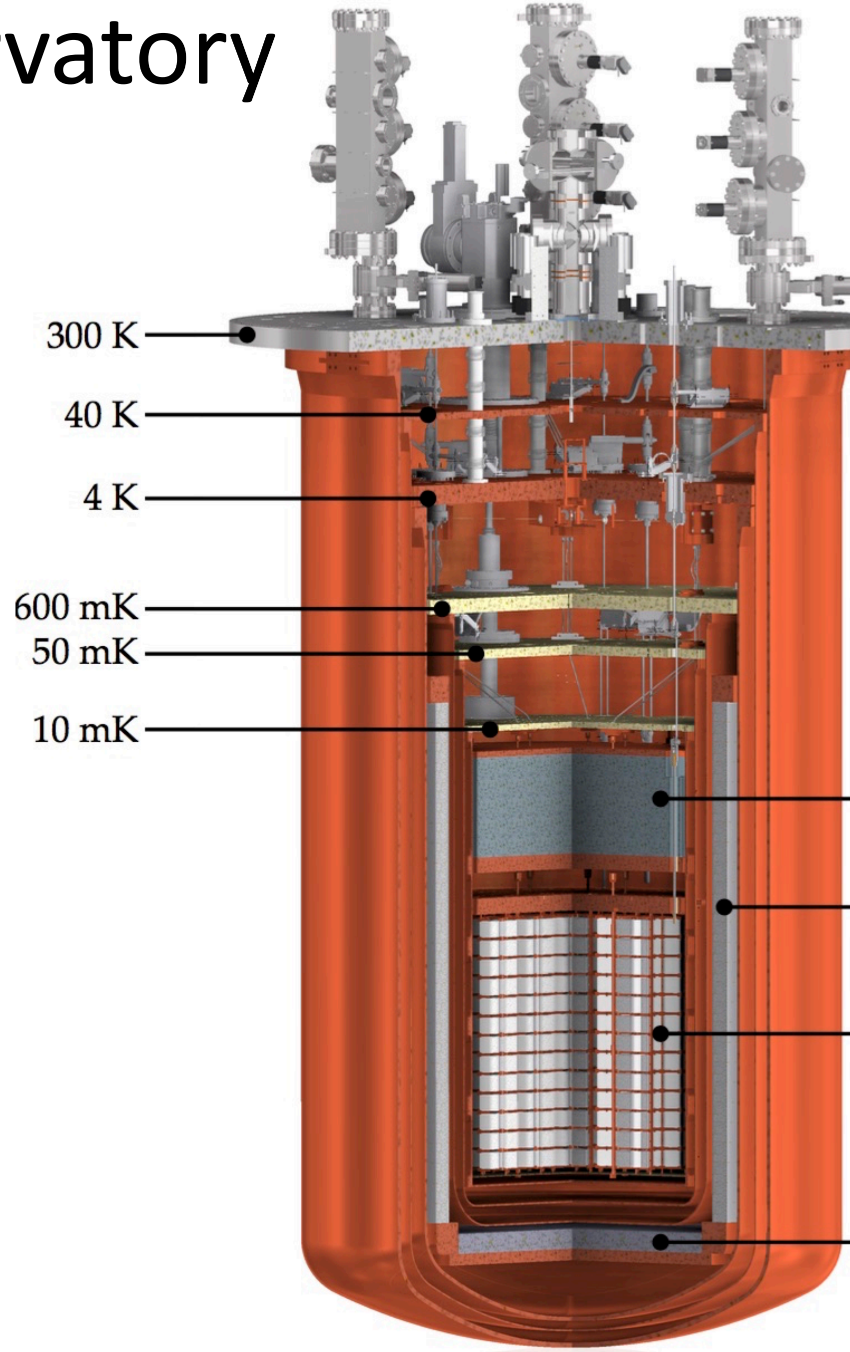


5 Pulse Tube Coolers

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



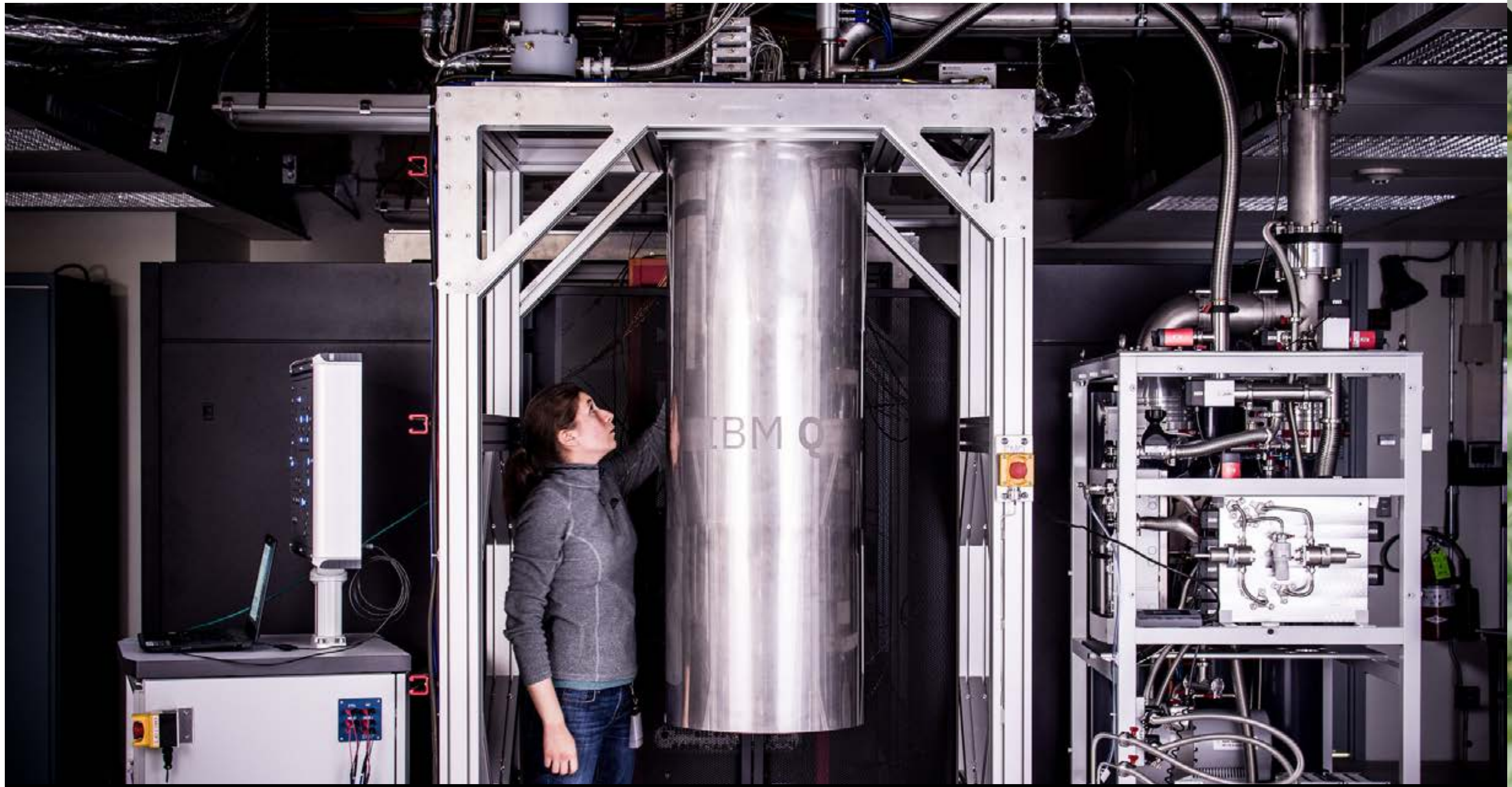

By Davide italy - https://commons.wikimedia.org/w/index.php?curid=63562559

cuore.lngs.infn.it/en/about/cryostat

# ROTA – rotating cryostat for superfluid $^3$He research

- Adiabatic Nuclear Demagnetisation Cryostat (combination of dilution refrigerator and nuclear cooling stage)
- Superfluid $^3$He temperature of 0.15Tc (~140$\mu$K at zero bar pressure)
- Rotation velocity of ~4 rad/s
- Has a cryopump to collect $^3$He-$^4$He mixture during rotation

# The cryostat for a 50 qubit quantum computer

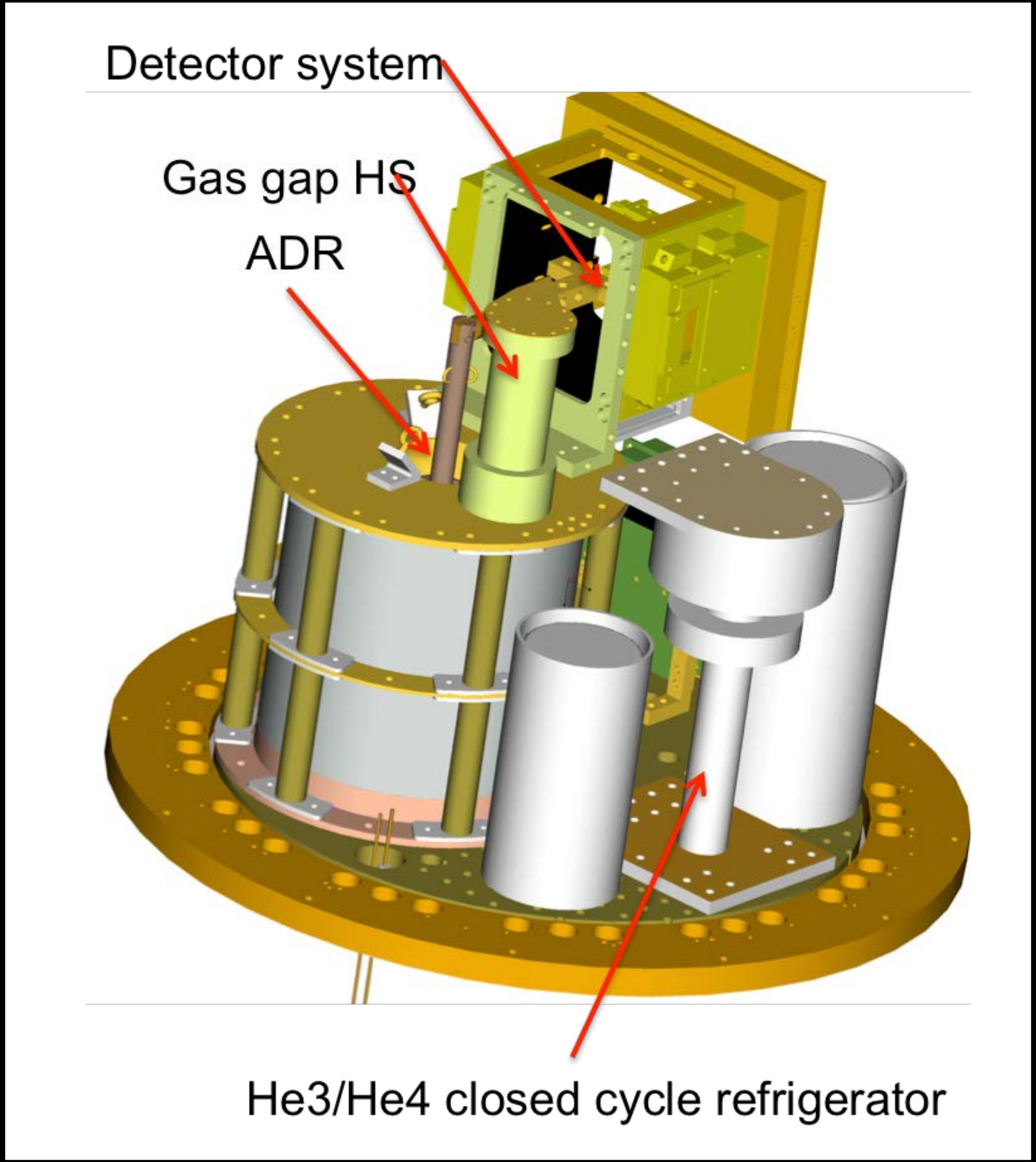


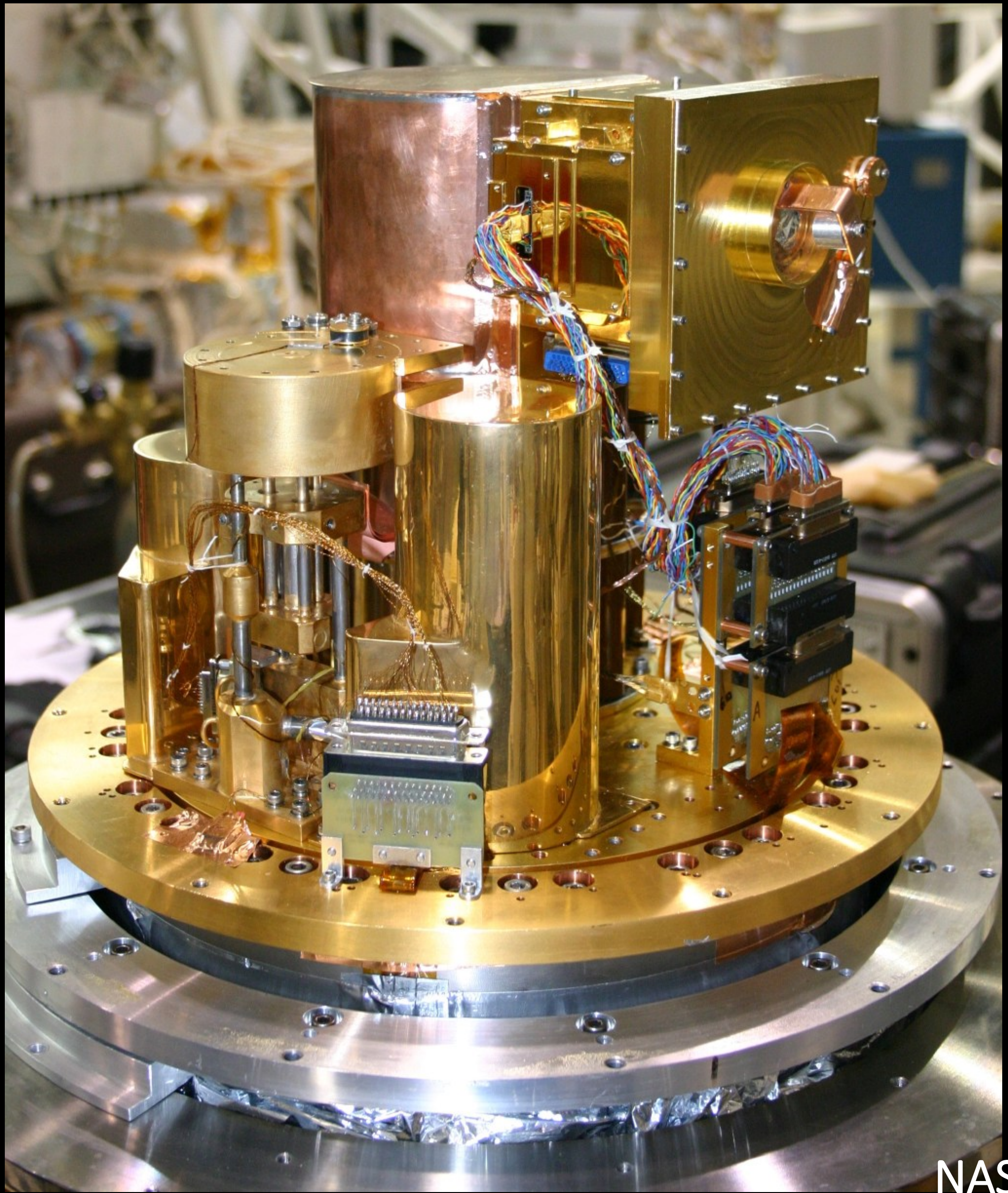
An IBM quantum computer cryostat (Image: Andy Aaron, IBM Research/Flickr)


IBM Research/Flickr

11/10/2017 https://gizmodo.com/ibm-announces-two-fancy-new-quantum-computers-1820311244

# Fully Automatic ADR for Space Missions (50 mK)



NASA GSFC

# Temperature Range and Cooling Methods

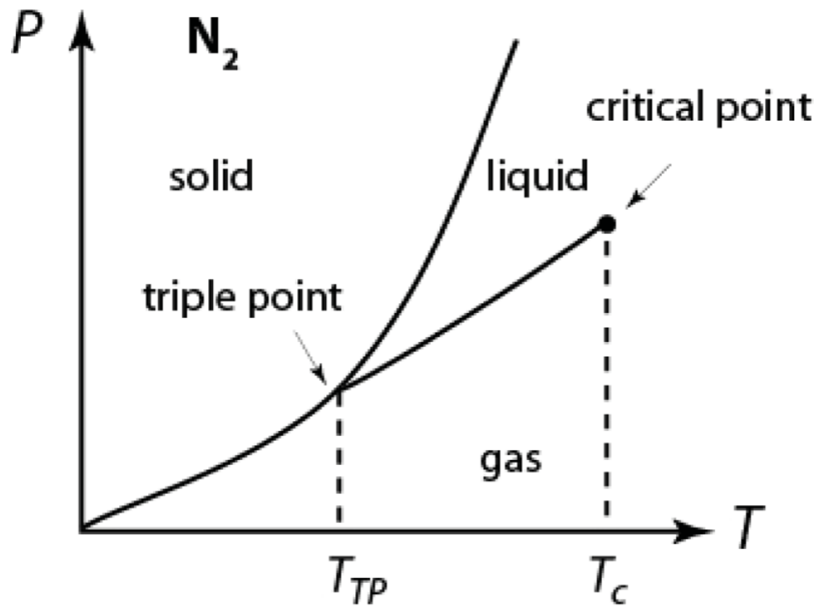
- $T > 0.9$ K — $^4$He evaporation cryostat (continuous)
- $T > 0.25$ K — $^3$He evaporation cryostat (continuous)
- $1.0 > T > 0.003$ K — $^3$He - $^4$He dilution refrigerator (continuous)
- $1.0 > T > 0.04$K — Adiabatic electron Demagnetisation Refrigerators (ADR)
- $T < 0.003$ K — Adiabatic nuclear demagnetization refrigerators

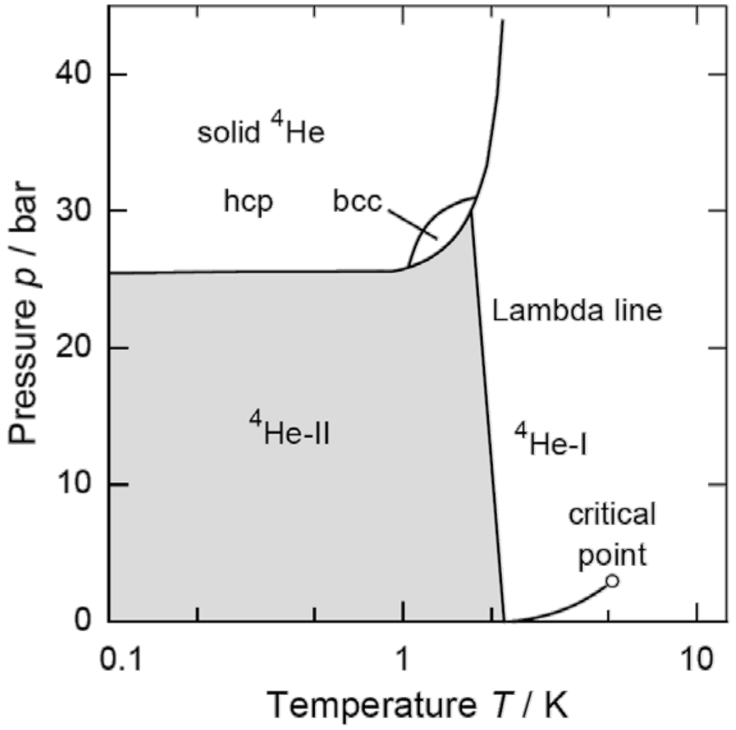
$^3$He cryostats, $^3$He - $^4$He dilution refrigerator & ADR's available commercially: Bluefors, Cryoconcept, Cryogenic, High Precision Devices, Janis, Leiden Cryogenic, Oxford Instruments, etc …

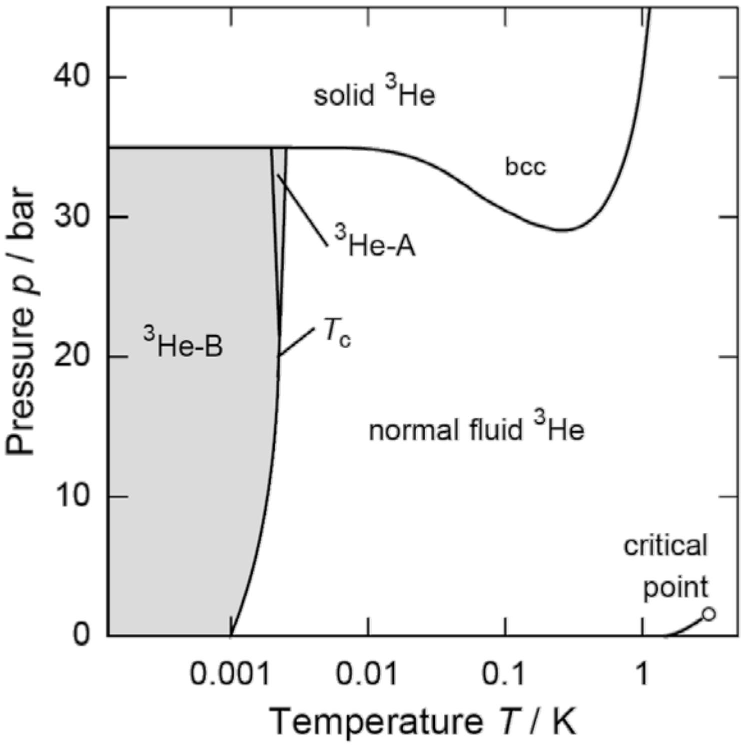
Other methods:

- Pomeranchuk cooling ($^3$He liquid-solid 1$^{st}$ order transition)
- Electron cooling for small samples

# Phase Diagrams: ordinary materials vs $^3$He and $^4$He




Almost everything behaves like this, apart from $^4$He and $^3$He

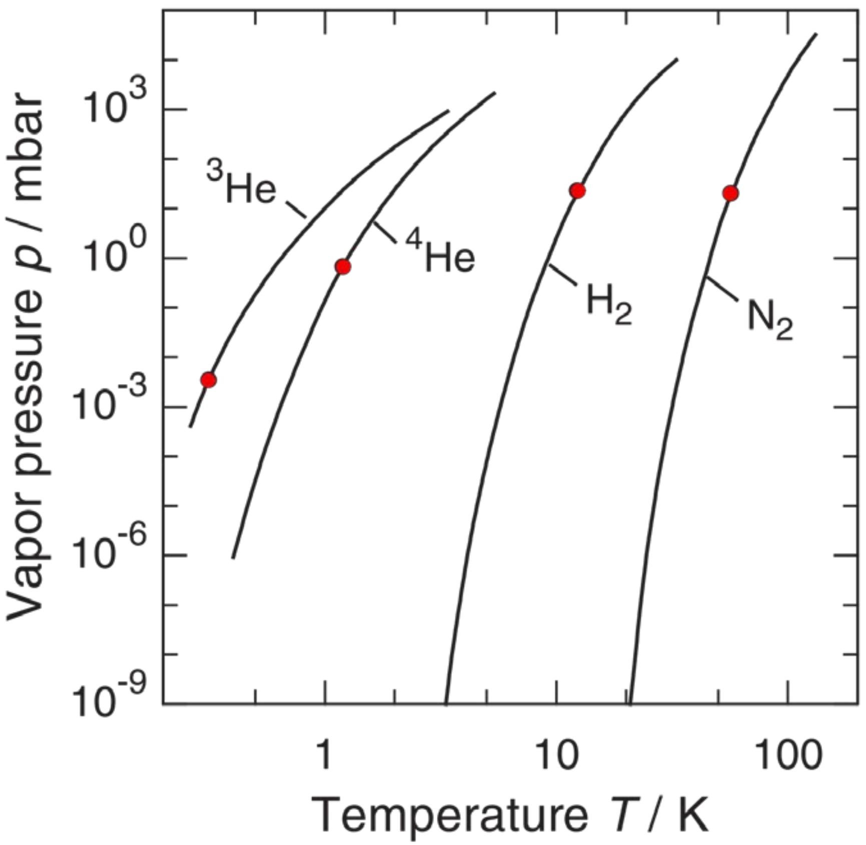
Similarities between $^4$He and $^3$He:

- Absence of triple point
- Critical point at low temperature
- High pressure to form solid
- Existence of superfluidity

Differences:

- Different statistics (Boson vs Fermi)
- $^3$He has magnetic field dependence
- Superfluidity has different origin
- Cost:
  - $^4$He £5-£10 per liquid litre
  - $^3$He £1.3M per liquid litre!

# $^4$He , $^3$He evaporation cryostats




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

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

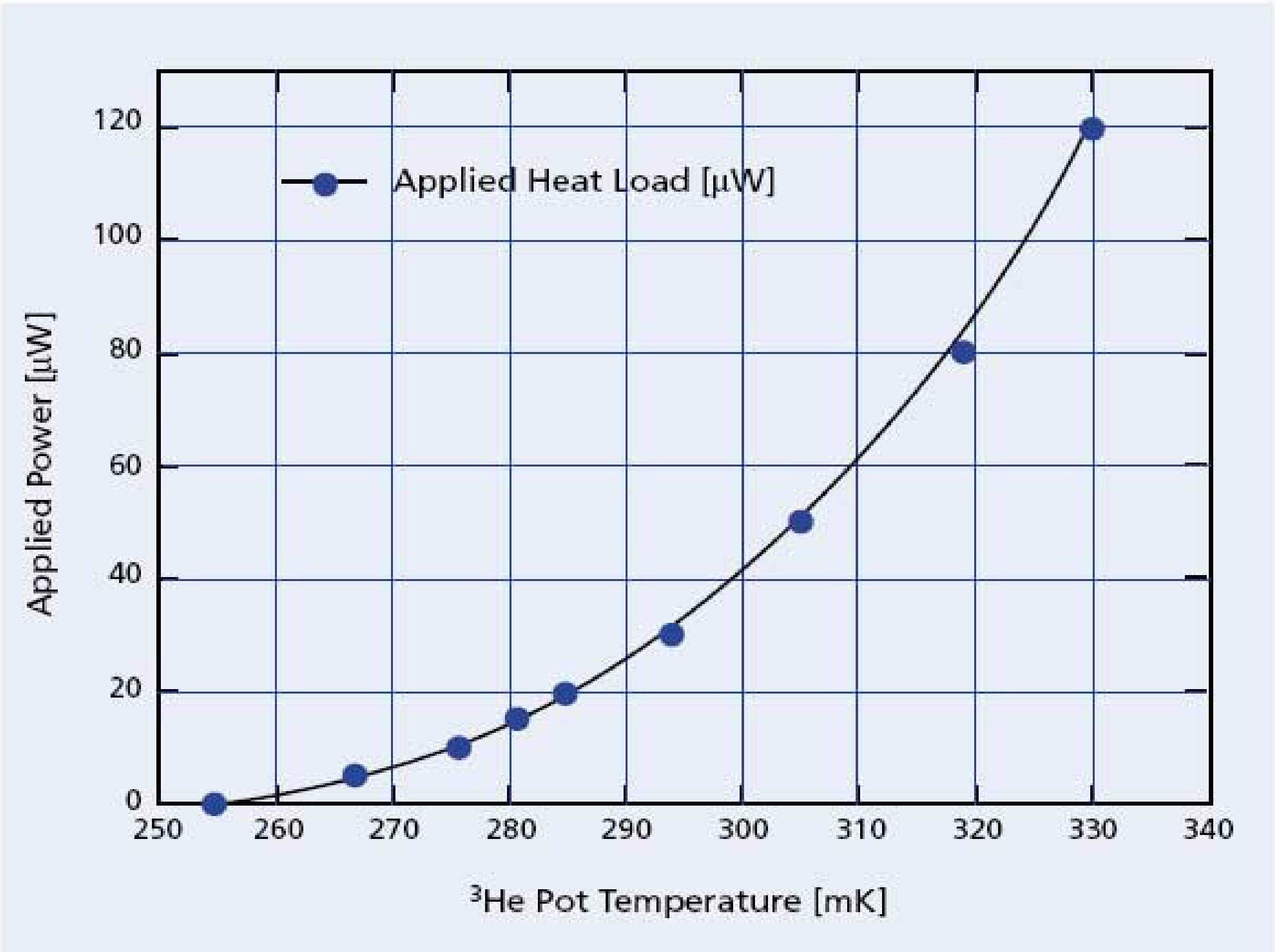
Cooling power $\propto$ mass flow across phase boundary $\propto P$.

$\therefore$ exponentially falling cooling power. $L/R$ = 2.5K limits $T_{base}$ to 0.2 - 0.3K

How large is change of cooling power when the operating temperature of a $^3$He cryostat changes from 0.3K to 0.2K?

How about from 0.2K to 0.1K?

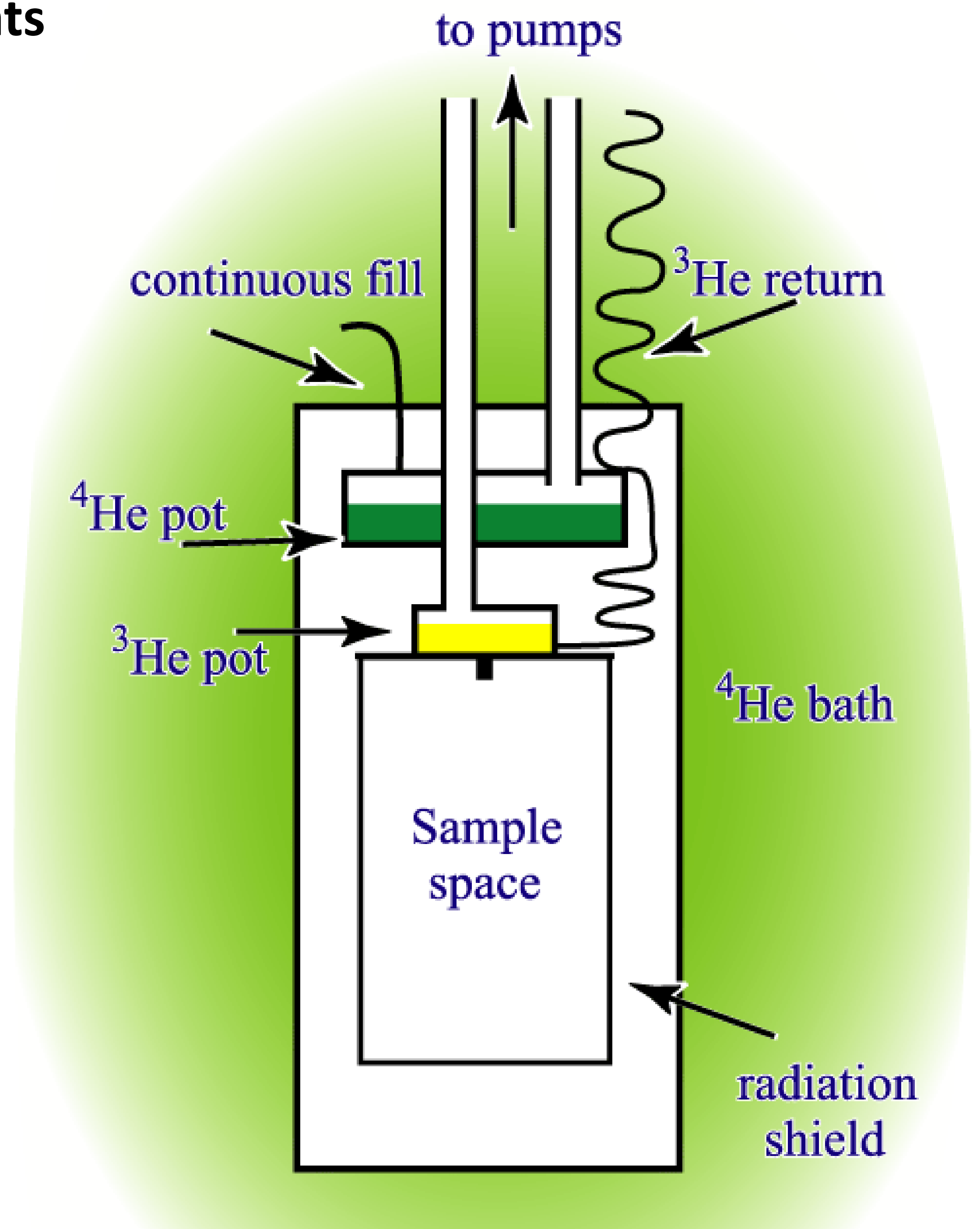
# $^3He$ evaporation cryostats



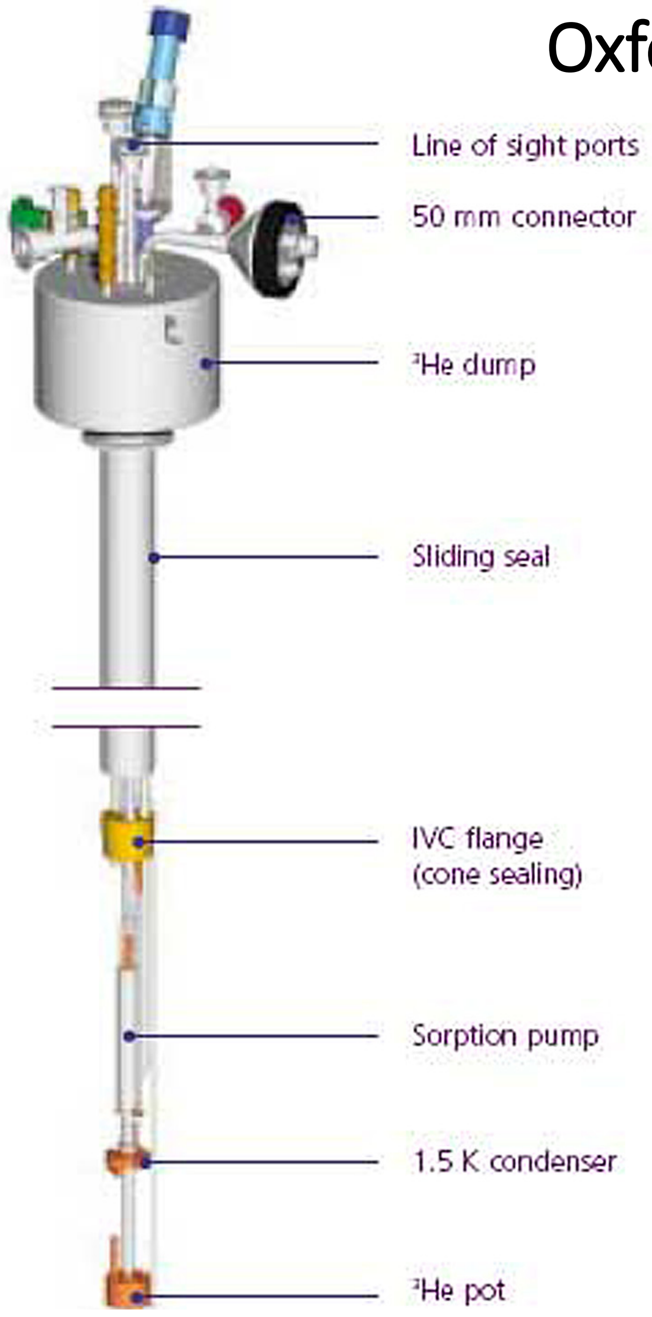

**Typical cooling power of the Oxford Instruments HelioxVL**

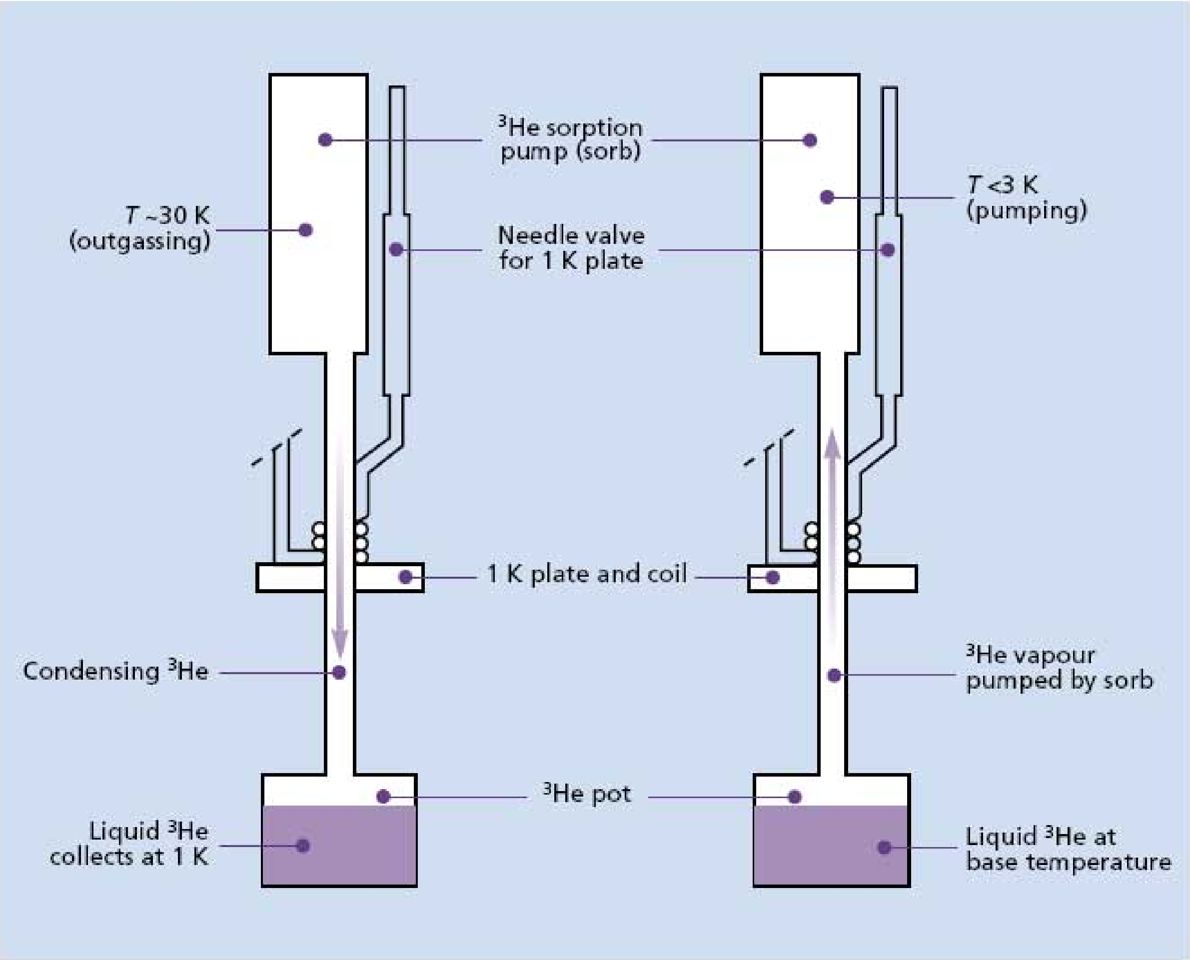
Base temperature: ≤ 245 mK for ≥ 90 hrs with no applied heat load

Cooling power: 40 μW at ≤ 290 mK, with a hold time > 10 hrs

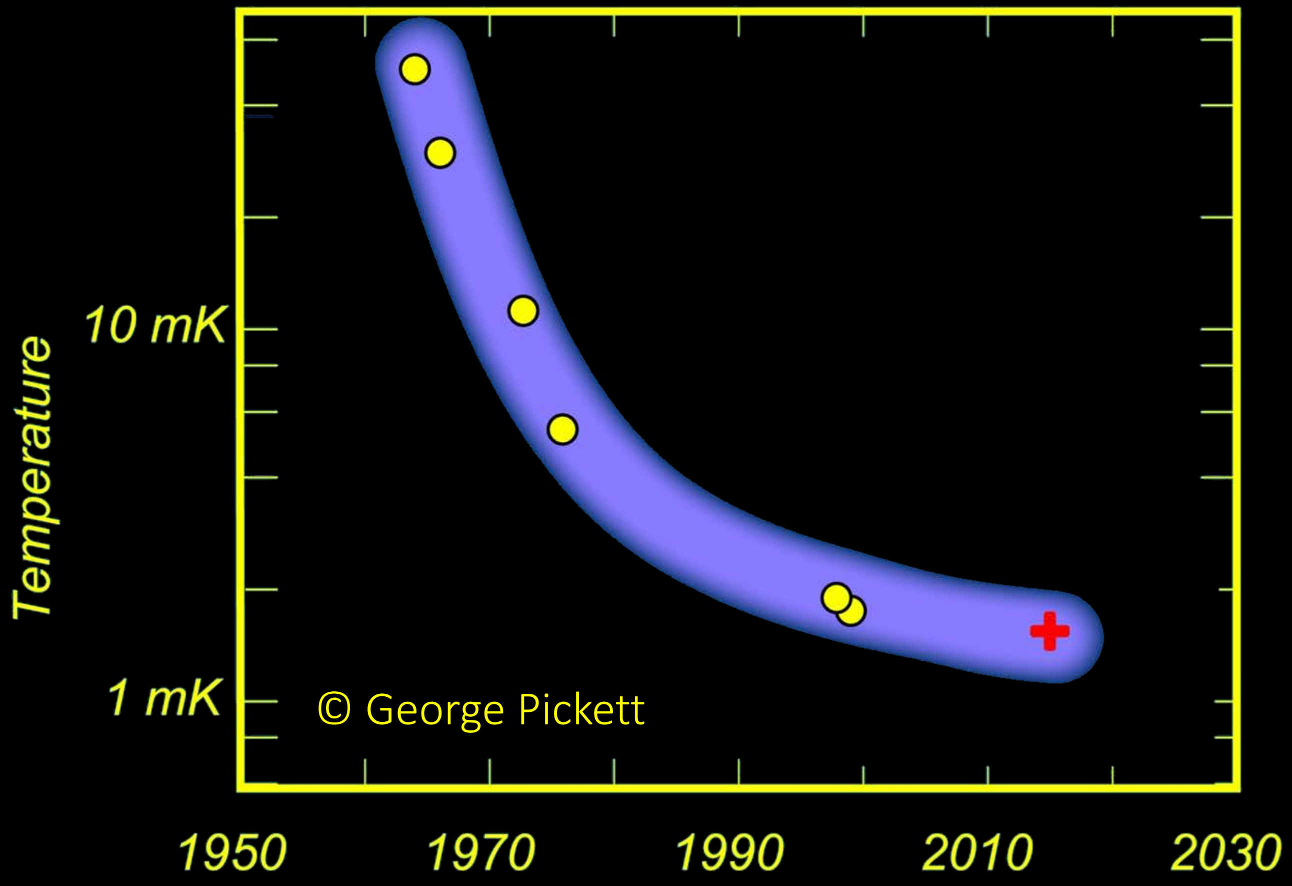


# Oxford Instruments Heliox system



# $^3He$ - $^4He$ dilution refrigerator: History and the Minimal Temperature




1951 Basic idea suggested by Heinz London

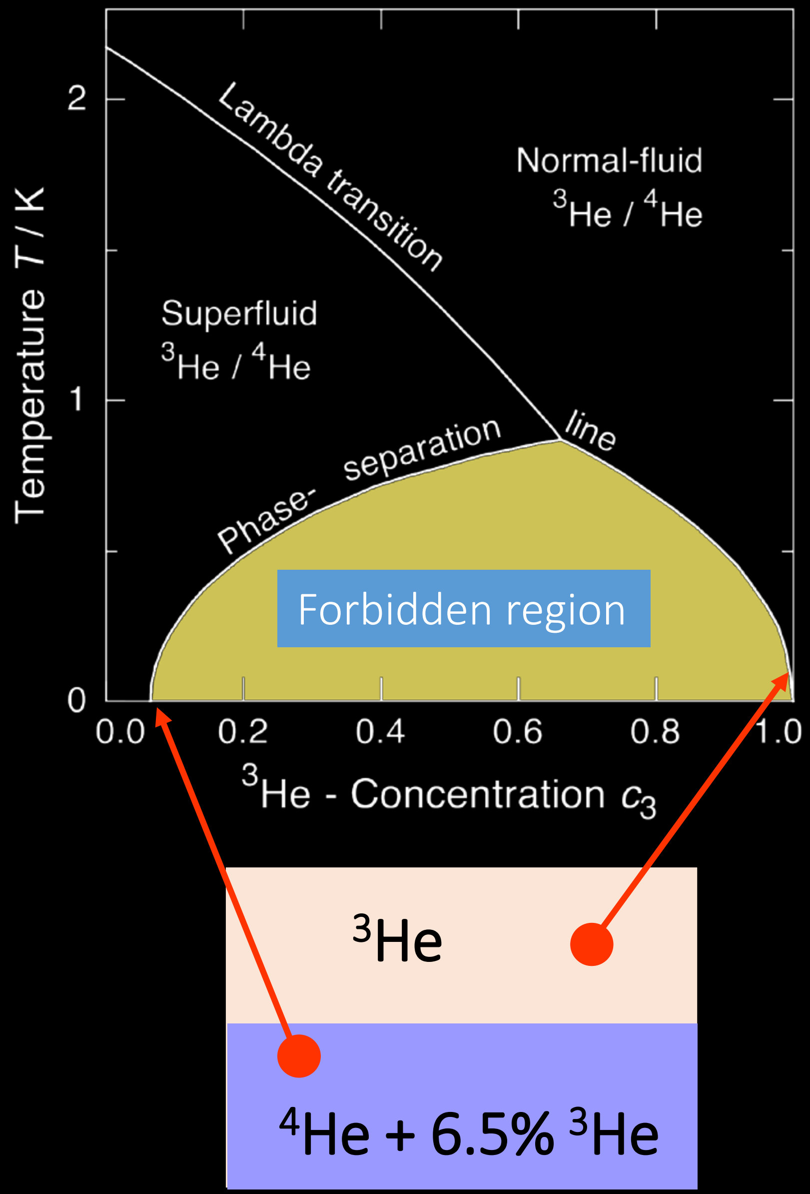
1962 Detailed Concept worked out by London, Clark, Mendoza

1965 First realisation Das, De Bruyn Ouboter, Taconis $T_{min}$ = 220 mK

1972 Single short, V. Edelman, T~60mK

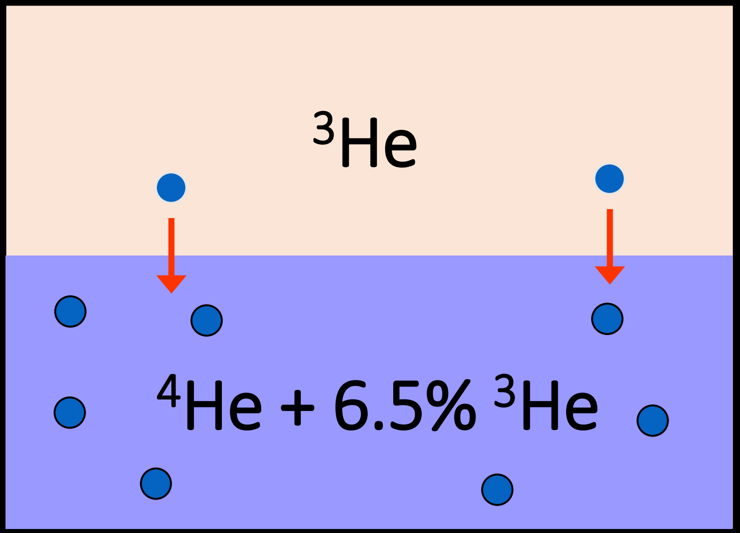
1999 Lowest Temperature obtained, J.C. Cousins *et al.*, $T_{min}$ = 1.75 mK

# $^3$He - $^4$He mixture:




# Principle of cooling is mixing $^3$He/$^4$He

Transition of $^3$He into the $^4$He rich phase



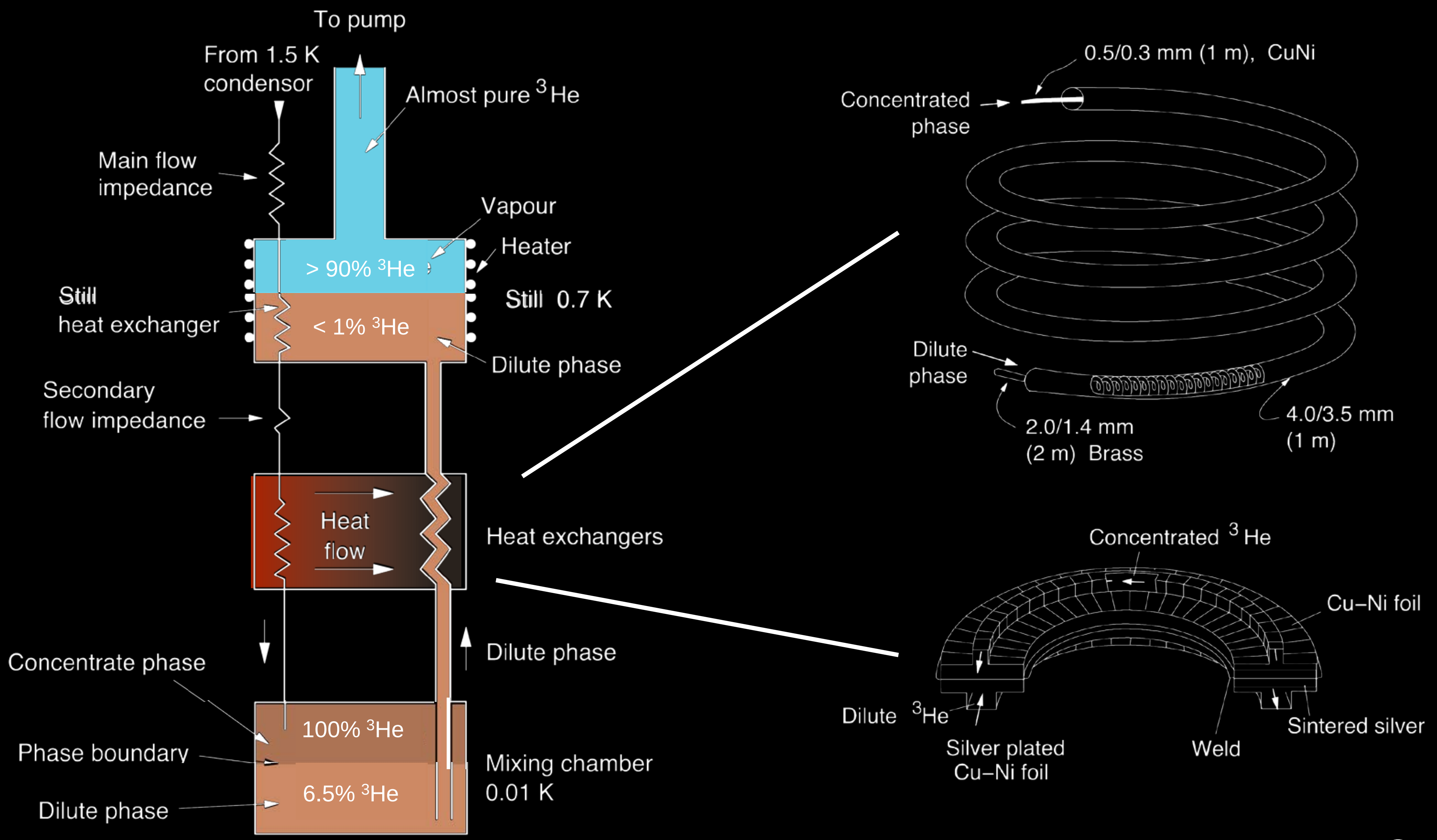

Cooling by evaporation of $^3$He into $^4$He quasi-vacuum

Cooling power $\propto$ $^3$He circulation rate $\dot{n}_3$ can be ~1000μmoles s$^{-1}$ with big pumps

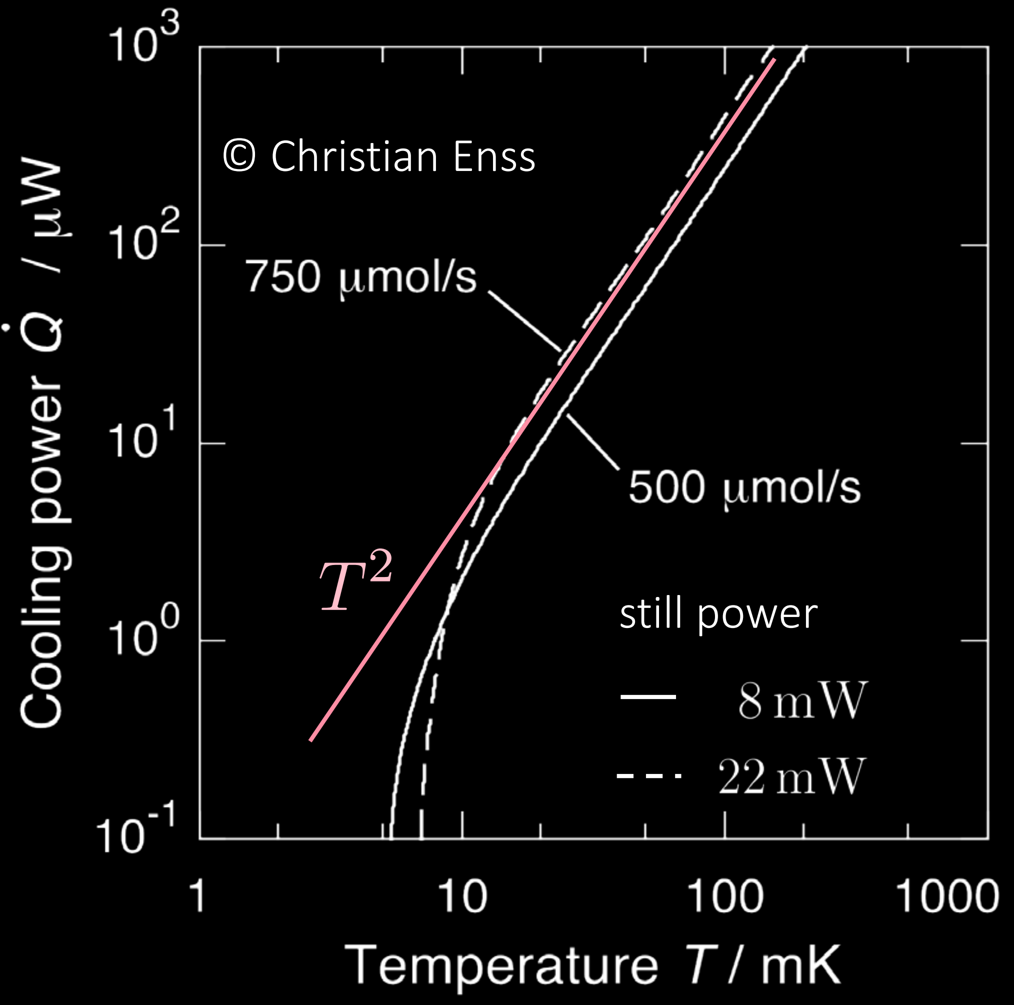
$$\dot{Q} \sim 80\dot{n}_3(T^2 - T_{base}^2)$$

http://clipart-library.com/clipart/1736015.htm

# Realisation of $^3He/^4He$ Cooling Cycle



# Minimum Temperature and Flow Dependence of Cooling Power




## Minimum Temperature

There is no principle limit … it is determined by the heat leak!

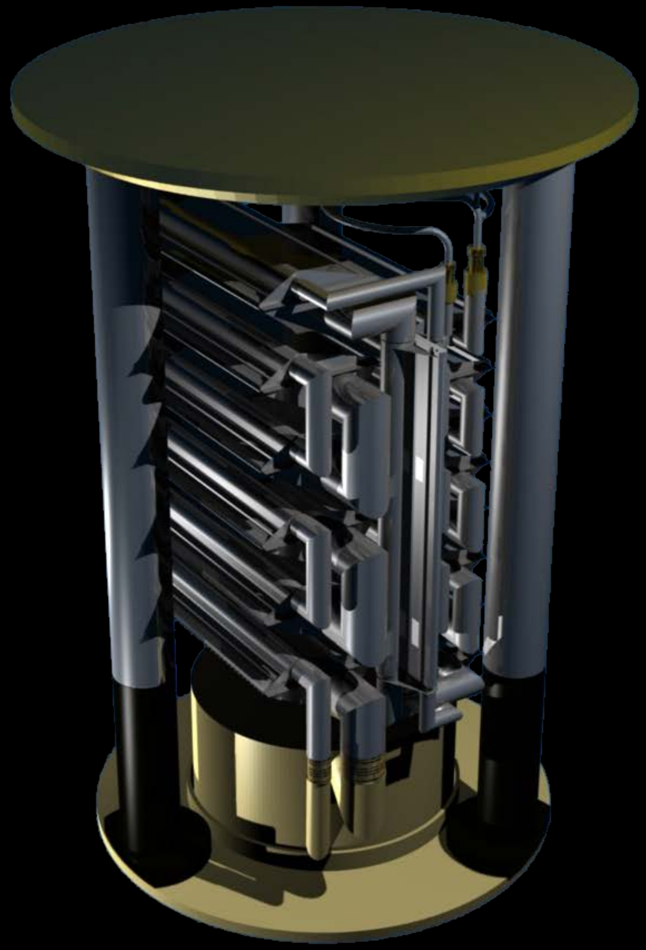
Limiting case of vanishing cooling power: $\dot{Q}_{\rm mc} = 0$

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

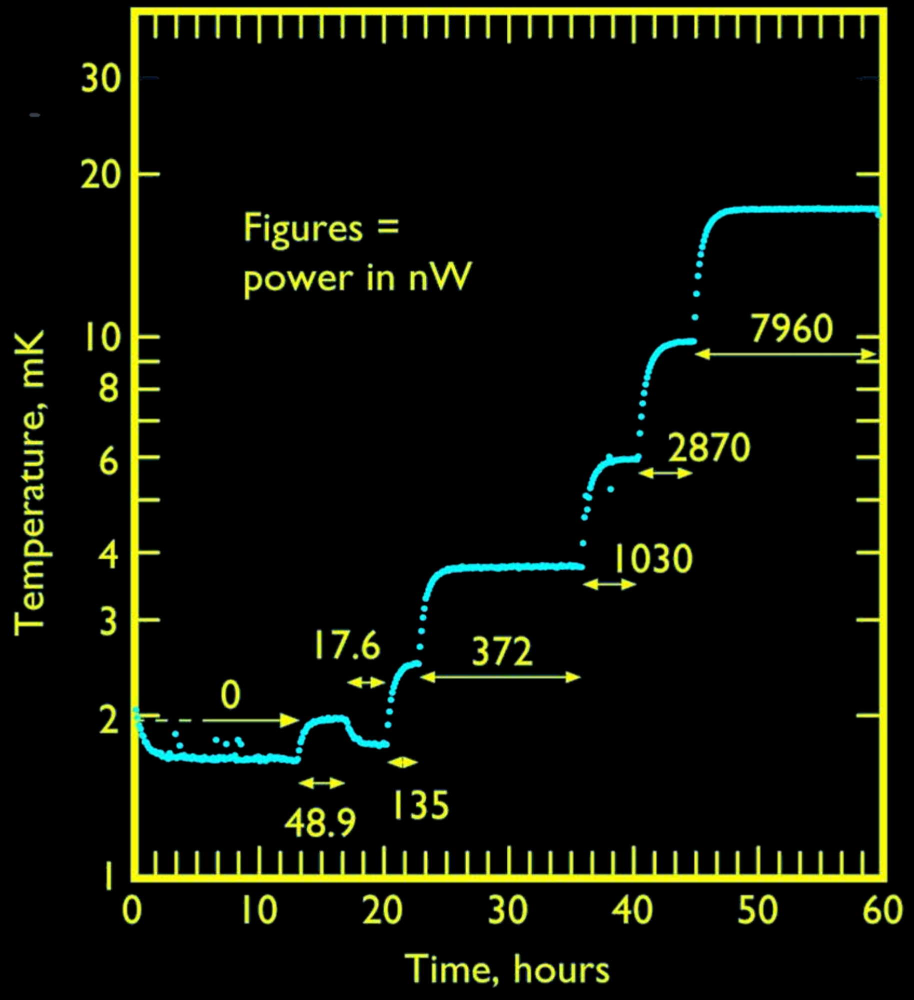
This underlines the importance of the heat exchanger quality

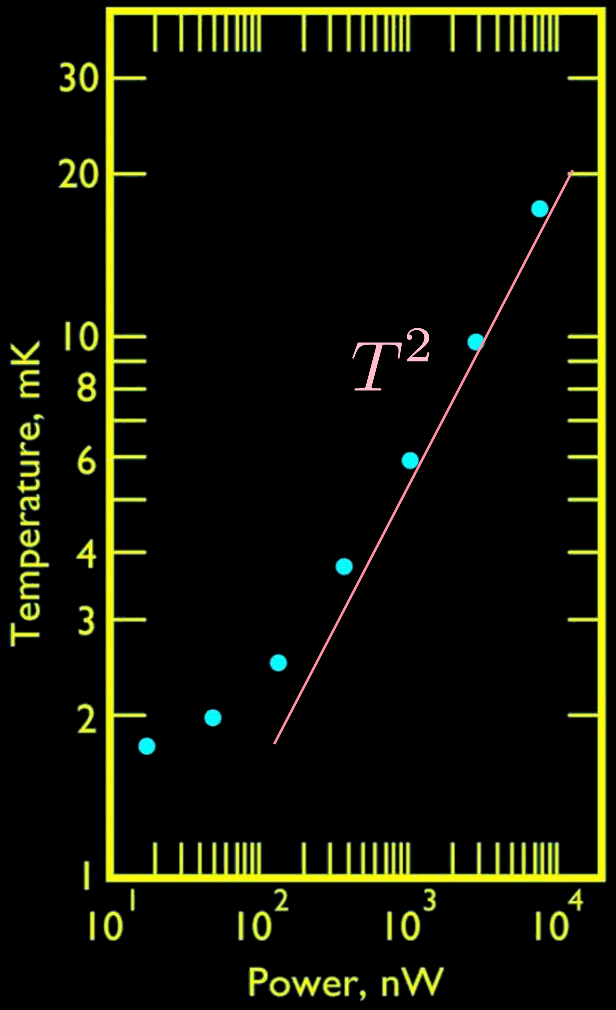
Unavoidable heat leak: viscous friction of $^3He$
Another contrain is the thermal conductivity of $^3He$

# Lancaster Dilution Refrigerator



Note tripod!

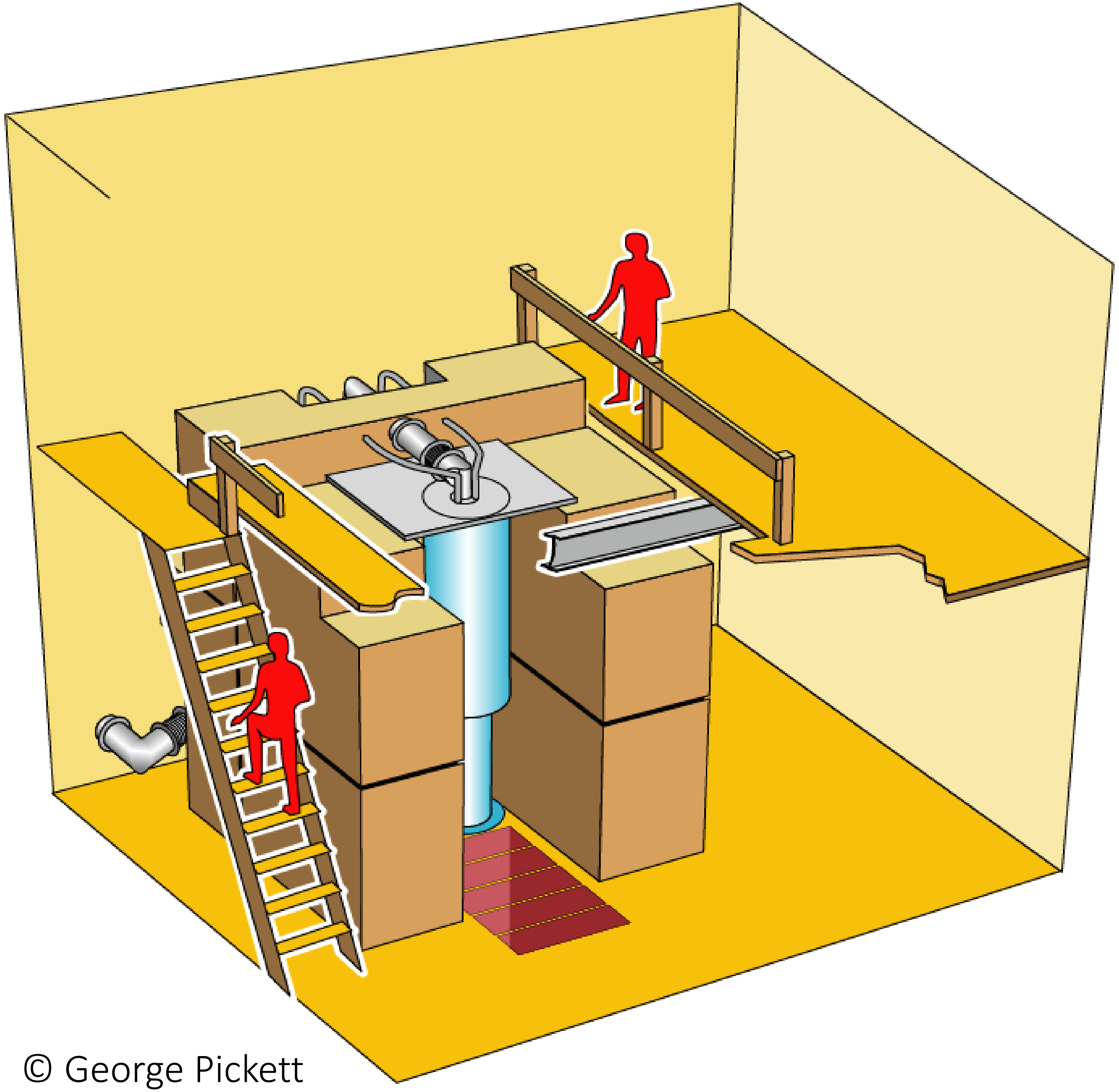


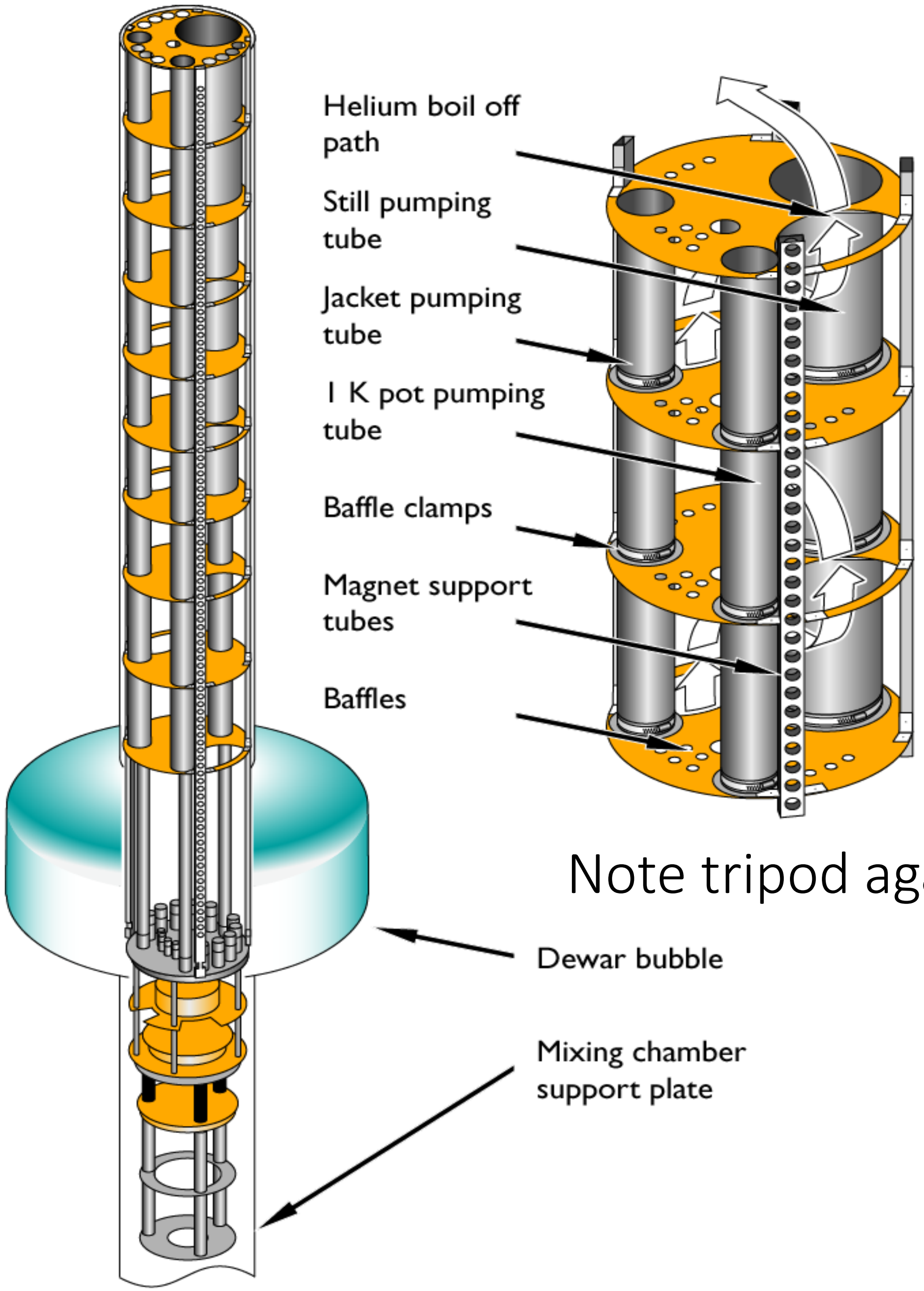




J.C. Cousins et al. , J. Low Temp. Phys. **114**, 547 (1999).

Just a few words about the external “heat leak precautions”

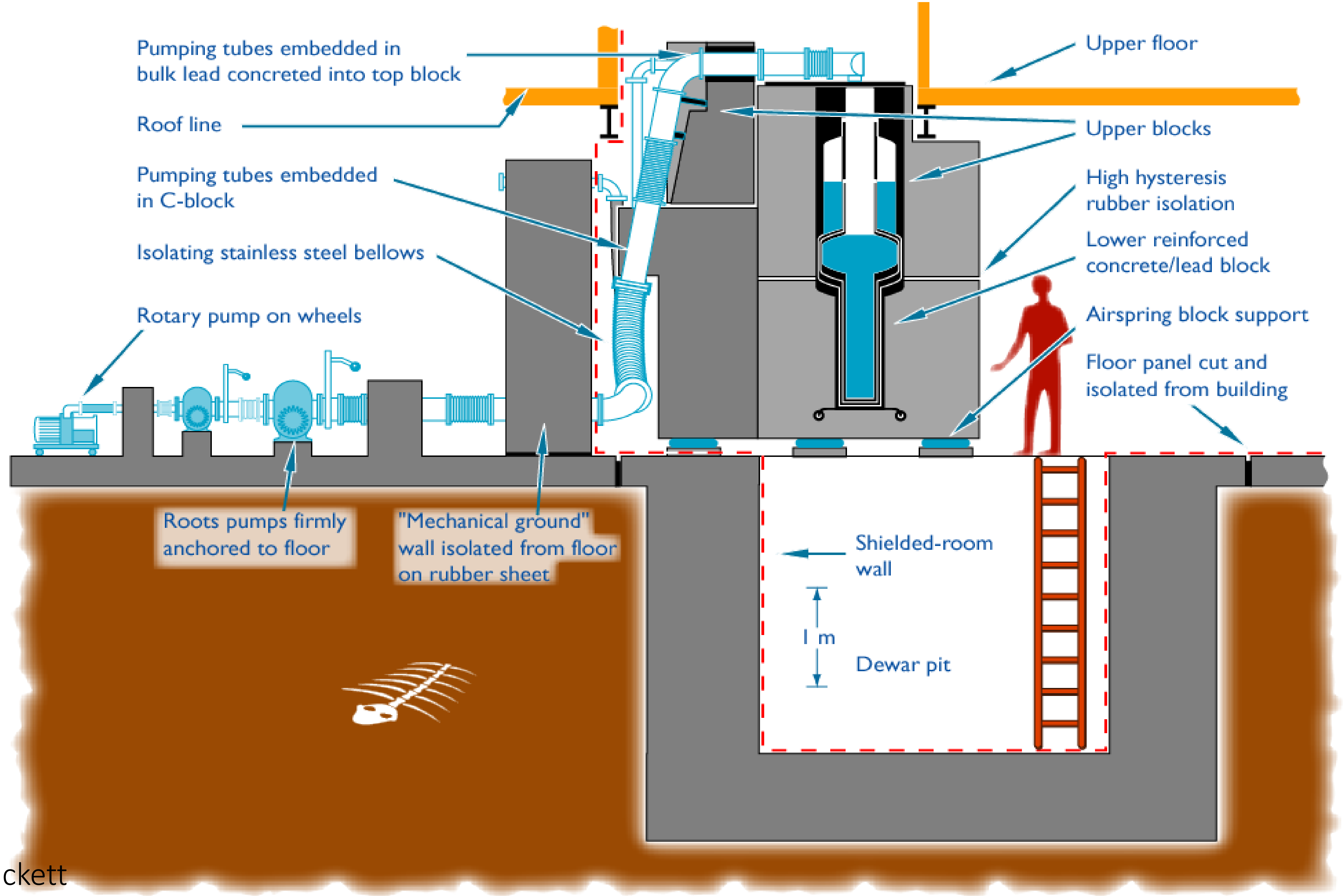
# Lancaster Advanced Cryostat





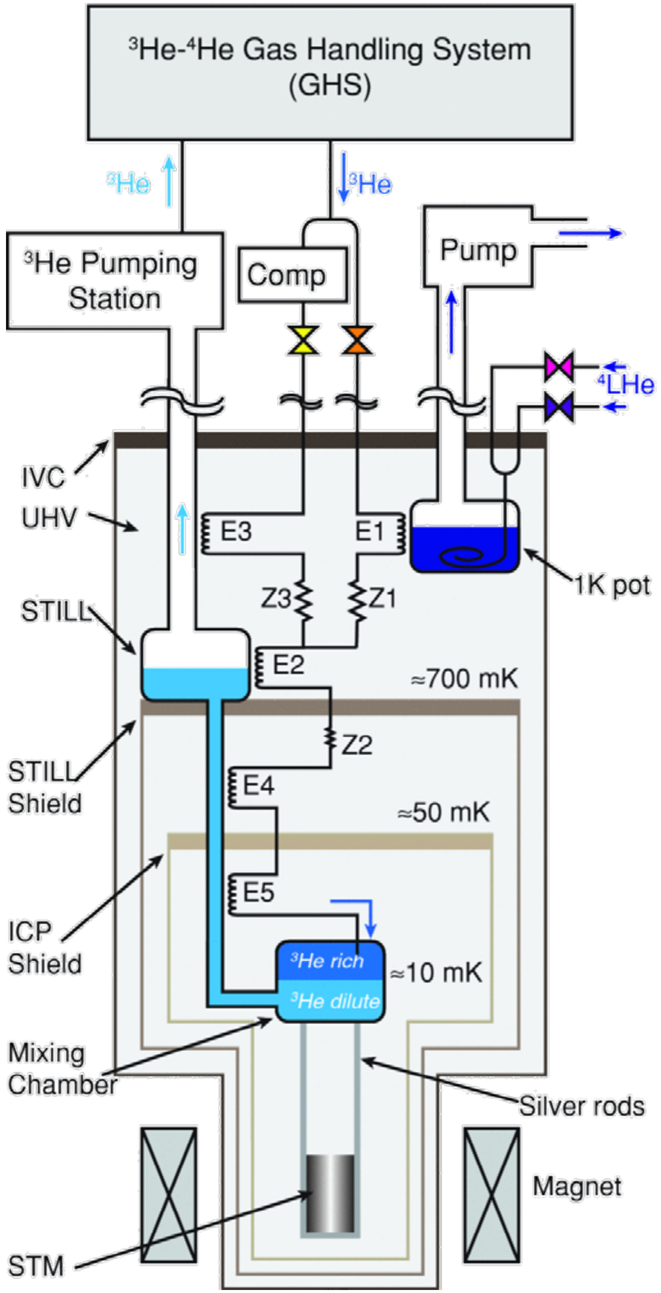

Note tripod again!

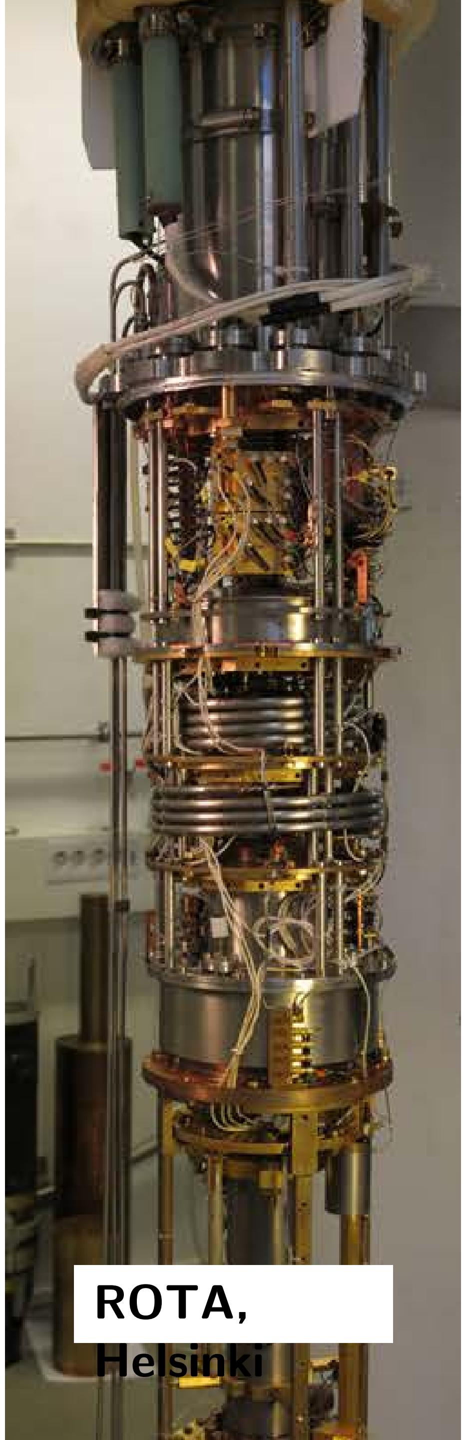
# Lancaster Advanced Cryostat



# Realisations

## Wet

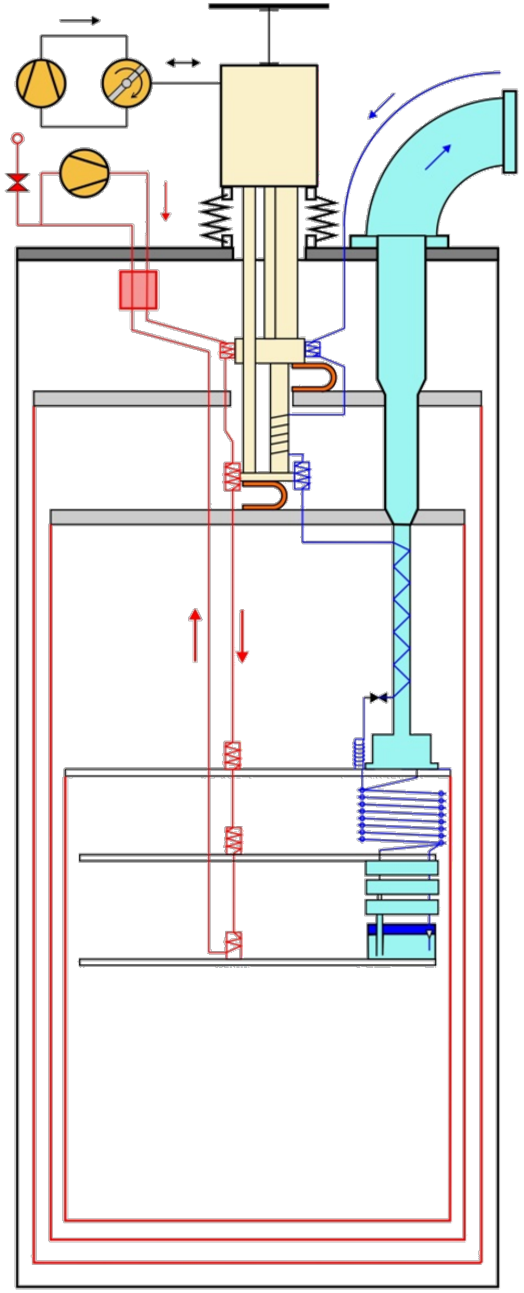


ROTA,
Helsinki

Oxford Instruments
Triton 400

## Dry



# Wet vs Dry


| Wet | Dry |
|---|---|
| refilling cycle | no refilling cycle |
| low vibrations (without 1 K Pot) | vibrations  from pulse tube |
| 4 K power 0.7 Watt x liters He evaporated | 4 K power 1-2 Watt x number of pulse tubes |
| vacuum cooled seals | no vaccum cooled seals |
| needs liquid helium handling | fast turn around times |
| higher energy efficiency | less energy efficient |
| limited amount of space/flange size | large space and flange size comes easy |
| | needs compressor |
| | needs precool system |

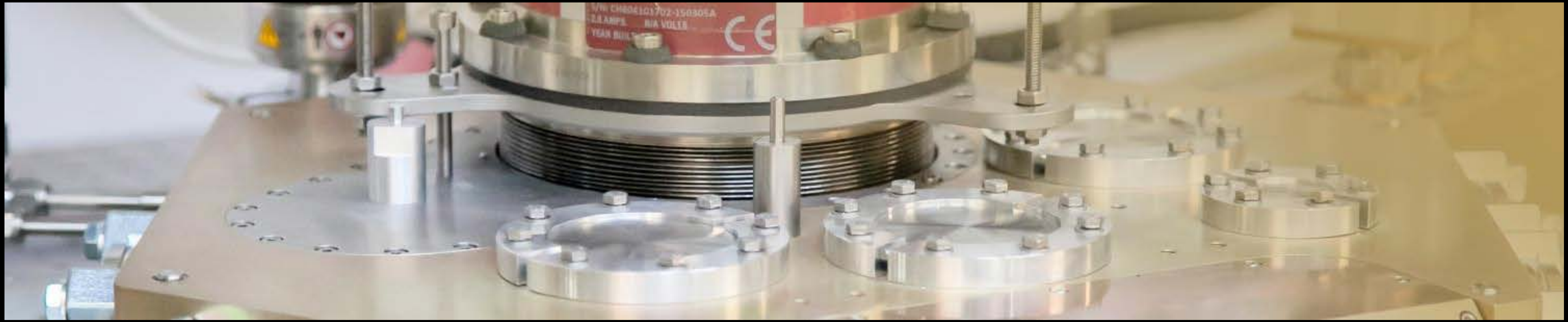

Things are strongly moving towards dry systems



http://www.cryomech.com/about-us/history/cpfront/

CryoConcept

# Ultra Low Vibrational Dry-Cryostat



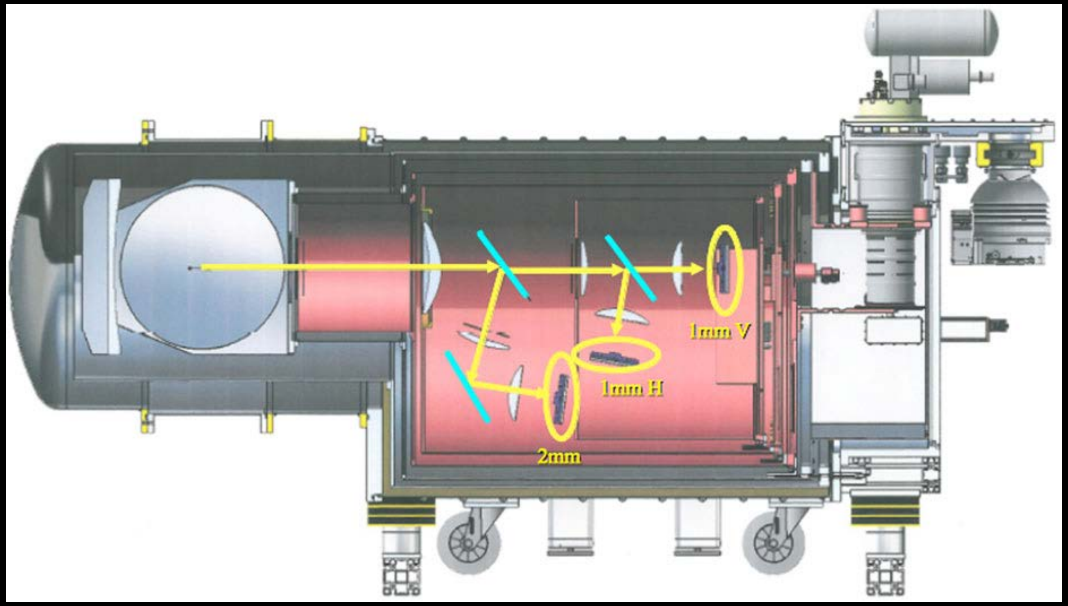
below 40 nm in the 5 Hz – 1000 Hz region

no mechanical contact of pulse tube head and dilution refrigerator

cooling by exchange gas

ULTRA QUIET TECHNOLOGY

# NIKA-2 (New Iram Kid Array) Cryostat



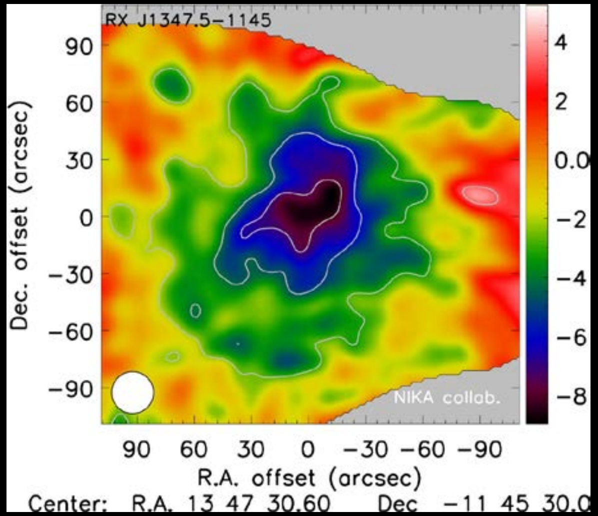

Two pulse tubes with 2 W cooling power

Dilution unit ~100mK

Compact integration for telescope

1000 2mm wavelength detectors, 2x2000 1.2 mm

Cluster: RX J1347.5-1145

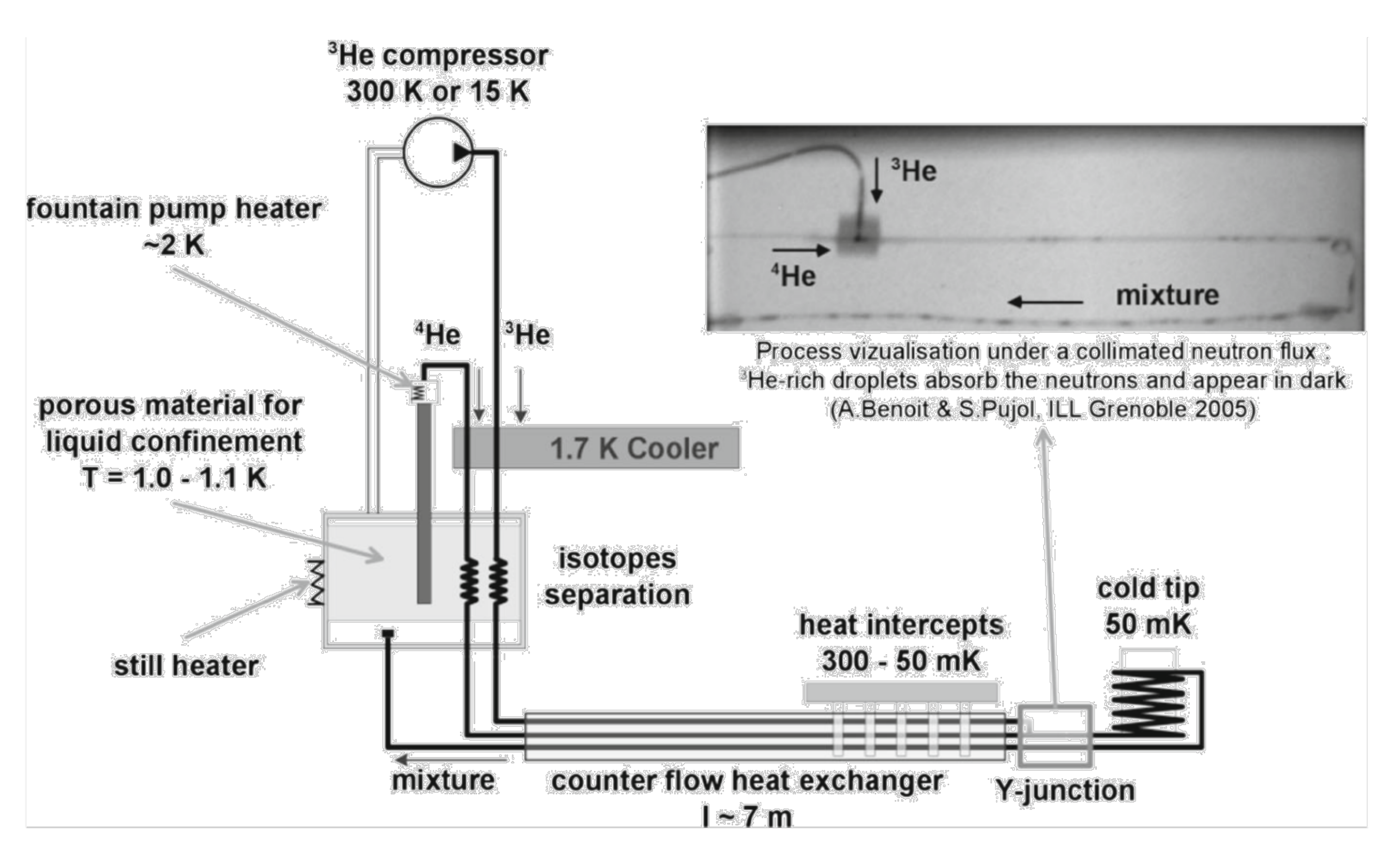




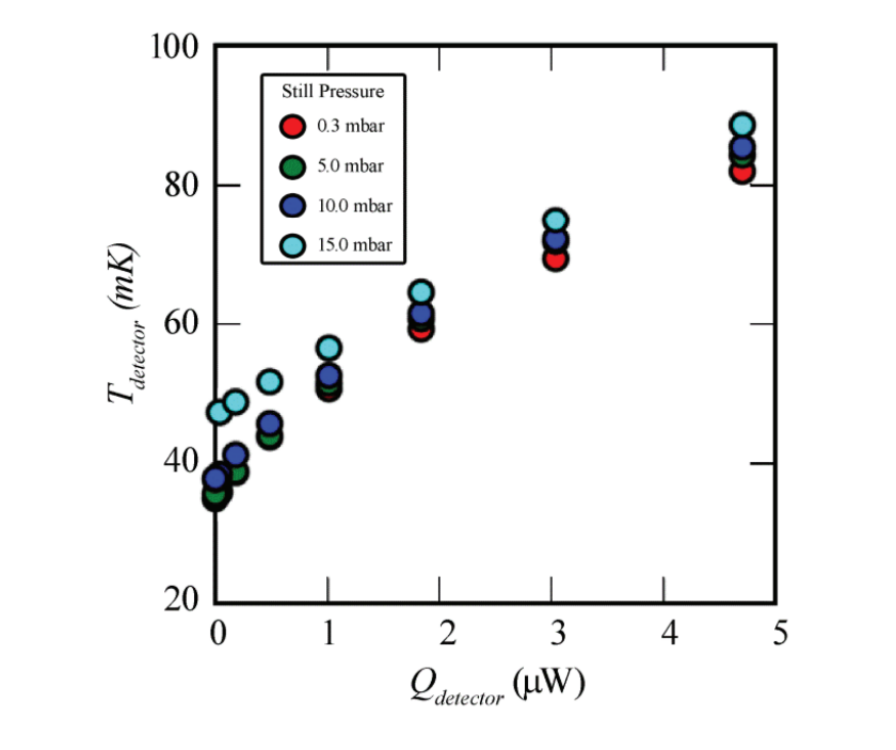
http://ipag.osug.fr/nika2/Instrument.html

A. Monfardini, et al., J Low Temp. Phys. **176**, 787 (2014)

# Closed-Cycle Dilution Refrigerator for Space

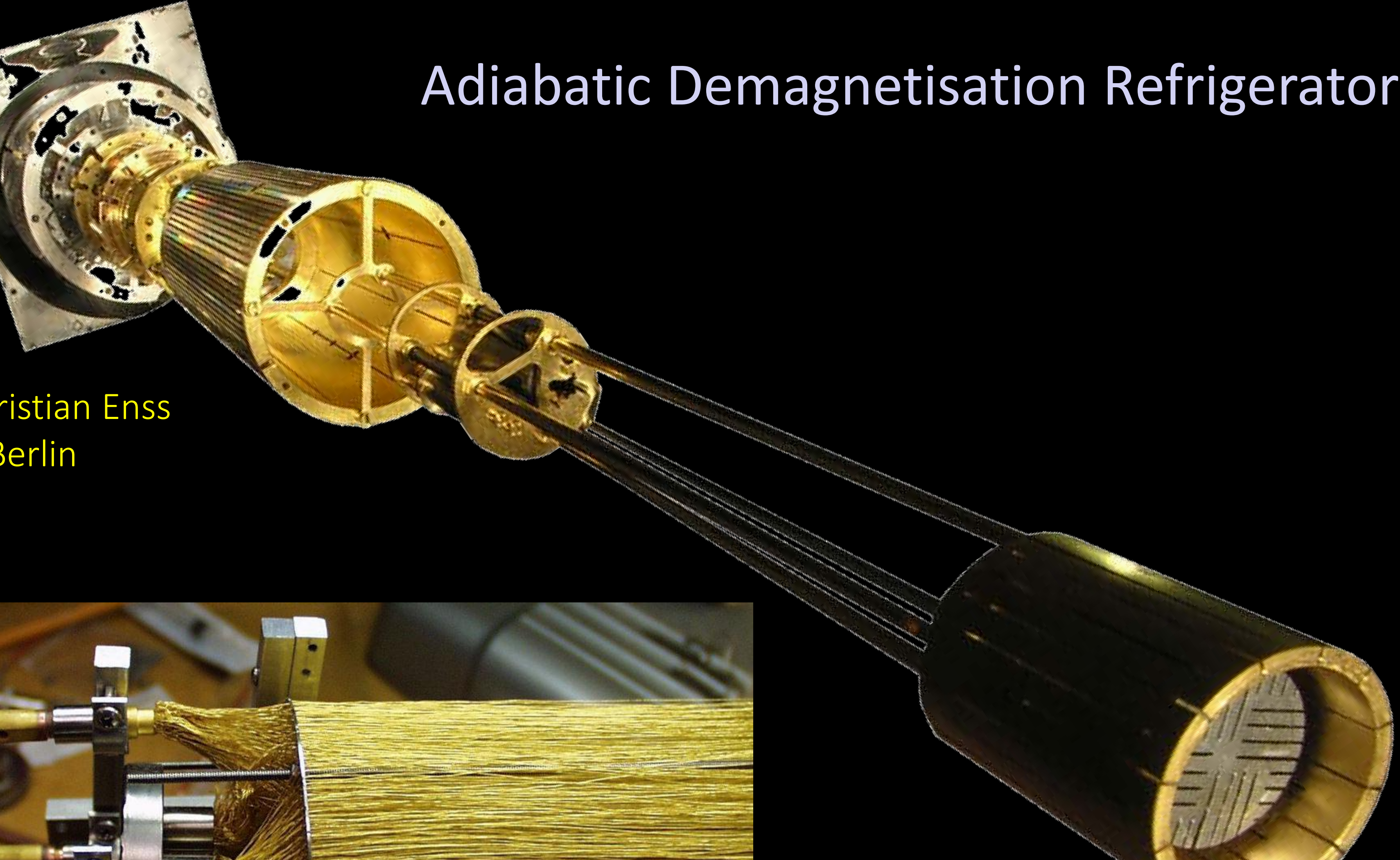



1 μW at 51 mK was demonstrated in 2012




P. Camus, *et al.*, J Low Temp Phys. **176**, 1069 (2014) https://cryocoolerorg.wildapricot.org/resources/Documents/C18/060.pdf

# Adiabatic Demagnetisation Refrigerators

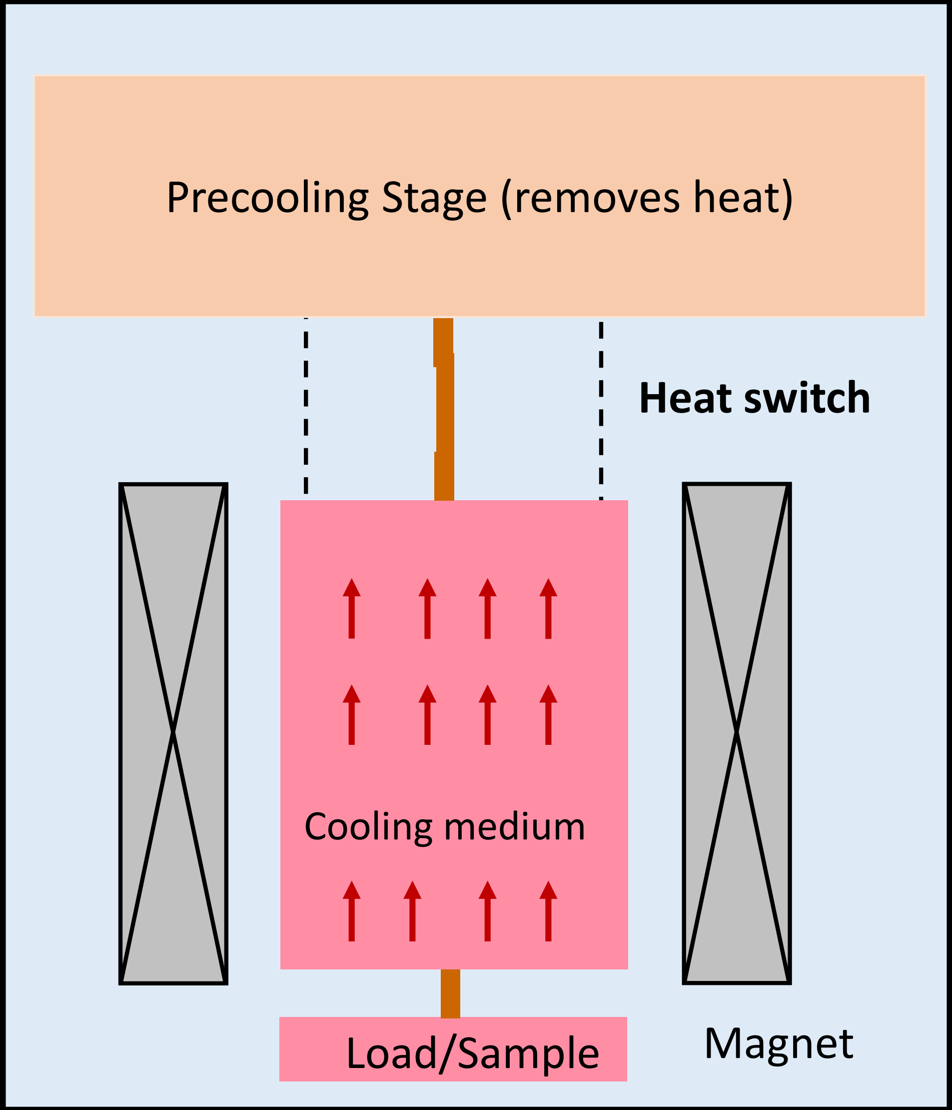


© Christian Enss
CBT, Berlin

© Dan McCammon, Wisconsin University

© George Pickett

# Adiabatic Demagnetisation: principle of operation




## 1. Precooling

Isothermal Magnetisation

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

2. Thermal Isolation

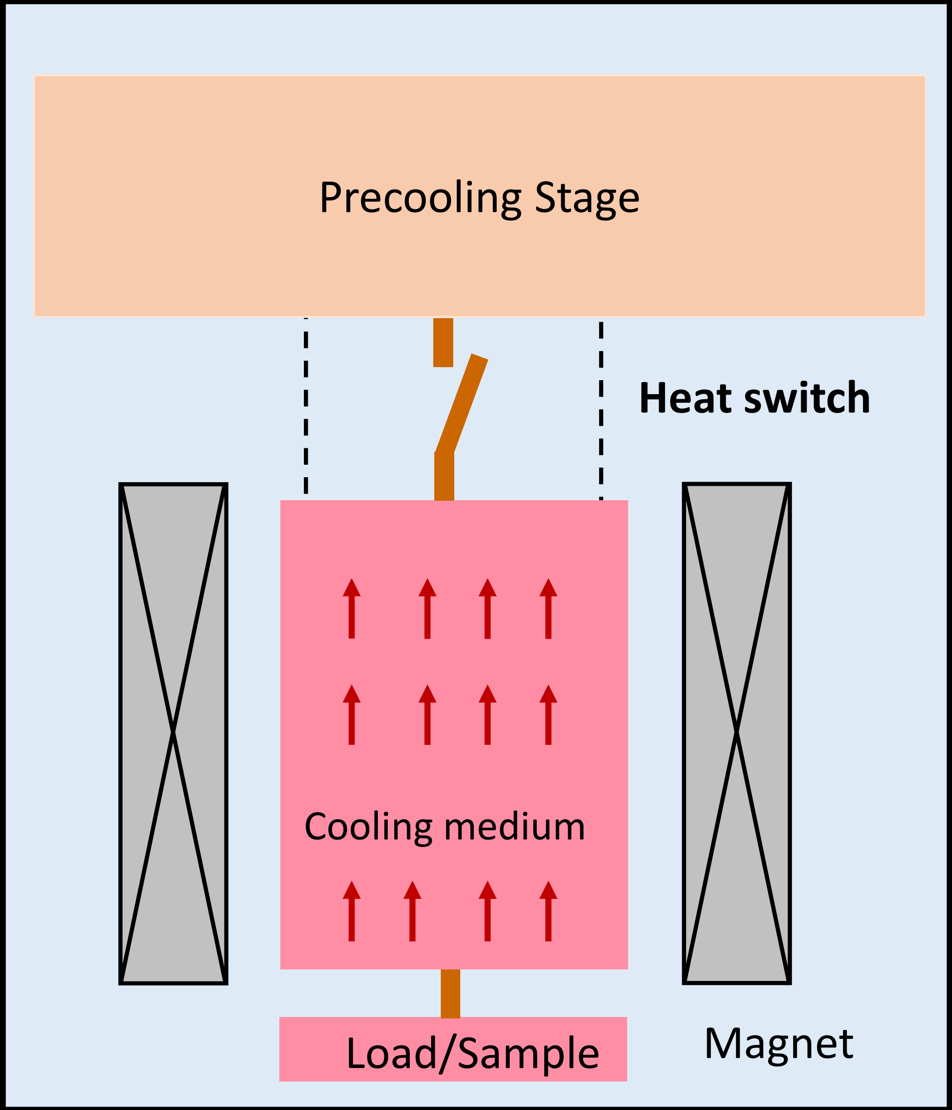
Heat switch closed

3. Adiabatic Demagnetisation

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

4. Warming up

# Adiabatic Demagnetisation: principle of operation




1. Precooling

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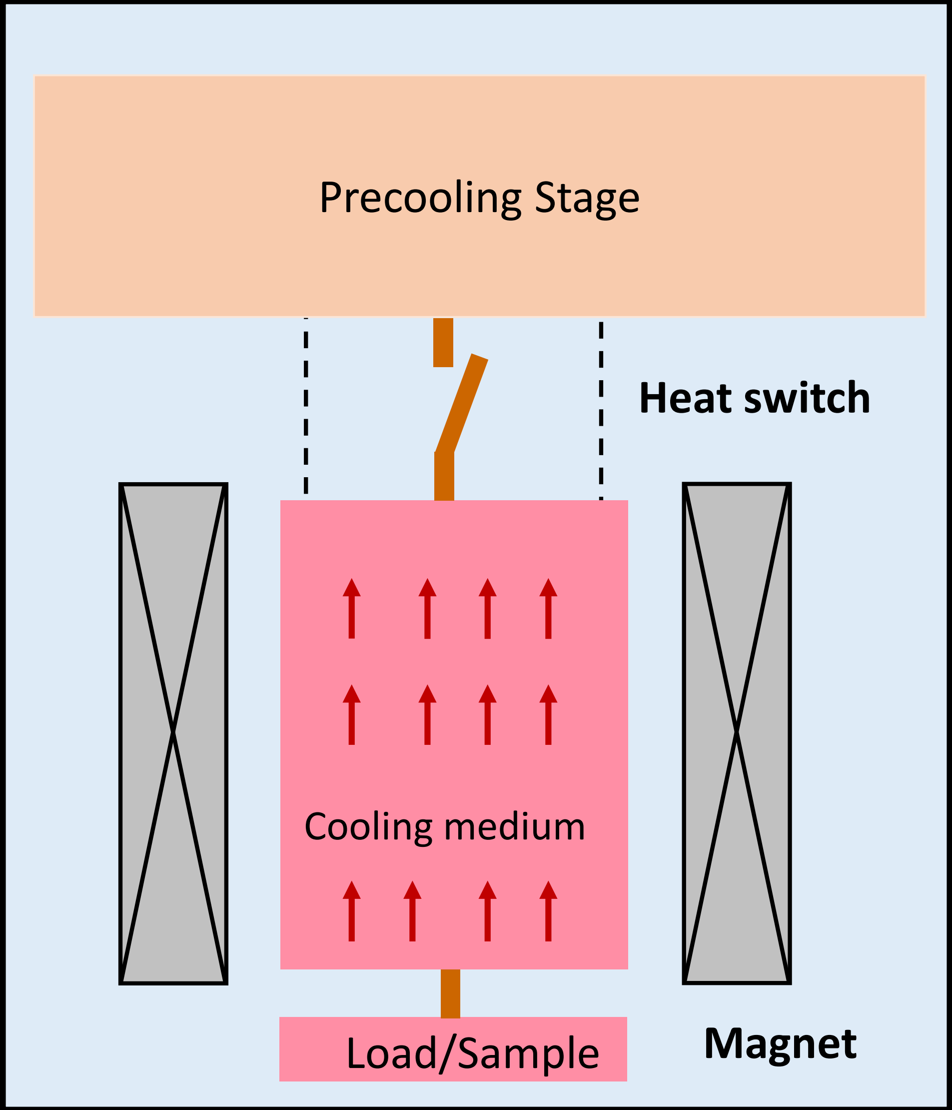


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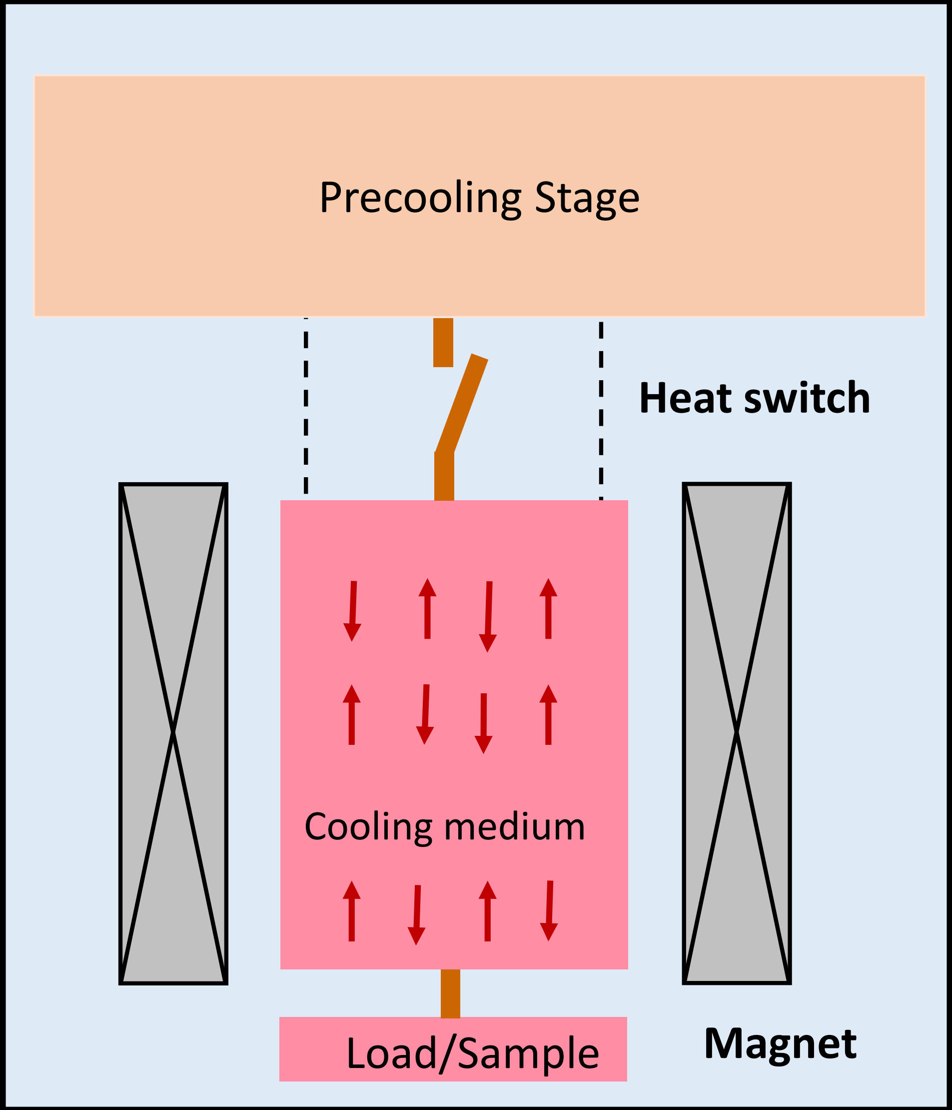
# Adiabatic Demagnetisation: principle of operation




1. Precooling

Isothermal Magnetisation

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

2. Thermal Isolation

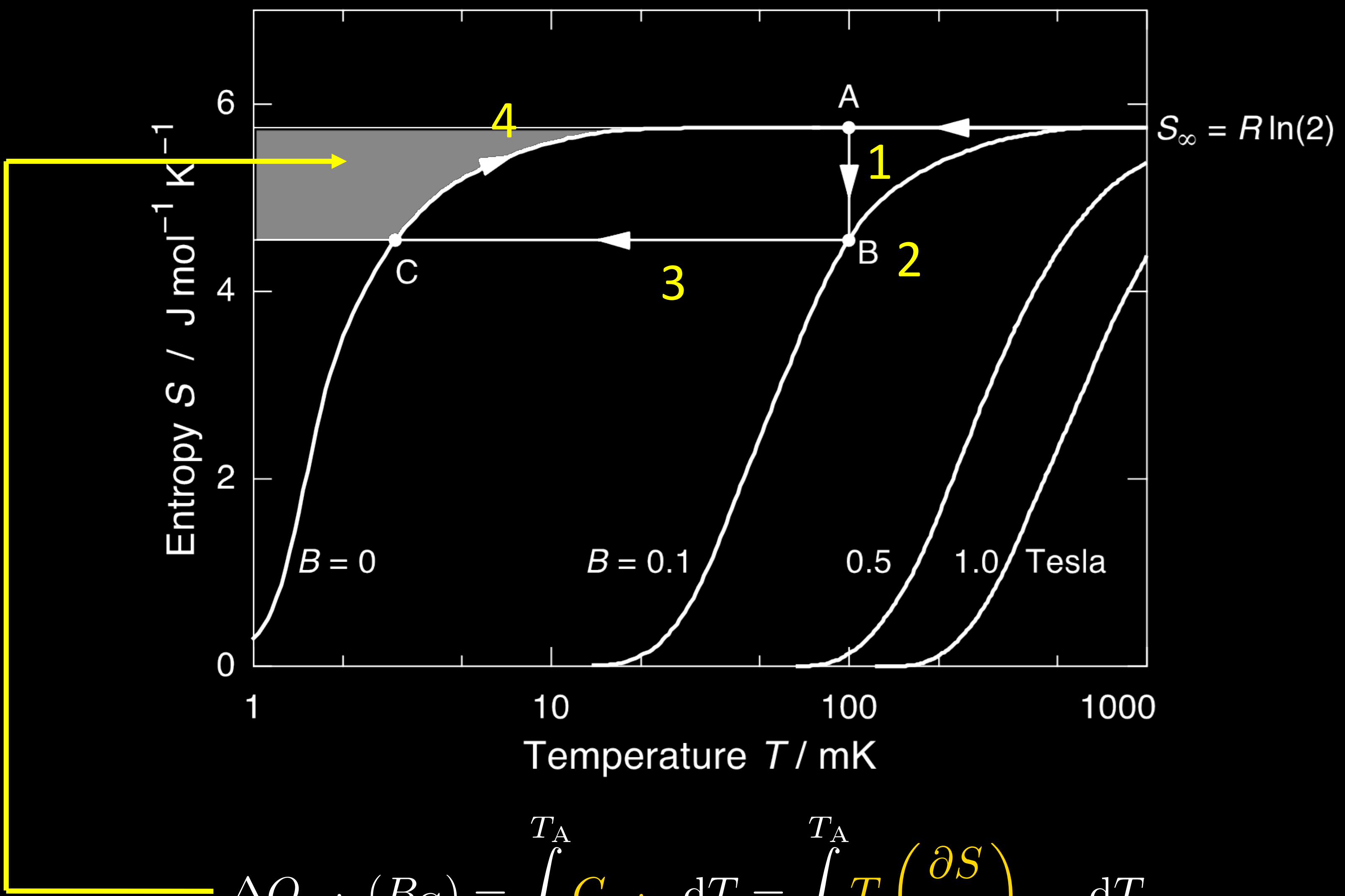
Heat switch opened

3. Adiabatic Demagnetisation (reduce B and hence T)

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

4. Warming up

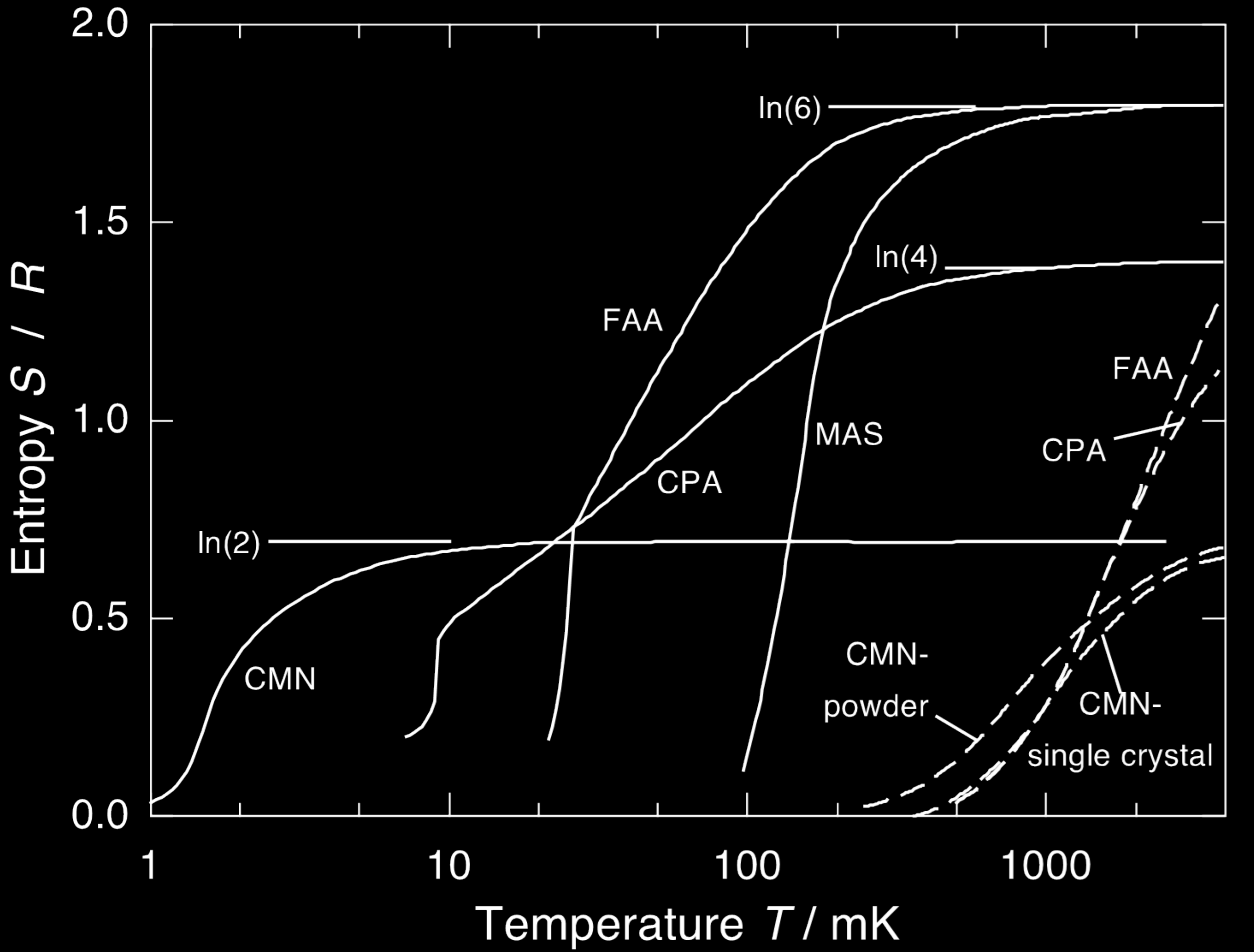
# Entropy and Cooling Capacity




For a system with 2 states (up and down)

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

# Entropy of Paramagnetic Salts




FAA: Ferric ammonium alum
MAS: Manganous ammonium sulphate
CPA: Chrome potassium alum
CMN: Cerium magnesium nitrate

Christian Enns, S. Hunklinger, Low Temperature Physics, (Springer 2005)

# Problem: Thermal Conductivity of Paramagnetic Salts



Salt pill for space application

15.000 gold wires

Salt pill grown around the wires

Possible solution: Super-heavy electron material as metallic refrigerant for adiabatic demagnetization cooling.

Science Advances 09 Sep 2016: Vol. 2, no. 9, e1600835
DOI: 10.1126/sciadv.1600835

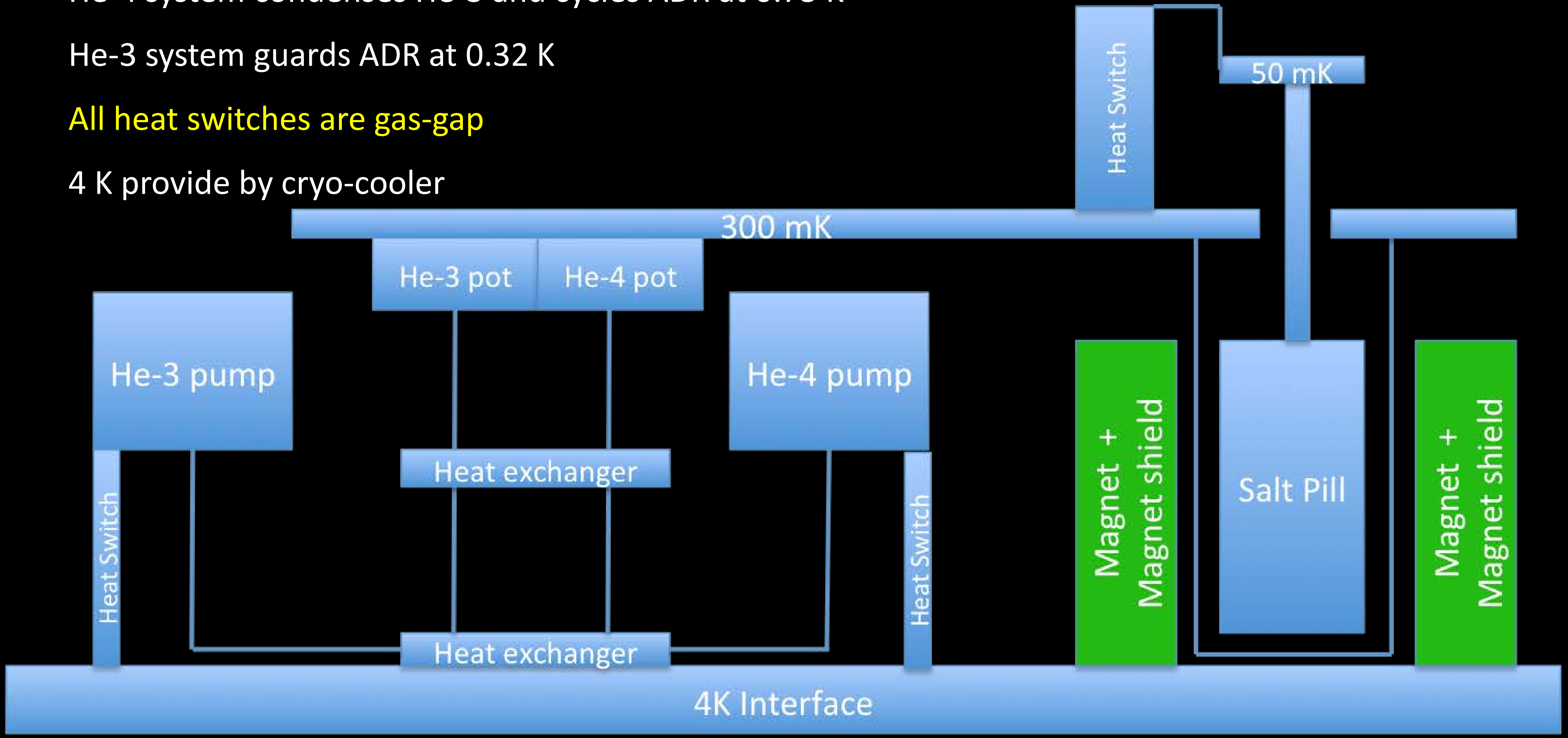
# Fully Automatic ADR for Space Missions

He-4 system condenses He-3 and cycles ADR at 0.75 K

He-3 system guards ADR at 0.32 K

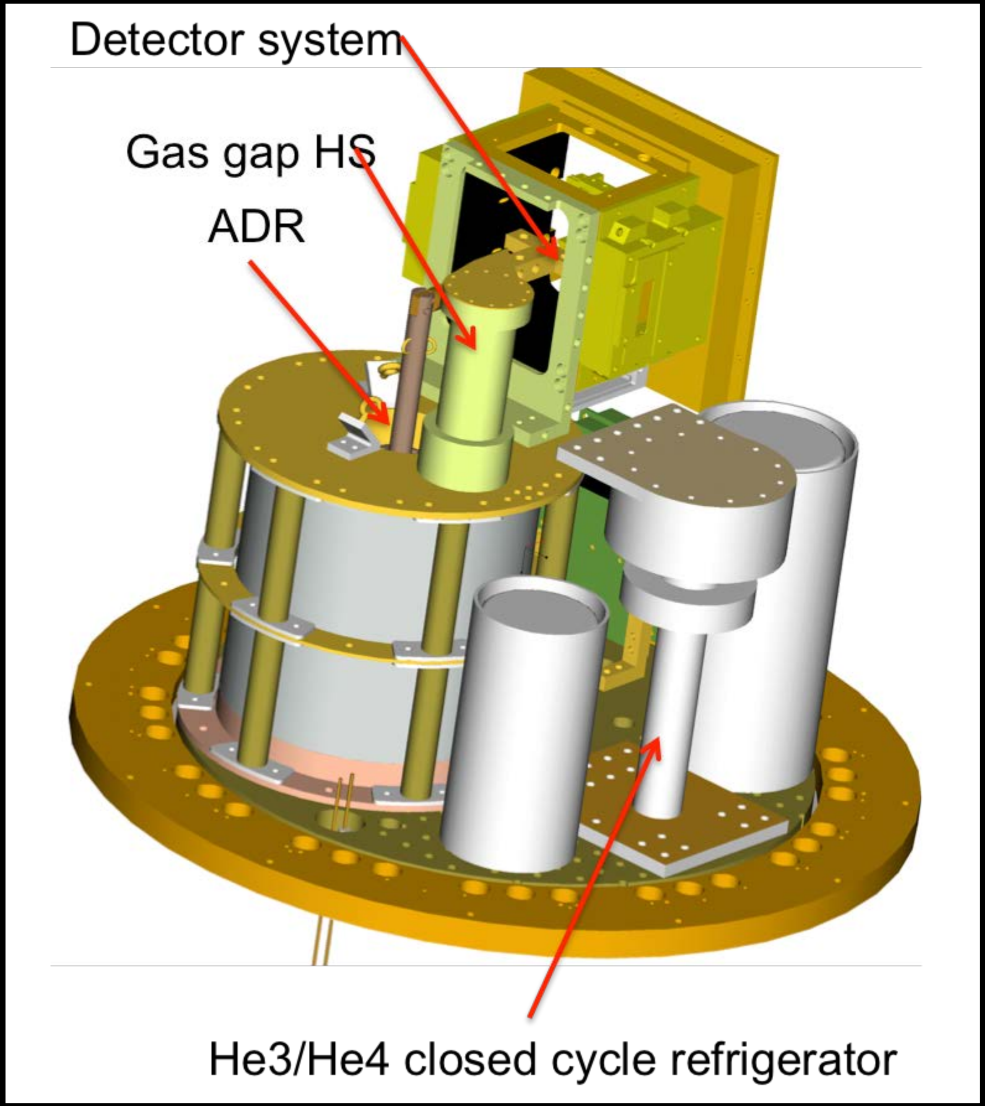
All heat switches are gas-gap

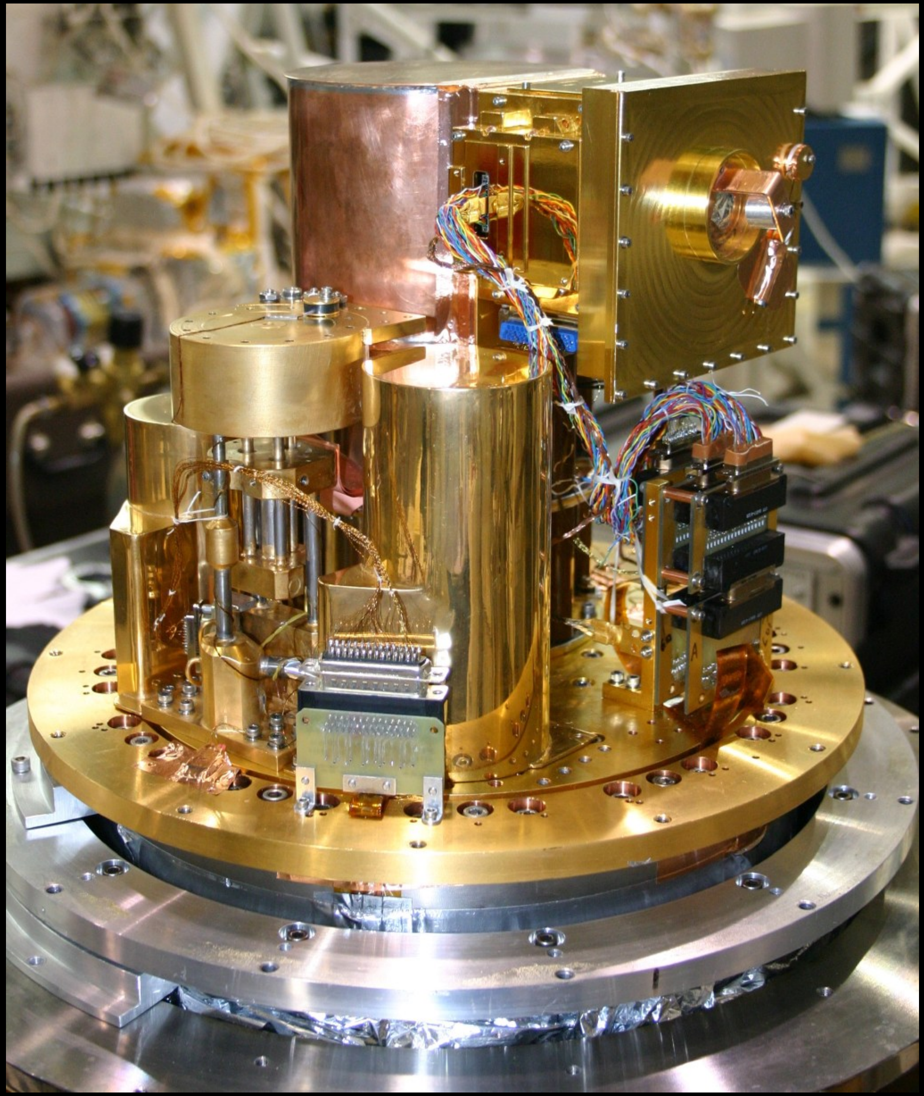
4 K provide by cryo-cooler




NASA GSFC

# Fully Automatic ADR for Space Missions (50 mK)



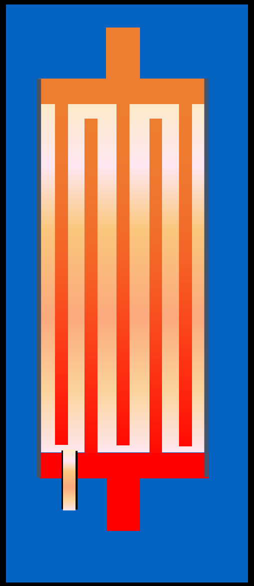



For latest developments check NASA Technical Reports Server https://ntrs.nasa.gov/search.jsp

NASA GSFC

# Heat Switches

## Gas gap heat switches



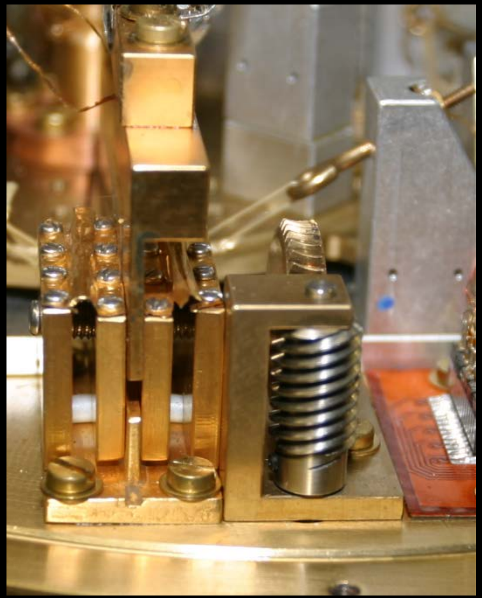
exchange gas → pumping

$^4He$ superfluid layer
→ creep

$H_2$ ortho-para conversion

$^3He$ no exothermic reation
no creep
high vapour pressure

## Mechanical heat switch

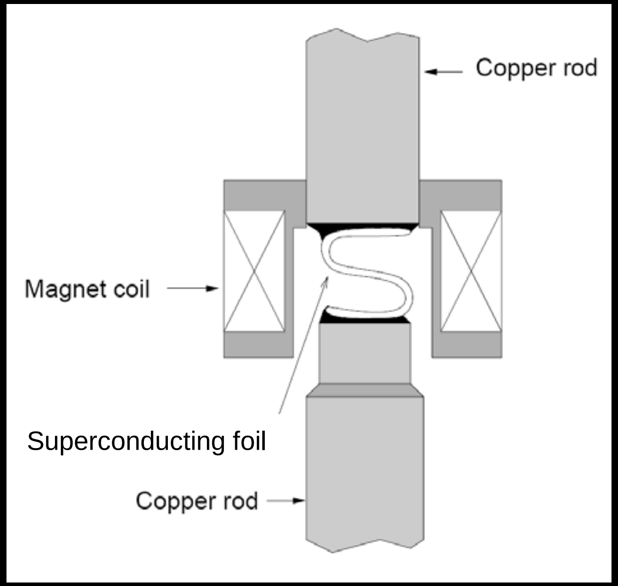


large force needed
typically 100 N

closed few mW/K
… 1 W/K @ 15K

heating on opening

## Superconducting heat switch



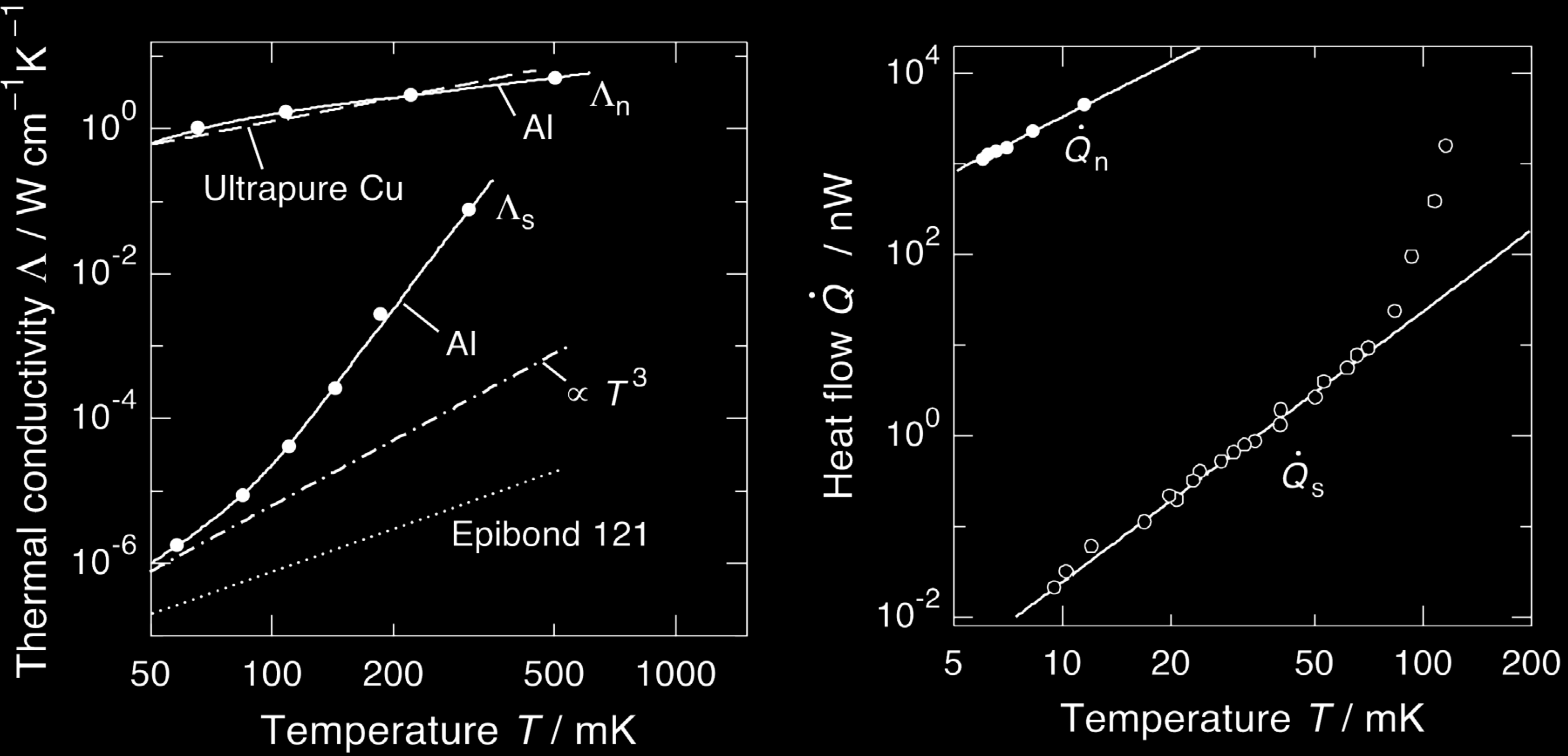

only well below $T_c$

open means low conductivity

eddy currents

flux trapping

# Superconducting Heat Switch



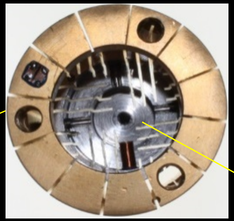

Christian Enns, S. Hunklinger, Low Temperature Physics, (Springer 2005)

# PTB Nuclear Demagnetisation Fridge

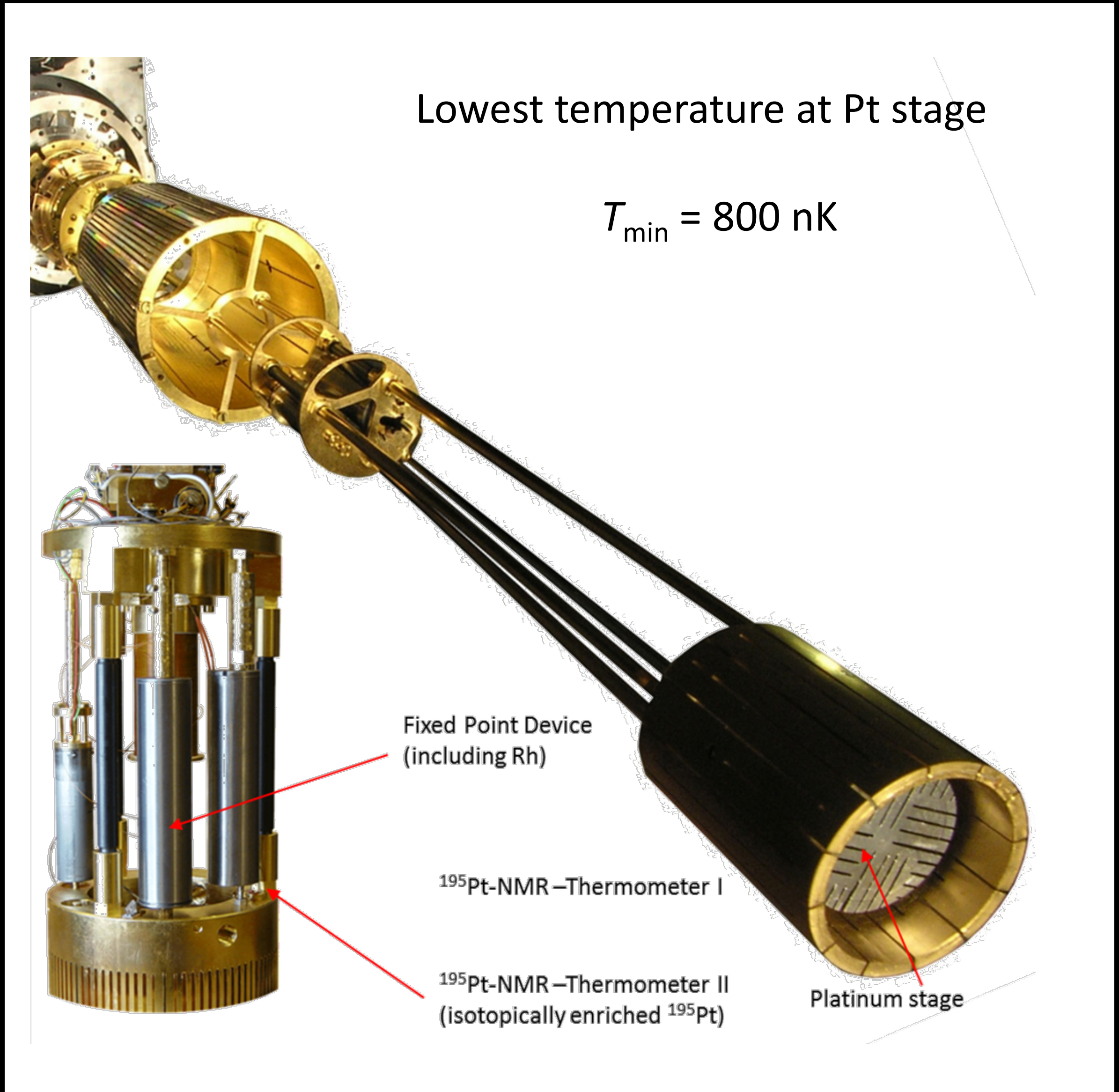
heat switch

Cu stage

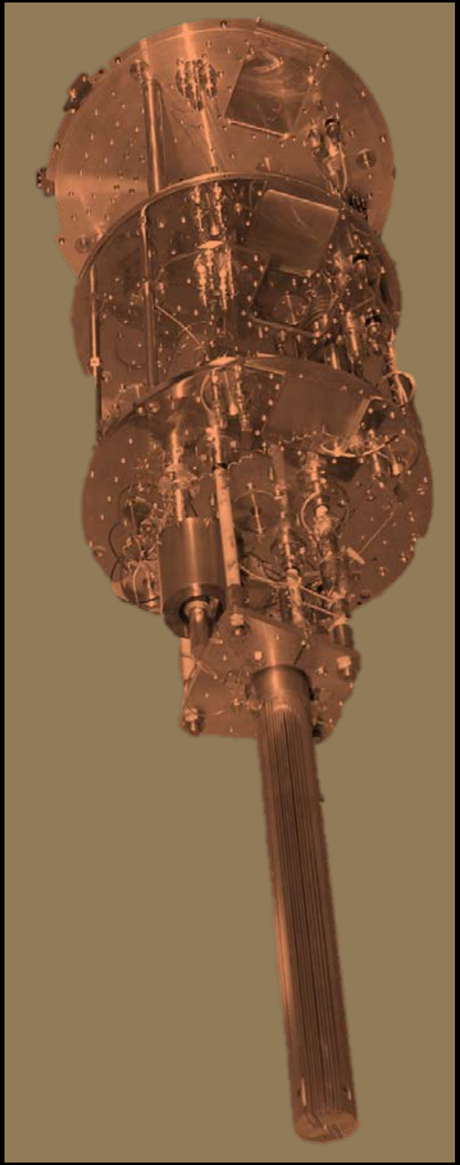
heat switch



Pt stage



# Cryogenic-Free Demagnetisation Fridge



Bluefors LD400 dry dilution fridge

9 T magnet

Superconducting Al heat switch

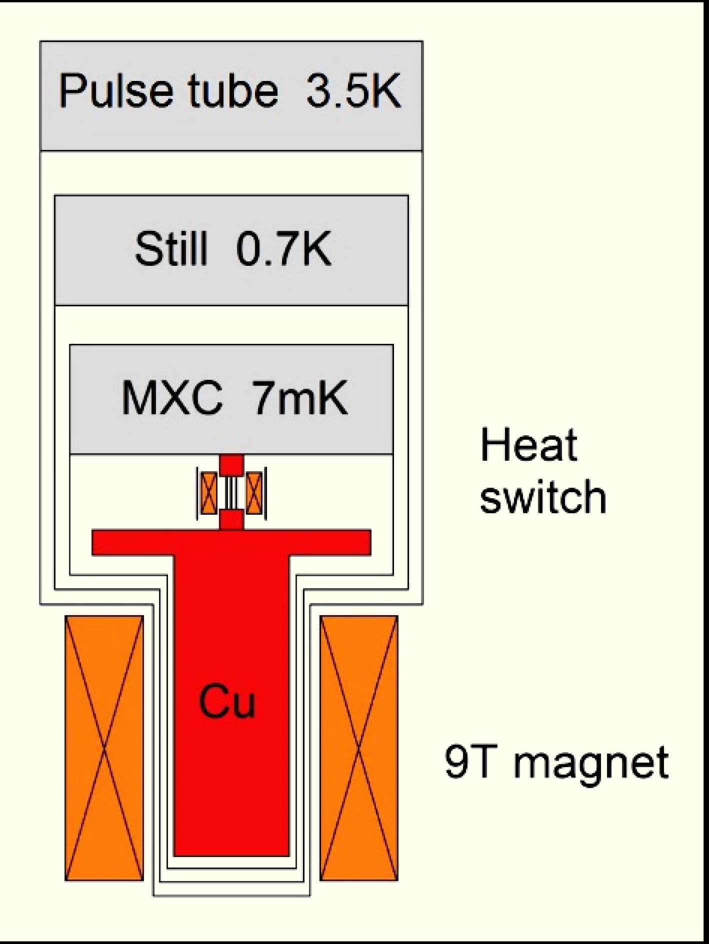
Copper nuclear stage

**Problem:**

Vibrations ⟶ eddy current heating

$\dot{Q}_{\mathrm{eddy}} \propto \dot{B}^2$

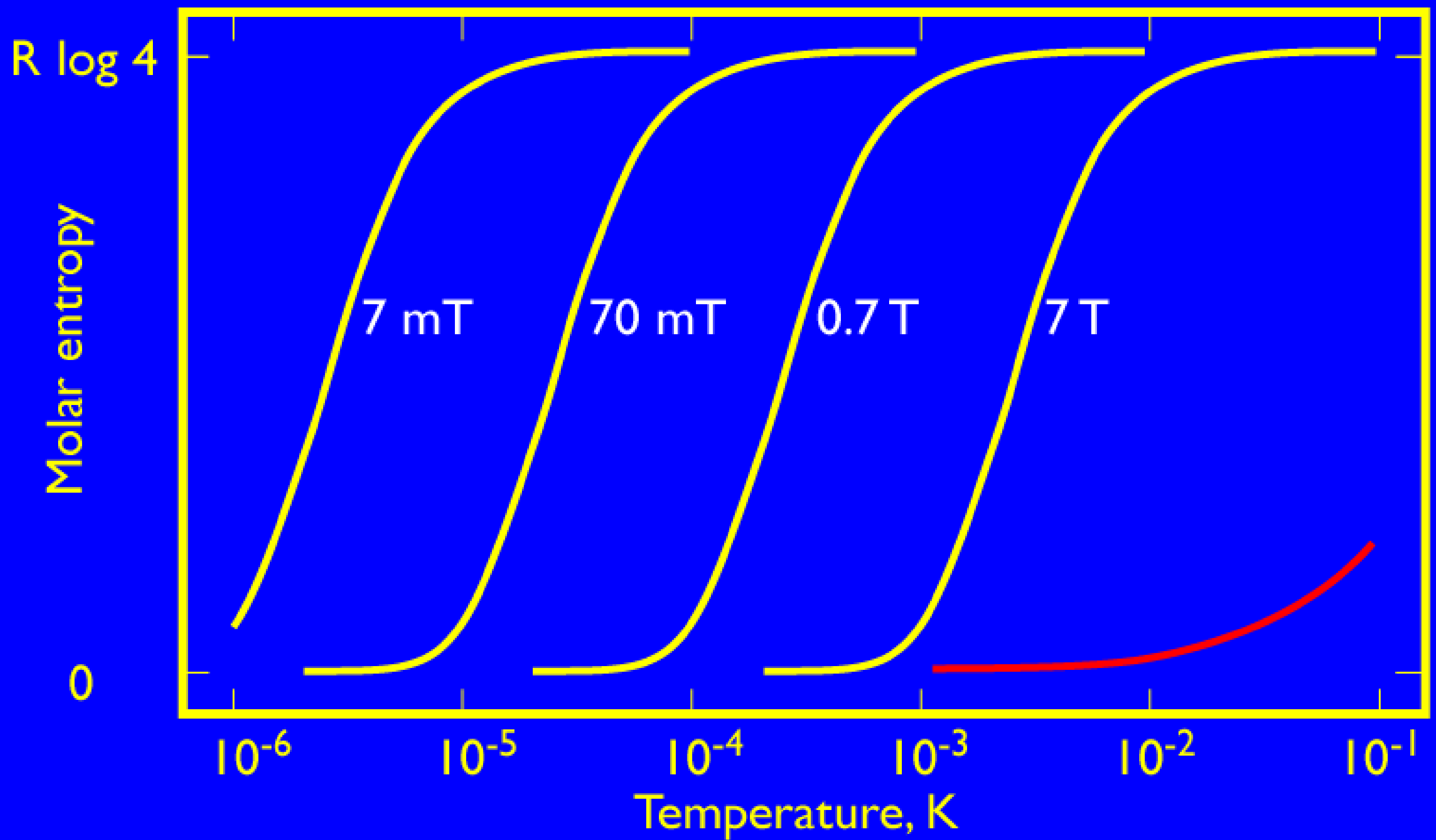
$T_{\mathrm{min}} \sim 0.16$ mK




BlueFors CRYOGENICS

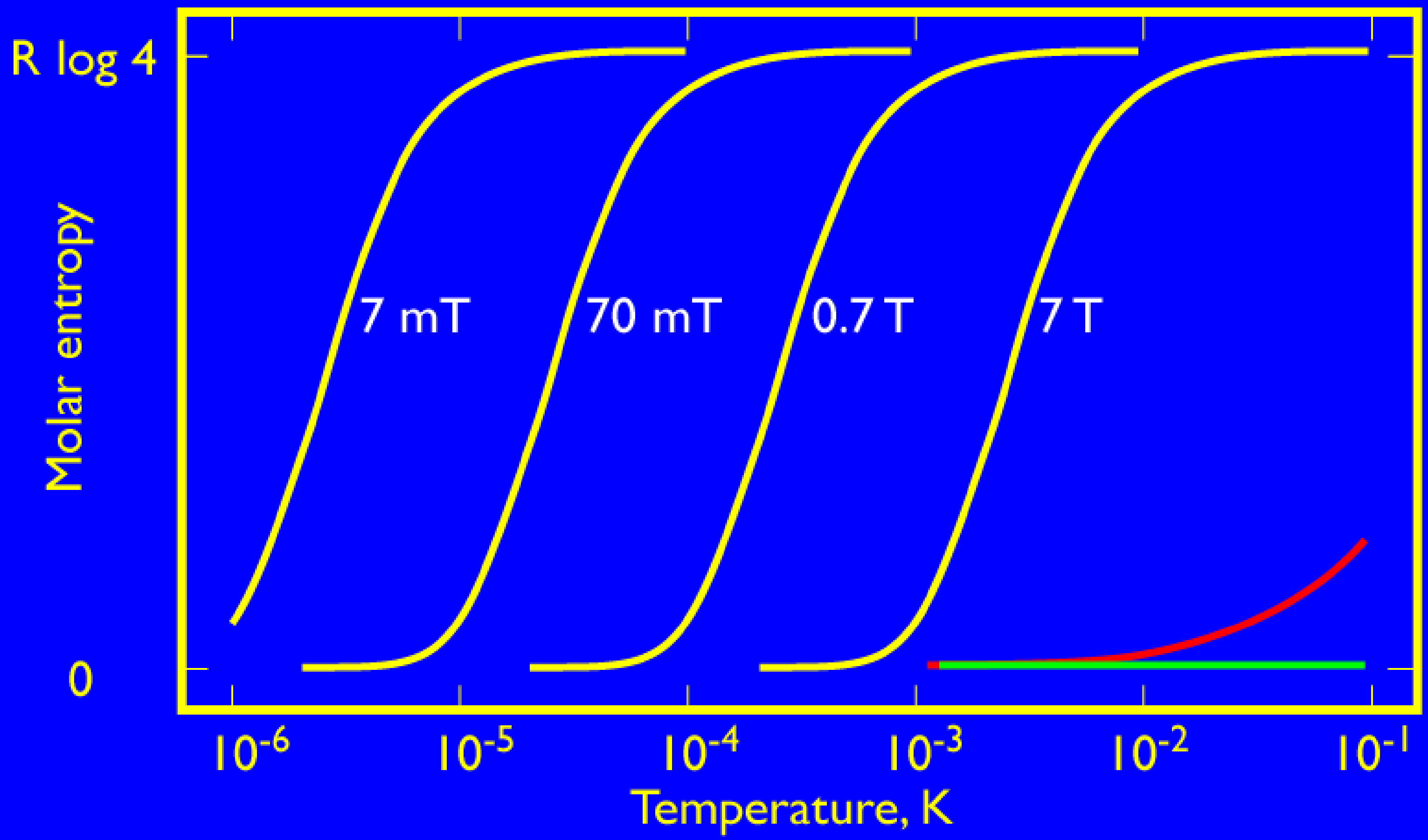
I. Todoshchenko, J.-P. Kaikkonen, R. Blaauwgeers, P. J. Hakonen, and A. Savin, Rev. Sci. Instrum. **85**, 085106 (2014).

# Lancaster Nuclear Demagnetization Cryostat




Molar entropy of liquid $^3He$ ~2 orders lower at 10 mK.

# Lancaster Nuclear Demagnetization Cryostat

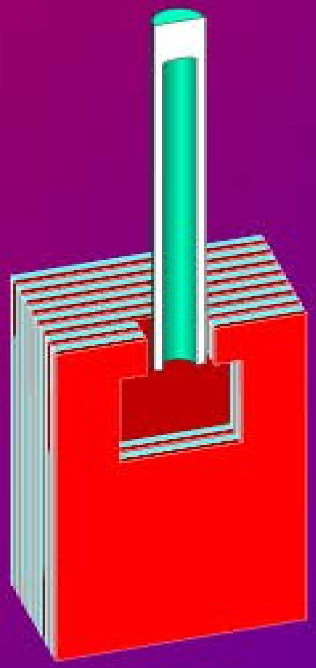



Molar entropy of Cu electron gas 8 orders lower at 10 mK.

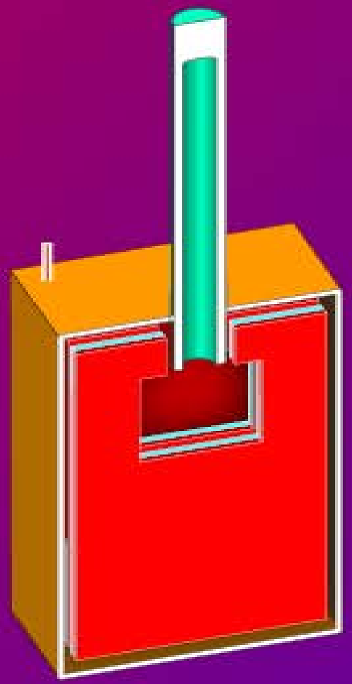
Since we only need a small volume of copper, let’s get it as close to the specimen as possible, that is immerse it in the liquid.

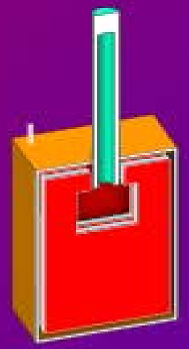
Since we only need a small volume of copper, let’s get it as close to the specimen as possible, that is immerse it in the liquid.

So here is our inner stage, a stack of Cu plates, immersed in the liquid, each with a layer of sintered silver on the surface for thermal contact.

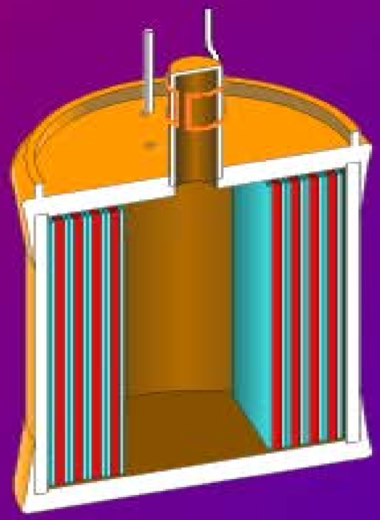


We wrap this in a thin-walled paper/epoxy box, and add a silver sinter pad to make contact for precooling and a filling tube.





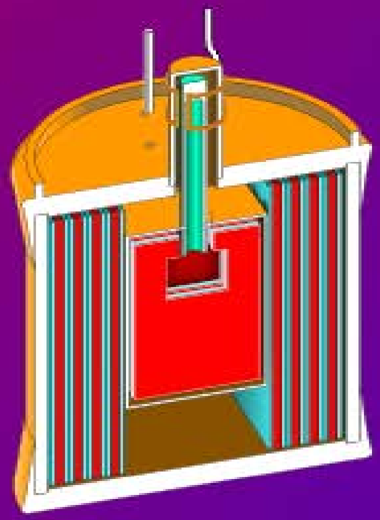
To cut down the heat leak we add a second stage, also furnished with a precooling link, and filling tube.



To cut down the heat leak we add a second stage, also furnished with a precooling link, and filling tube.

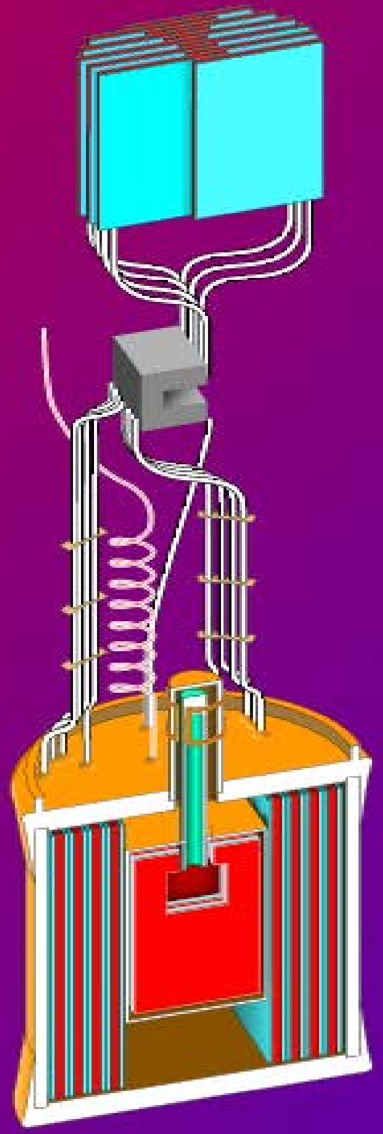
And we put the inner cell inside.

This allows the inner cell to have a very thin wall (und thus low slow-release heat leak) because the pressure is supported by the outer cell wall.

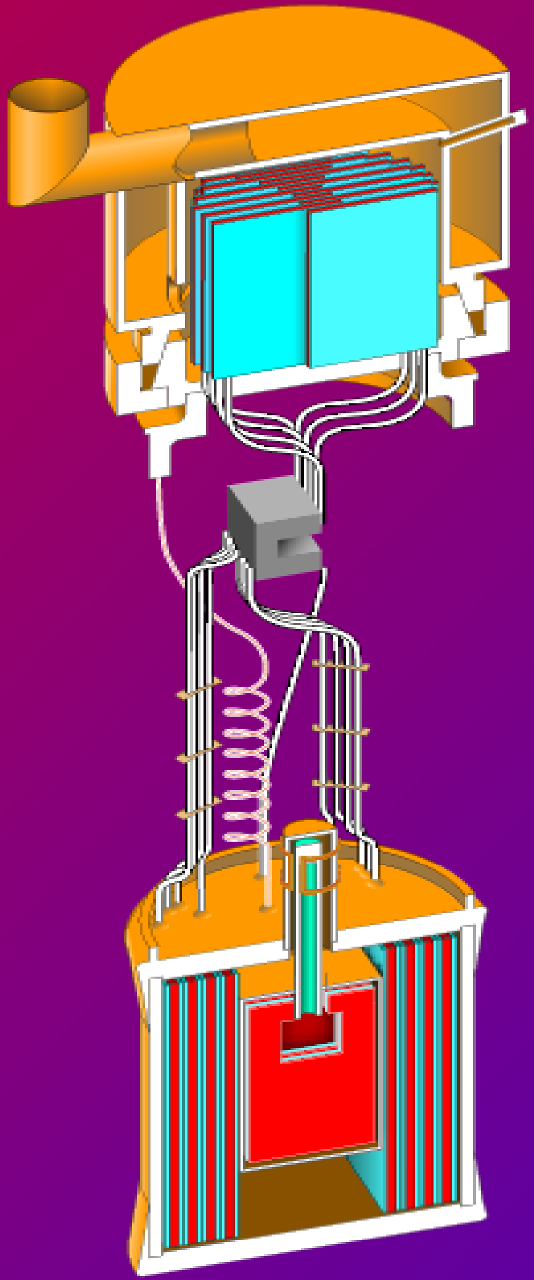


The thermal contact to the mixing chamber is also made by silver sinter plates and connected to the specimen via high purity Ag wires (RRR~$10^3$):

Plus a single crystal Al heat switch.

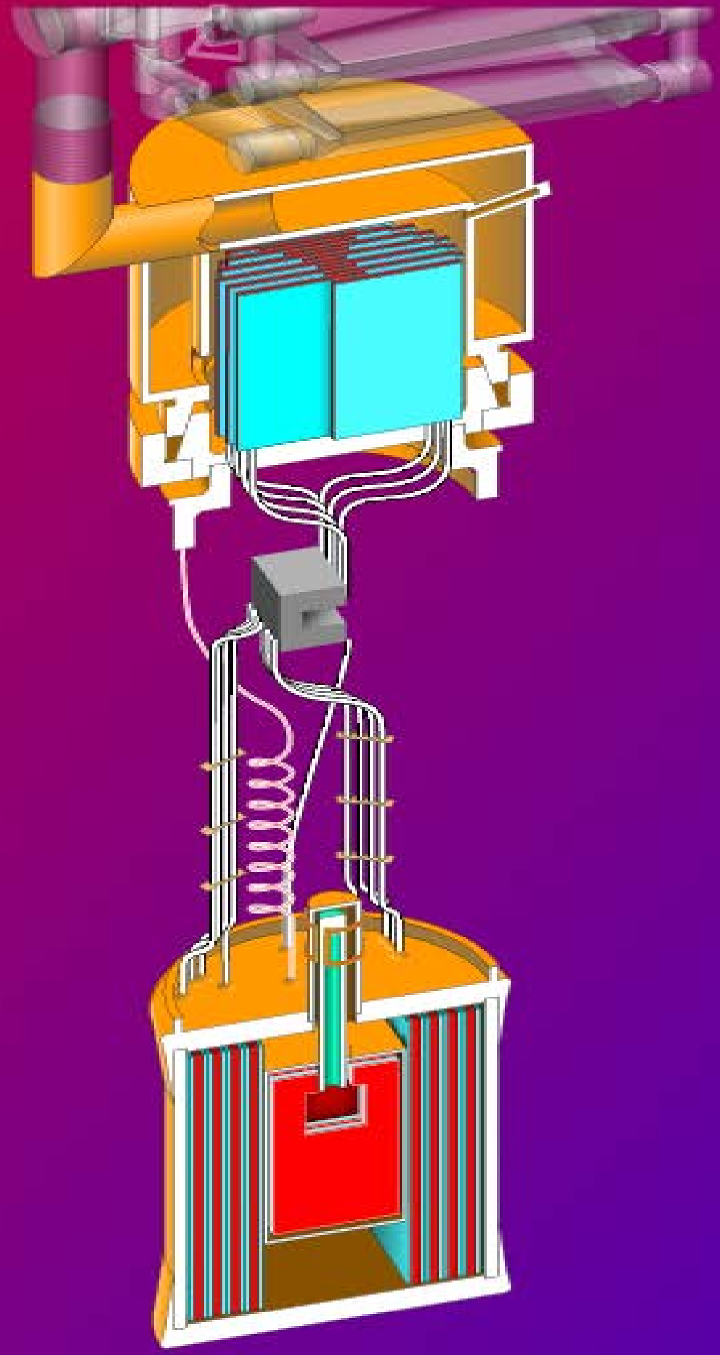


We add the mixing chamber round the MC cooling plates:



We add the mixing chamber round the MC cooling plates:

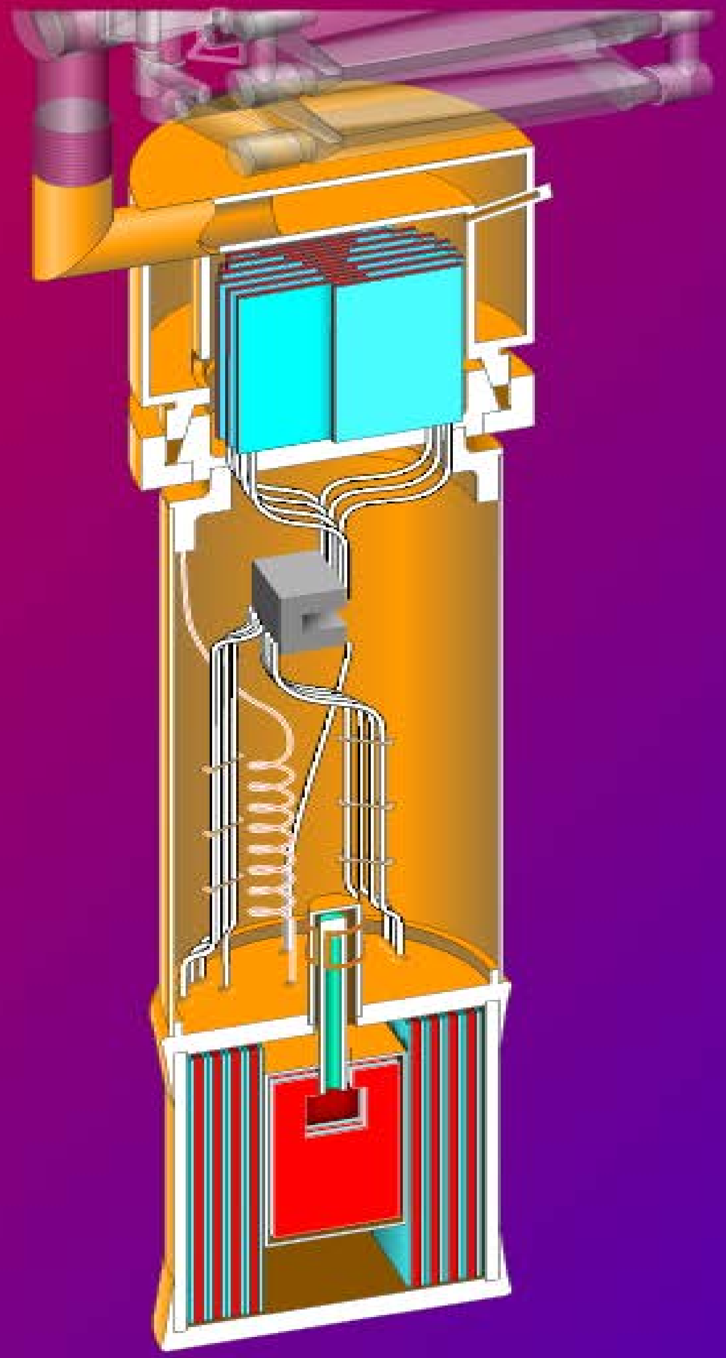
and the rest of the dilution refrigerator



We add the mixing chamber round the MC cooling plates:

and the rest of the dilution refrigerator,

And finally the rigid thin-walled plastic support tube for the nuclear stage.



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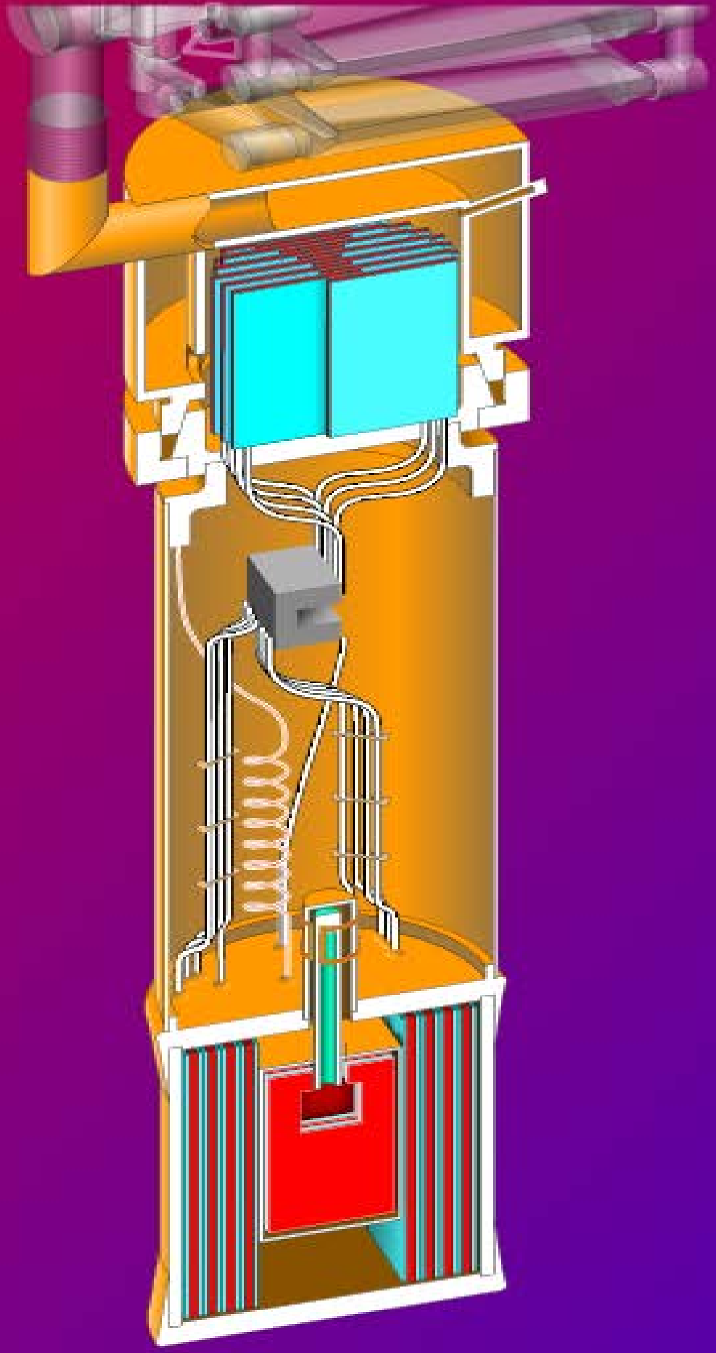
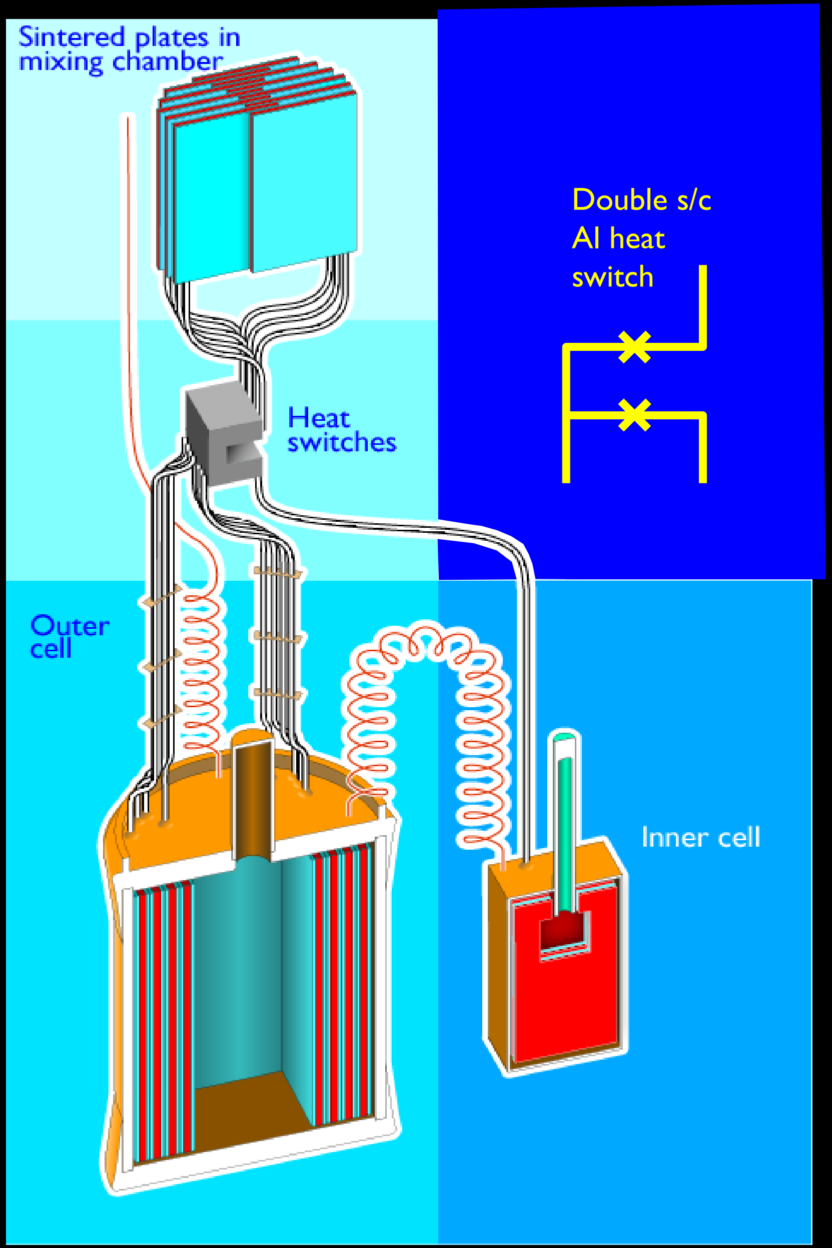


First, the “nuclear” stage is part of the experiment and dispensible.

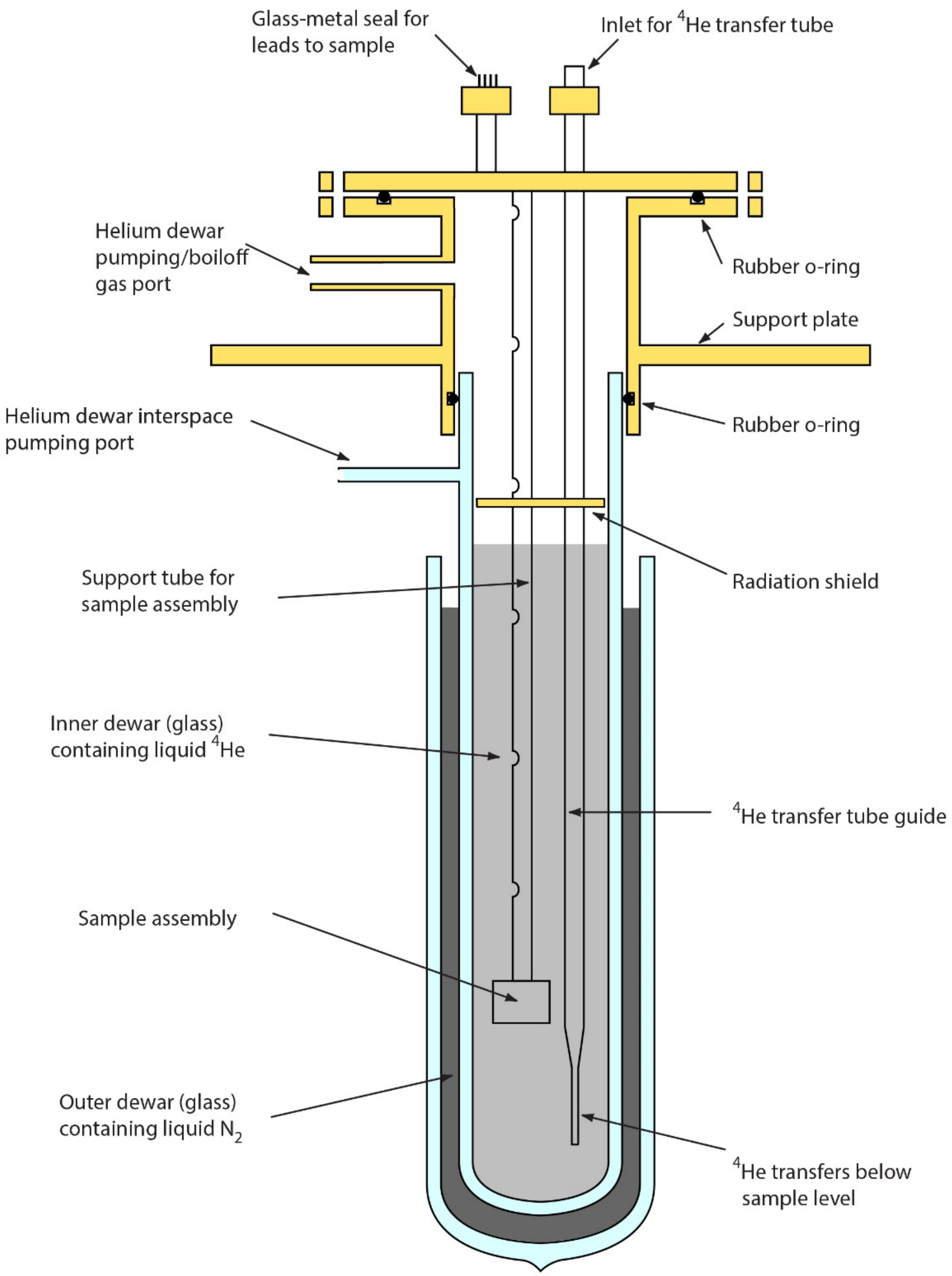
Secondly, the whole thing is built with very low level technology, with glued plastic pieces more like schoolboy model aeroplane methods. (This temperature regime is marginal enough already.)

# Lancaster Nuclear Demagnetization Cryostat




Temperature of superfluid He3
~80 micro Kelvin

Stay cold for up to a week




# Cryostat design considerations

- Conduction along glass walls
- Black body radiation from the silvered glass walls:

$$Q = \varepsilon \sigma T^4 S,$$

where $\varepsilon$ is the emissivity, $\sigma$=5.67x10$^{-8}$ WK$^{-4}$m$^{-2}$ is Stefan-Boltzmann constant

- Conduction through residual gas in the vacuum space - catastrophic if helium!
- Conduction through the support tube
- Conduction along experimental wires.

# Difficulty of 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

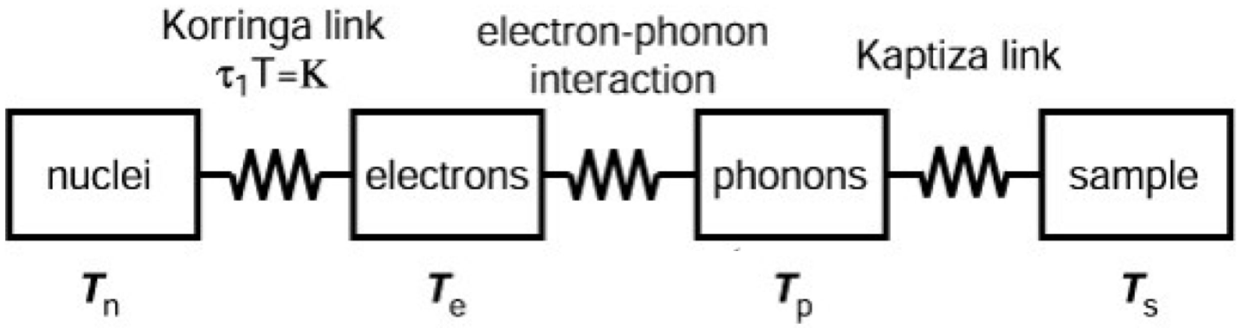
Heat flow $\dot{Q}$ through finite thermal resistance $R$ causes $\Delta T$

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

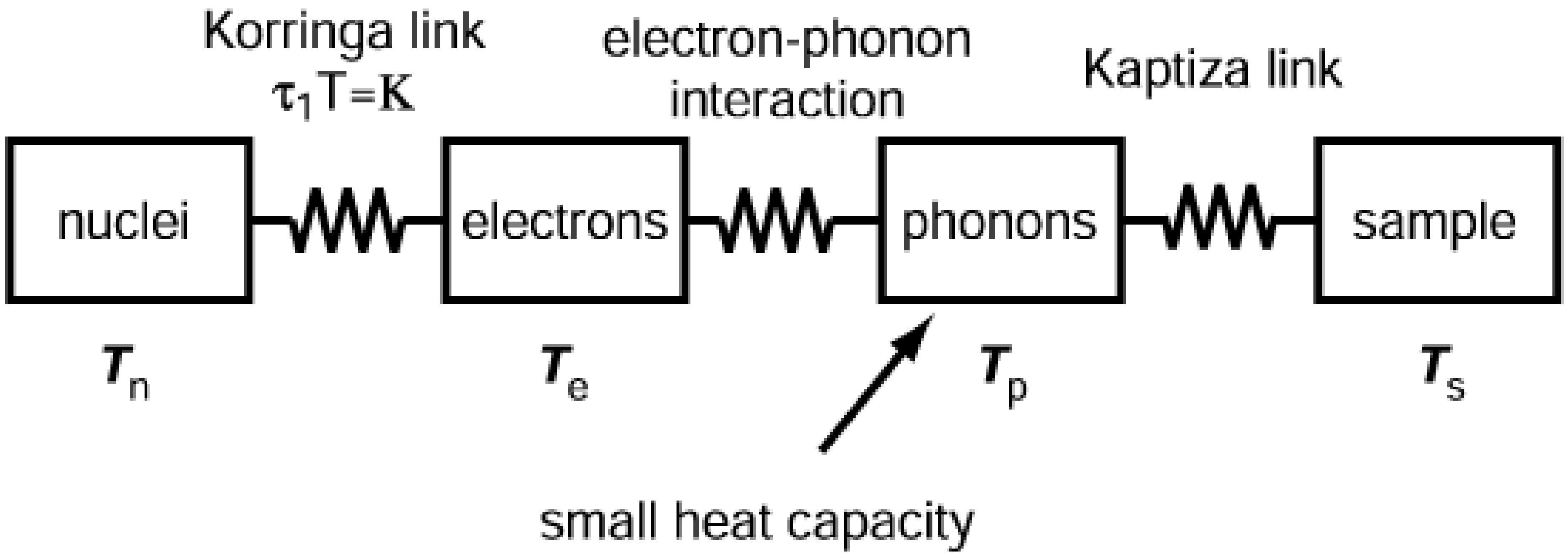
Thermal time constant is

$$\tau = RC$$

where $C$ is the sample heat capacity



# Difficulty of thermal contact




Colling Limits

- Nuclear spins $T_{nuc}$~100 pK
- Electrons $T_e$~1 μK
- $^3$He $T_3$~80 μK
- $^3$He-$^4$He mixtures $T_3$~100 μK ??

# Metallic contact

Heat flow $\dot{Q}$ through finite thermal resistance ***R*** causes **Δ*T*:** $\dot{Q} = \Delta T / R$

Weidemann-Franz law relates thermal and electrical conductivities *K*, σ

$$L_0 = \frac{K}{\sigma T} = 2.45 \text{ x } 10^{-8} \text{ W } \Omega \text{ K}^{-2}$$

Residual resistance ratio RRR = $\rho_{293K} / \rho_{4K}$ typically:

50 –100 for copper wire

1 for alloys - Stainless Steel, CuNi

but for pure material, heat treating can raise this to 1000 – 10000

Thermal resistance $R = \ell / K A$, length $\ell$, cross sectional area *A*

$$K = L_0 \frac{RRR}{\rho} T, \quad \rho = \sigma^{-1}$$

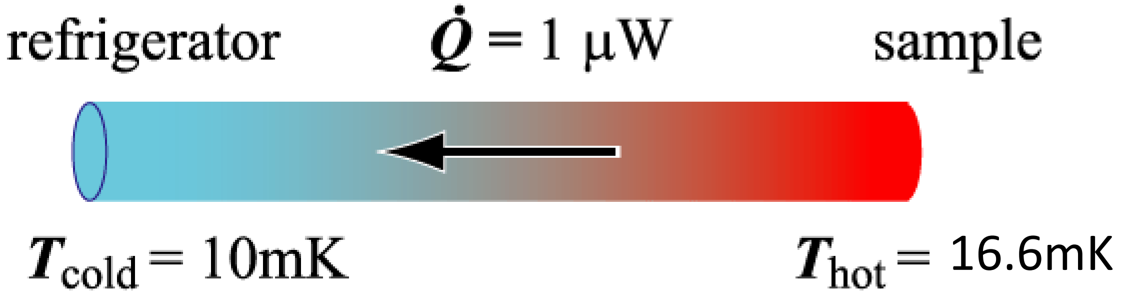
so $$\dot{Q} = \frac{KA}{\ell} \Delta T$$

If Δ*T* is big, must integrate

$$\dot{Q} = \int_{T_{cold}}^{T_{hot}} \frac{KA}{\ell} \, dT$$

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

Example: copper rod, one end at fridge base temp with a 1μW load…




$\rho = 1.7 \text{ x } 10^{-8}\ \Omega \text{ m}$   RRR=100

diameter = 1 mm   length = 1 cm

Metallic sinters

Boundary resistance between fine metal particles (sinters) and the He liquids inversely proportional to the sinter area.

T dependence complicated (treat with care):

| | |
|---|---|
| $^3He - ^4He$ | $R \propto 1 / T^2$ |
| $^3He$ $T > 10$ mK | $R \propto 1 / T^3$ |
| $^3He$ $T < 10$ mK | $R \propto 1 / T$ |

Screw Joints

screw joints can have contact resistance < $0.1\mu\Omega$ with care.

$R \sim 4 / T$ $K^2/W$

Insulators

Best material

Vespel SP22

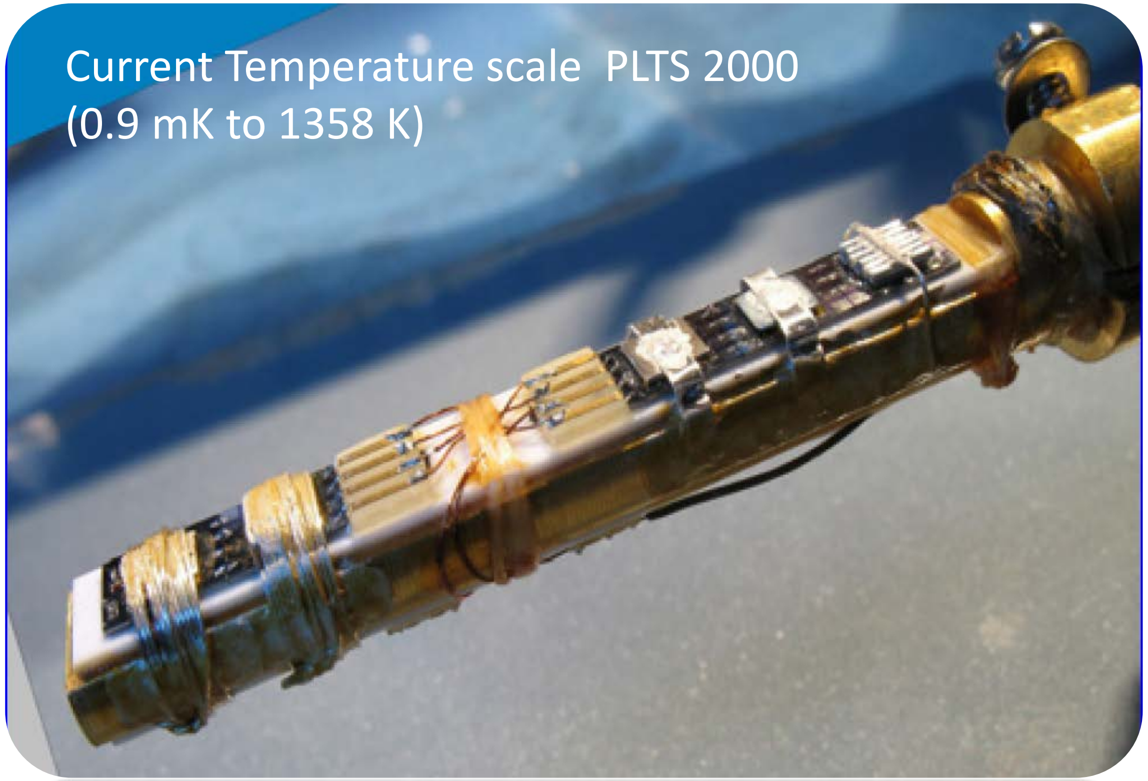
- ✓ strong, machinable
- ✓ low thermal conductivity

$K = 17 \times 10^{-4}\ T^2\ W\ m^{-1}\ K^{-1}$

Locatelli, Cryogenics **16**, 374, 1976

# Thermometry at Low Temperatures

- determination of temperature is often as difficult as the experiment itself
- anything can be used as a thermometer as long it has a temperature dependence
- temperature is by far the most uncertain scale …



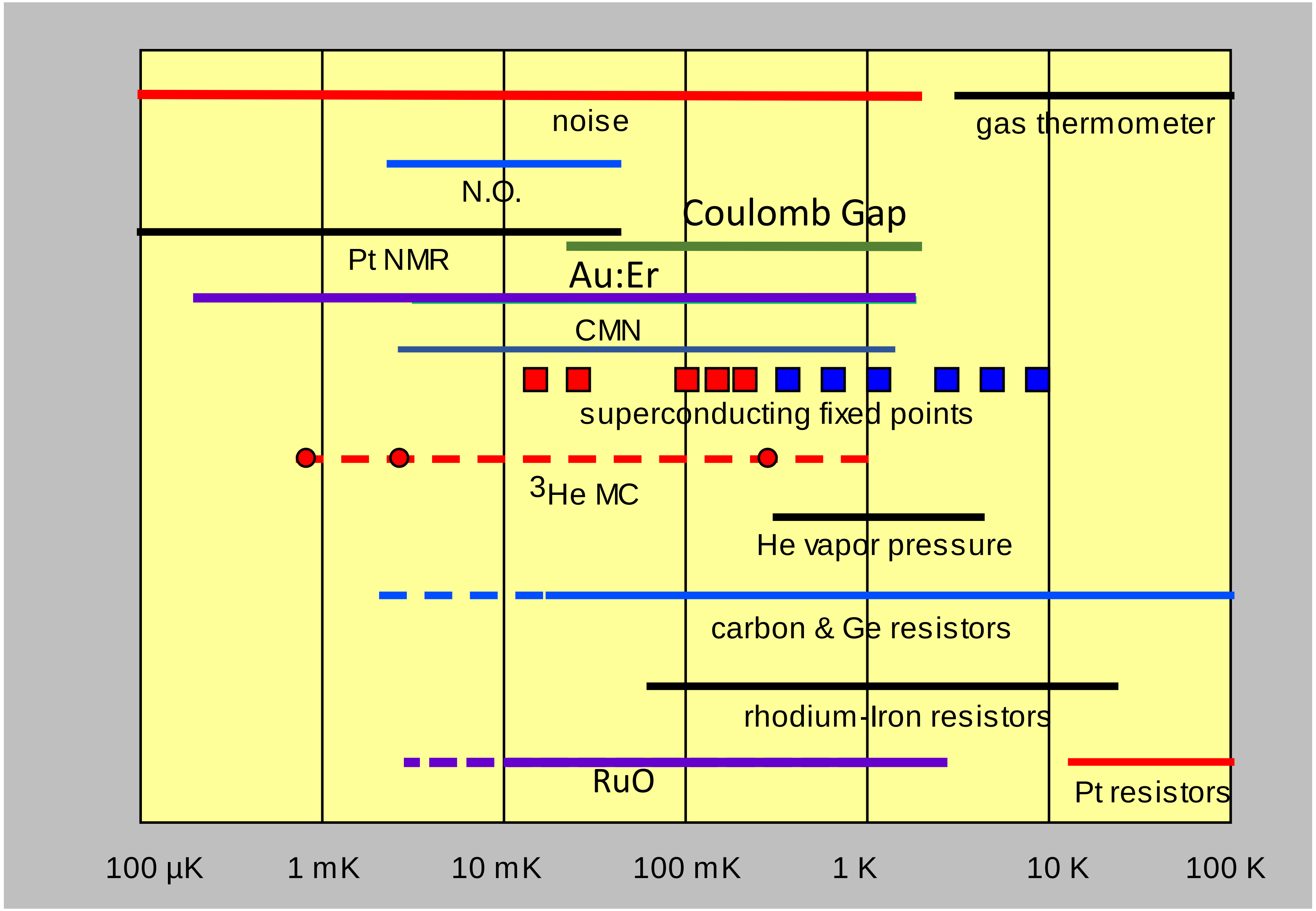

Primary thermometers: can be used without any prior calibration

Secondary thermometers: must be calibrated against an other thermometer

Primary thermometers
Superconducting fix points
Noise

Secondary thermometers
Resistance
Capacitance
Magnetic susceptibility

# Working Range of Thermometers



# Books


- O. V. Lounasmaa: “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 2007
- G. White & P. Meeson, “Experimental Techniques in Low-Temperature Physics”, Oxford University Press 2002
- C. Enss & S. Hunklinger, “Low-Temperature Physics”, Springer Verlag 2005
- J. W. Ekin, “Experimental Techniques for Low Temperature Measurements”, Oxford University Press 2011