

# PHY524 Cryogenics-5

Liquefaction, Helium & Dilution Refrigerator

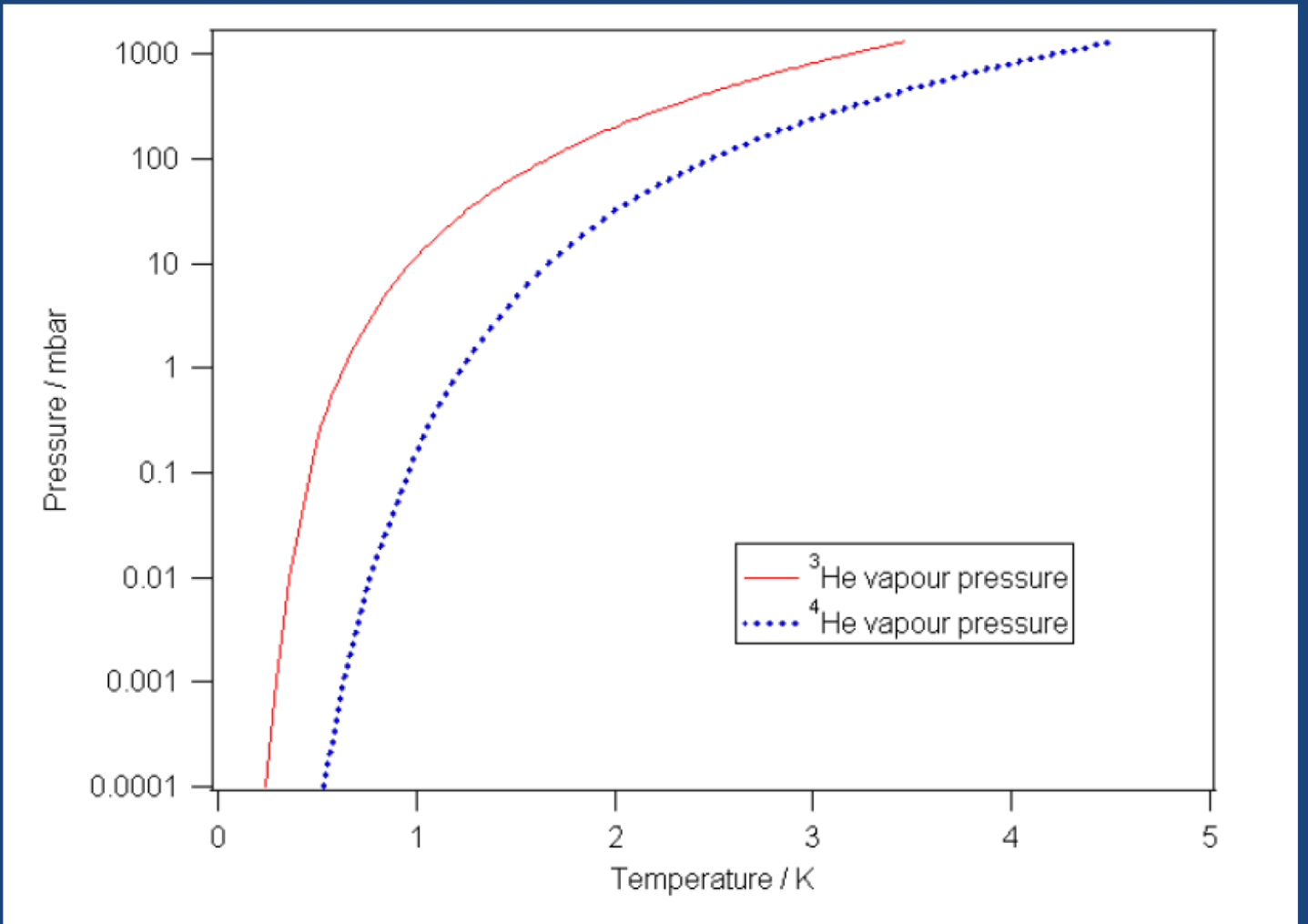
11/20/2025

Material adopted from lectures by Henri Godfrin, Grenoble

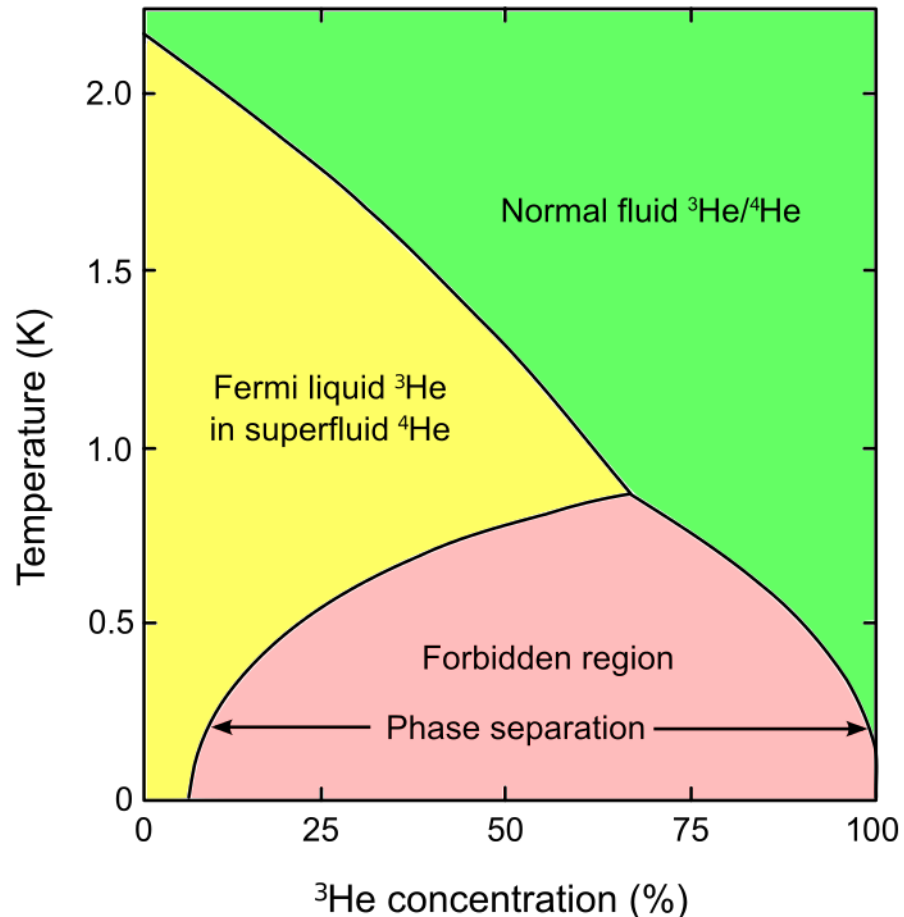
# Helium vapor pressure

Note the  
log scale

Fast rolloff of the  
Helium-4 vapor pressure  
below the  $\lambda$ -point



# Phase diagram of helium mixtures

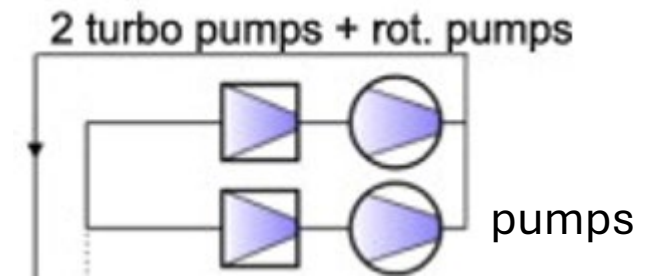


Diluting  $^3\text{He}$  into  $^4\text{He}$  depresses the superfluid transition temperature. Superfluid transition disappears completely for  $x > 67.5\%$

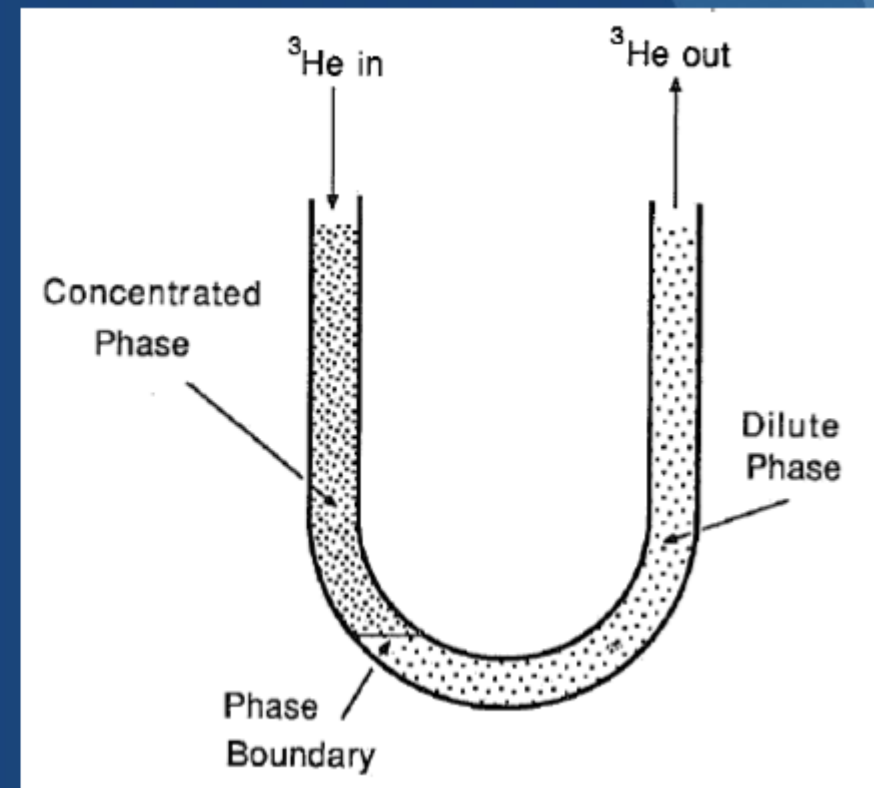
- As a mixture with  $x > 6.6\%$  is cooled, it will **separate** into 2 phases.
- One phase (the “concentrated phase”) will approach  $x=100\%$ , the other (the “dilute phase”) will approach  $x= 6.6\%$  @ 0 K.

**This finite solubility and phase separation is key to the dilution refrigerator process**

# DR cooling principles



- **Simplest model for the refrigerator is a U-tube**, with the concentrated phase on one side and the dilute side on the other.
- As observed previously, the less dense **concentrated phase floats on top of the dilute phase** since the superfluid has much higher density.
- Removing  $^3\text{He}$  from the dilute phase surface upsets the equilibrium.

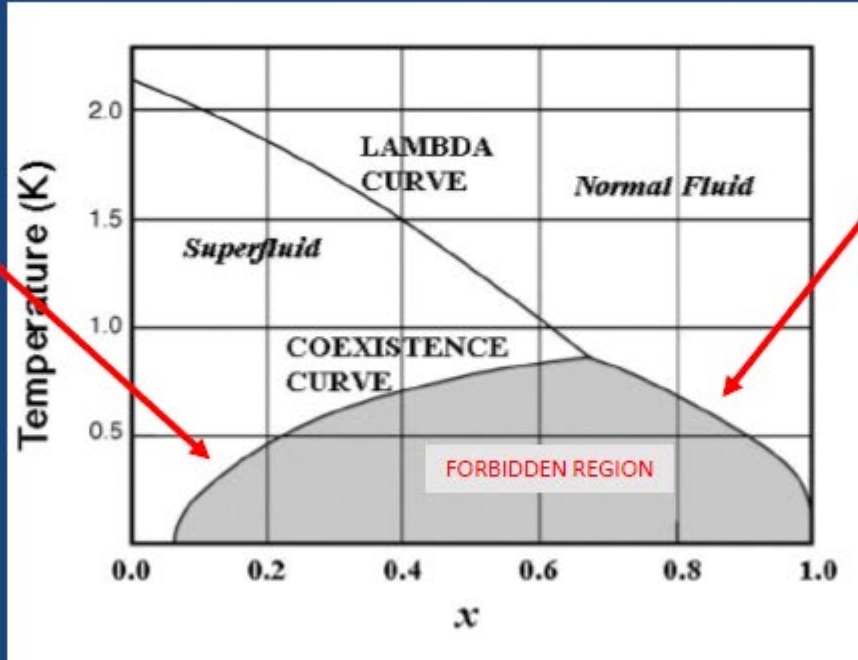


From Richardson & Smith (1998)

# $^3\text{He}$ - $^4\text{He}$ mixtures

$^4\text{He}$ -rich phase

Higher density



$^3\text{He}$ -rich phase  
(essentially pure  $^3\text{He}$ )

Lower density, floats on  
top of the  $^4\text{He}$ -rich phase

$^4\text{He}$  obeys **Bose statistics** and will condense into the quantum mechanical ground state at low enough temperature, i.e., superfluid.

In a helium mixture, the condensed  $^4\text{He}$  acts as an “inert superfluid background” which contributes to the volume of the liquid and the effective mass of the  $^3\text{He}$  atoms, but has **zero heat capacity**.

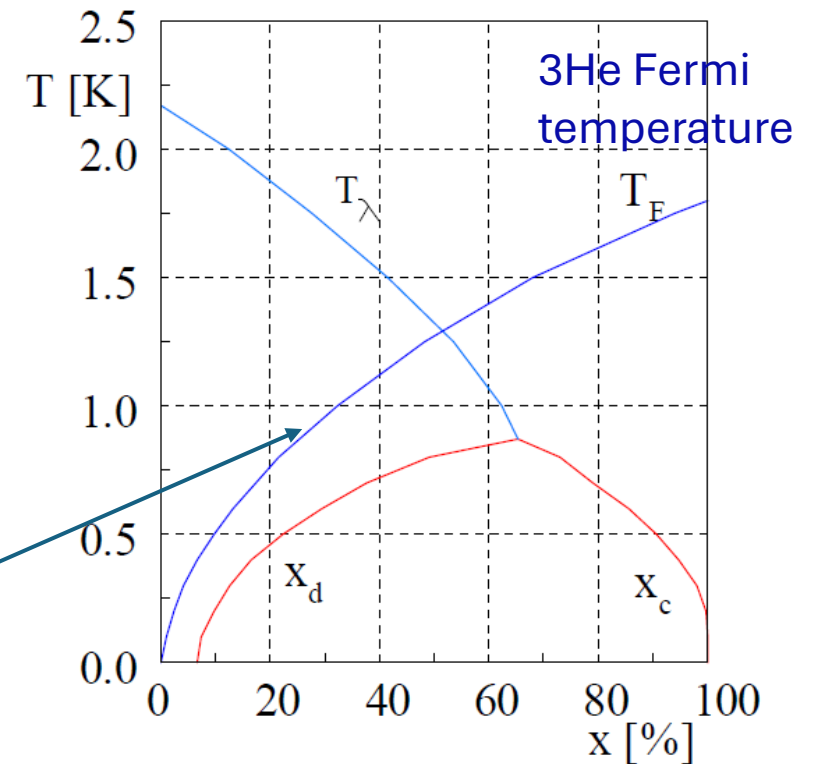
The  $^3\text{He}$  obeys **Fermi statistics** and will behave in a similar way to conduction electrons in a metal.

However, whereas conduction electrons have a Fermi temperature of 10,000 K, **the Fermi temperature of the  $^3\text{He}$  is around 1 K**.

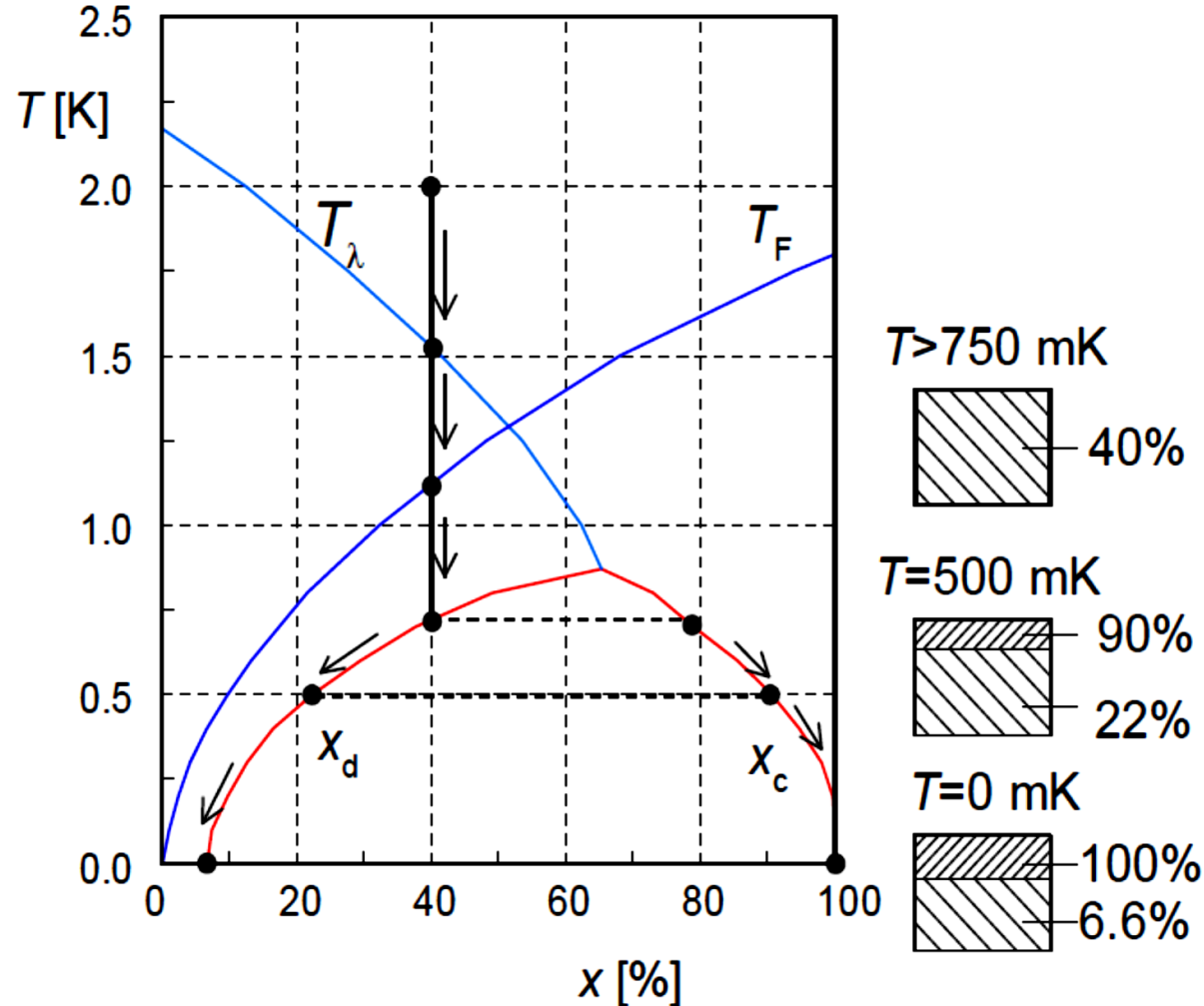
# Phase Separation & Finite Solubility

- The van der Waals forces between 4He and 3He are identical, since their electronic structure is identical. However, the **3He atoms have larger zero-point motion** (due to their smaller mass), hence the 4He atoms occupy a smaller volume than the 3He atoms, and the **3He atoms will feel a stronger attraction to a 4He atom**.
- However, recall that the 3He atoms must obey the **Pauli Exclusion Principle**: If we add additional 3He atoms in the liquid, they have to go into successively higher energy states
- Eventually, all the states up to the Fermi energy will be filled with two 3He atoms. *The binding energy of the 3He has to decrease if the number of atoms is increased.*
- **The chemical potential of a 3He atom** in the dilute mixture is thus:

$$\frac{\mu_{3,d}(0)}{N_0} = -\epsilon_{3,d}(x) + k_B T_F(x)$$



# Solubility decreases upon cooling



Removing  $^3\text{He}$  from the top of the dilute phase will reduce the concentration below the 6.6% equilibrium concentration.

- The osmotic pressure difference will drive the flow of  $^3\text{He}$  across the phase boundary.

Since we will be dealing with helium mixtures with varying concentration and temperature, we will see osmotic pressure differences.

# History

- Dilution refrigerators are a real workhorse apparatus for millikelvin cryogenics; they are the only cooling machine capable of obtaining millikelvin temperatures **continuously**.
- Very useful at higher Sub-1 Kelvin temperatures as well due to high cooling power compared to other refrigeration processes in the same range.
- Cooling comes from the heat of mixing of Helium-3 and Helium-4, the two common helium isotopes.

The dilution refrigerator principle was suggested by **Heinz London in 1952**

**H. London, G.R. Clarke, and E. Mendoza** proposed a prototype of continuous refrigerator in 1962

**It was realized in 1964 in the Kamerlingh Onnes Laboratorium at Leiden University**

*Das, P.; Ouboter, R. B.; Taconis, K. W. (1965). "A Realization of a London-Clarke-Mendoza Type Refrigerator". Low Temperature Physics LT9. p. 1253.*  
**( $T_{min} \sim 220 \text{ mK}$ )**

B.S. Neganov and co-workers in **Dubna** and H.E. Hall and co-workers in **Manchester** went below 100 mK (1966). Dubna rapidly reached **25 mK**.

**The principles and methods of dilution refrigeration** have been substantially developed by J. Wheatley et al. at **La Jolla**.

**Modern « wet » refrigerators** are based on the **Grenoble design** by Frossati and coworkers.

The development of sintered silver heat exchangers  $T_{min}$  led to  **$T_{min} \sim 2 \text{ mK}$** .

« **Dry** » refrigerators were developed

- by K. Uhlig et al. **on GM coolers** (1993),
- and **on Pulse-tube coolers** by K. Uhlig et al. (2002) and H. Godfrin (1999 to 2003, date of first commercial unit, delivered by l' Air Liquide)

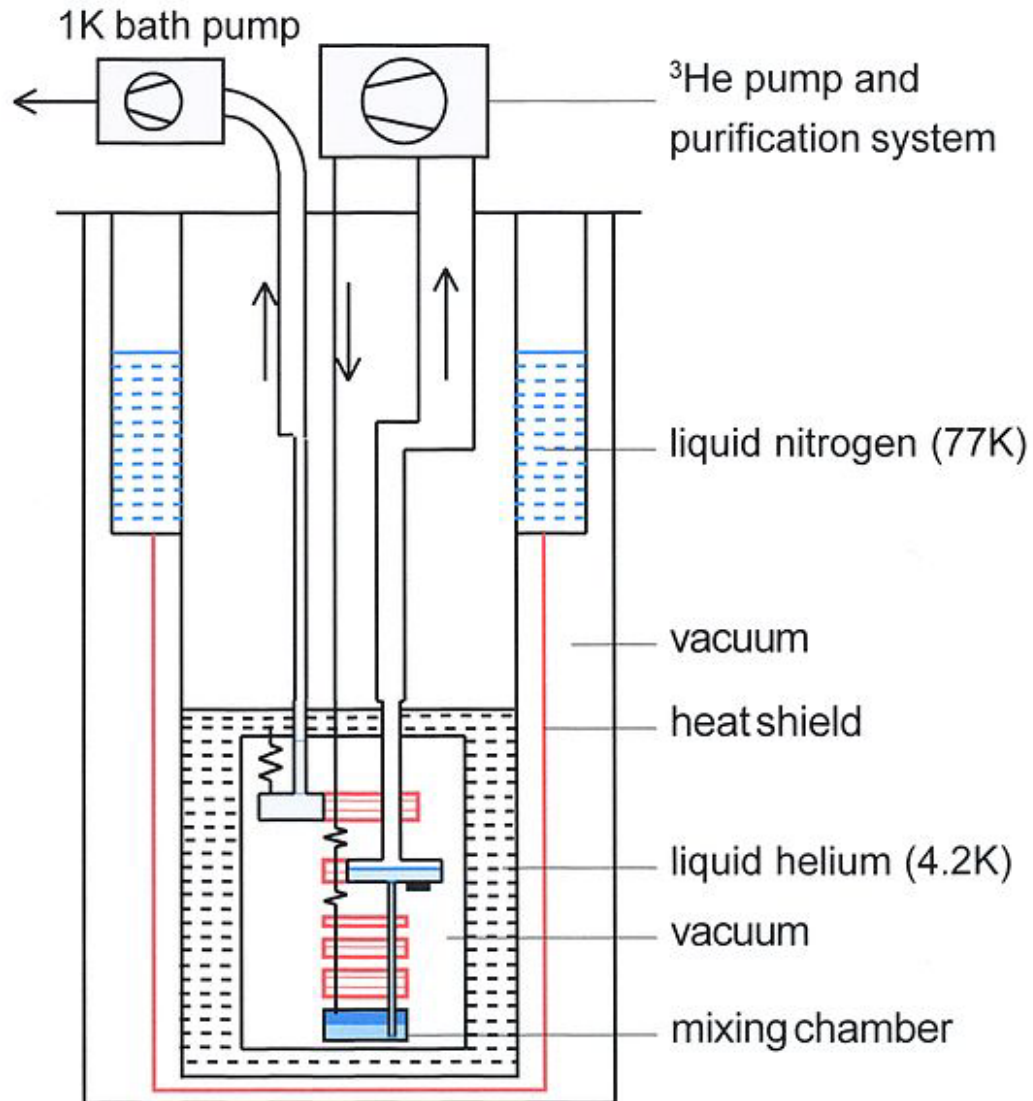
Lancaster has the present record of low temperatures, **1.75 mK**



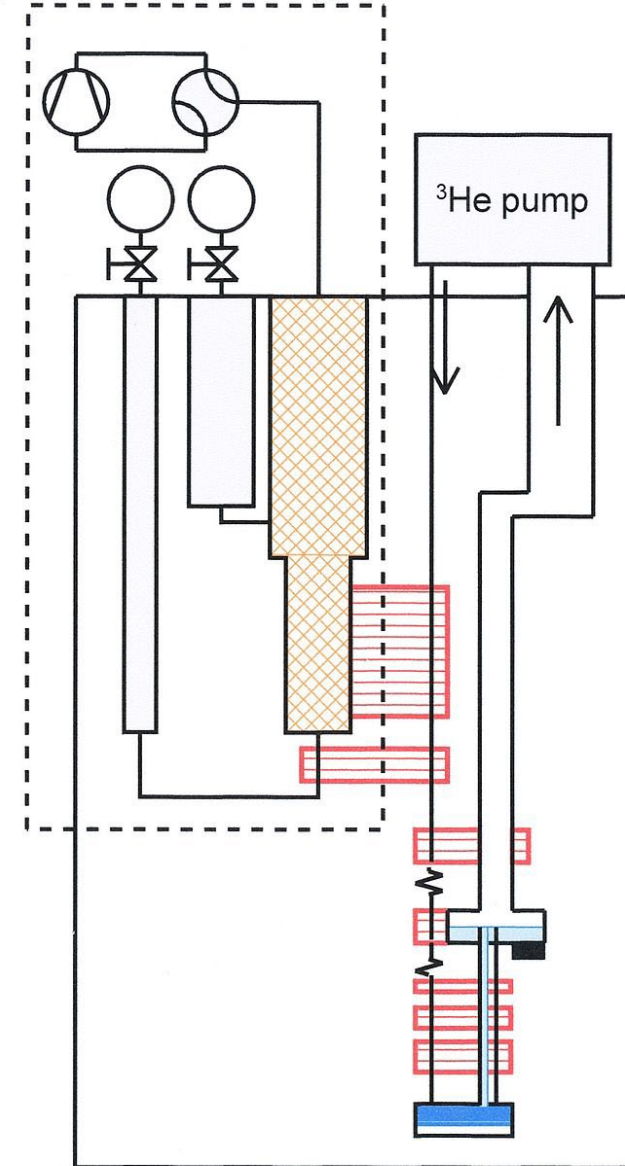
# Wet vs Dry Dilution Refrigerator (DR)

A 'wet' DR:

A 1K pot from Lhe is used to cool  $^3\text{He}$



pulse-tube refrigerator



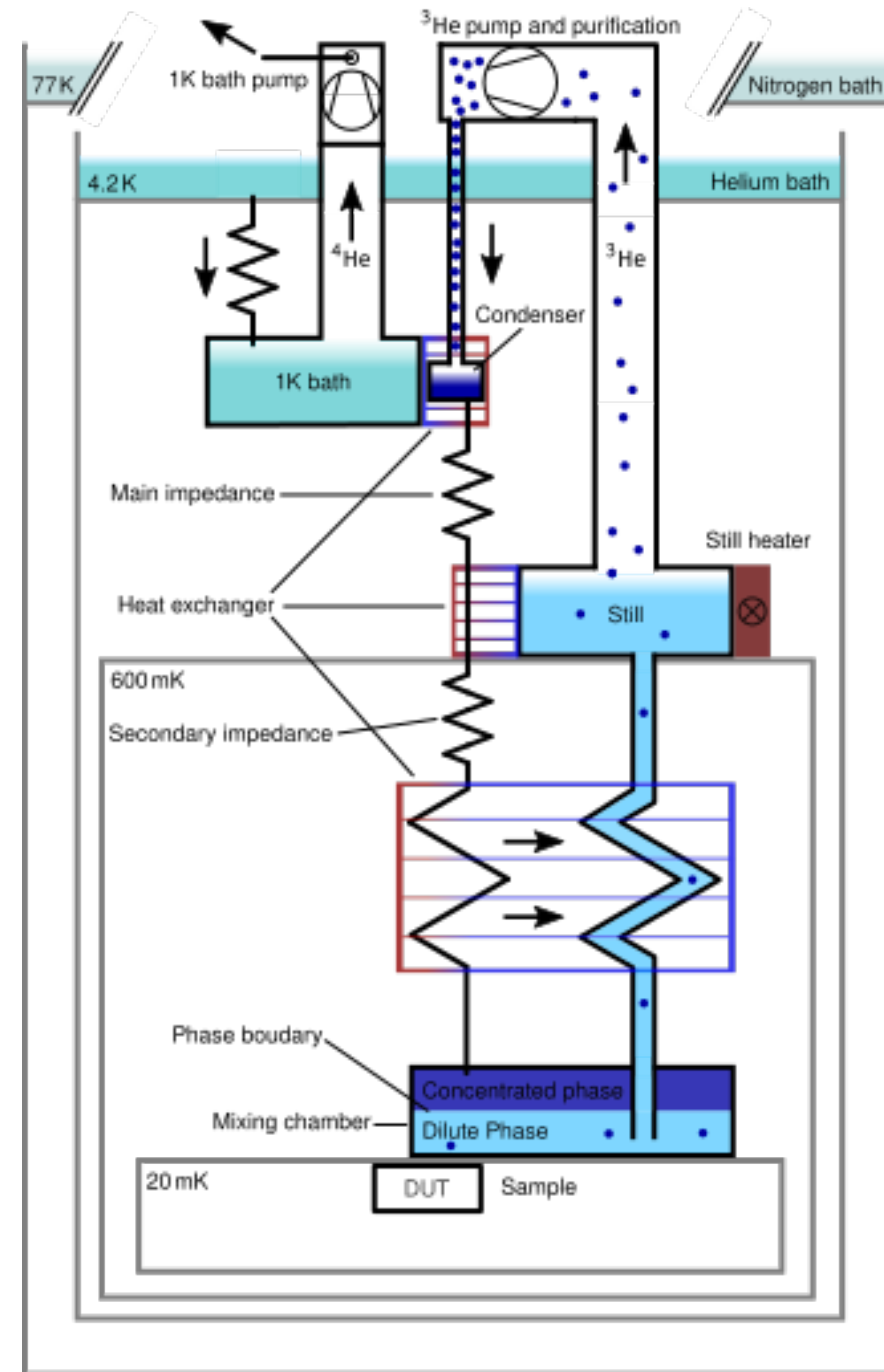
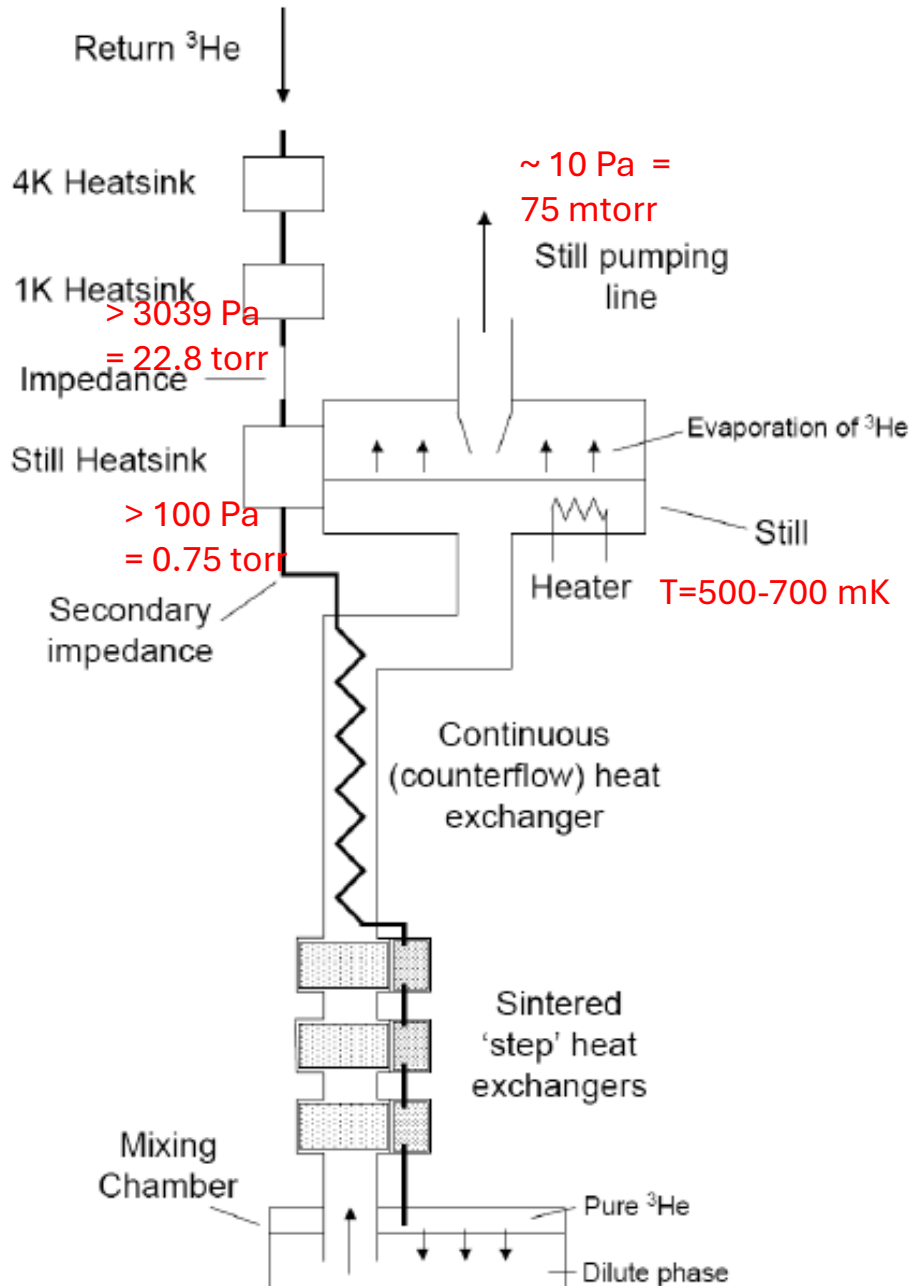
A 'dry' DR:

In the left corner is the **two-stage PTR** cooling the radiation shields and the incoming  $^3\text{He}$  (also via the regenerator of the PTR)

PTR: see previous lecture

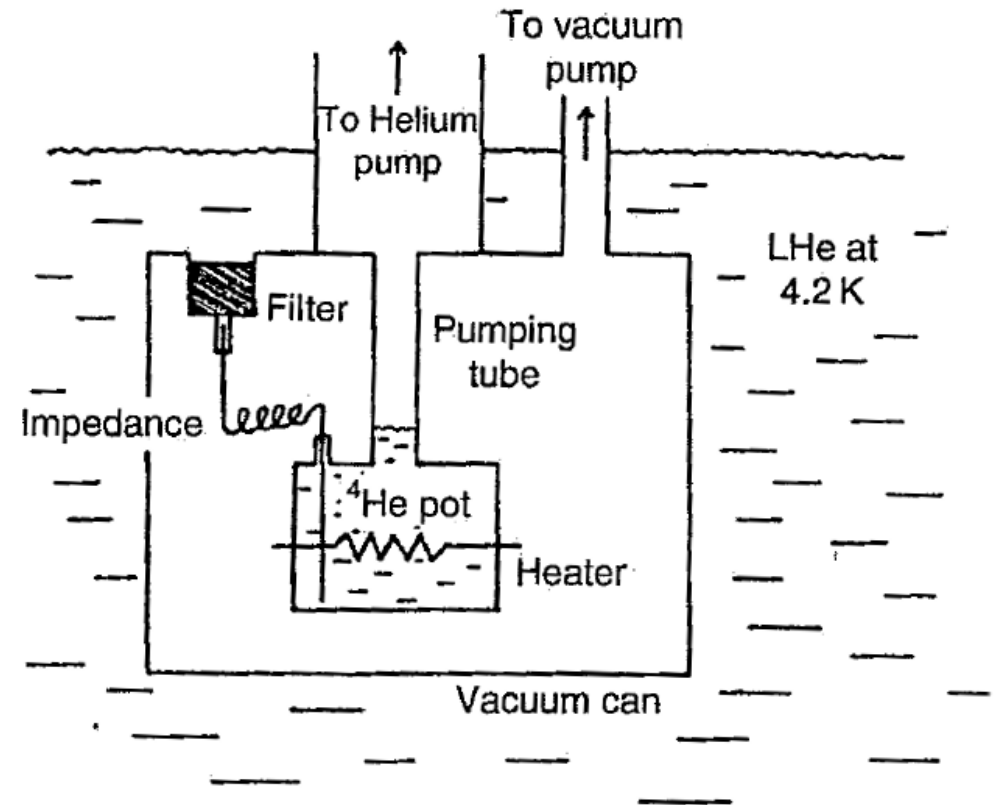
# DR loop

- $^3\text{He}/^4\text{He}$  mixture from the room temperature pump system is cooled (by 4K heatsink & 1K pot) and liquified at/above the still.
- Cooled further on the way to the **mixing chamber (MC)**.
- $^3\text{He}/^4\text{He}$  phase separates inside the **MC**.
- $^3\text{He}$  is pumped away from the **still**.
  - a heater is in the still to ensure enough  $^3\text{He}$  are in vapor.



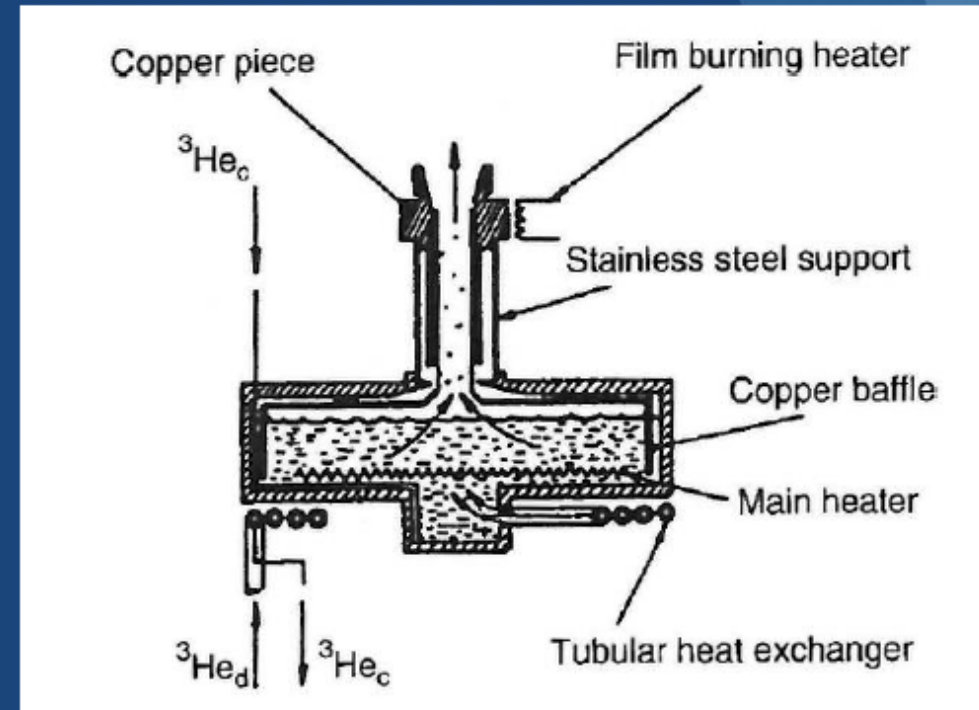
# 1K pot: precool incoming $^3\text{He}$ gas

- A helium-4 bath connected to a pump. Approximately 50% of the liquid volume will be evaporated during pump down.
- 1K pot: a small pot of Helium-4 that is fed from a bath at atmospheric pressure.
  - Much lower pumping capacity required.
  - Requires a cryogenic vacuum seal for the inner vacuum can.
  - Must be aware of contamination blocking the flow impedance.
- 1K pot is used to cool incoming  $^3\text{He}$  for further condensation in the still and mixing chamber.



# Still: remove $^3\text{He}$ from dilute phase

- The main function of the still is to allow  $^3\text{He}$  to be pumped away from dilute phase of the system.
- The optimum still temperature is  $\sim 600 - 700$  mK. Pumping on  $^3\text{He}$  will tend to cool towards 400 mK (similar to a  $^3\text{He}$  fridge). A heater is used to raise the temperature to drive the helium evaporation.
- To keep the amount of  $^4\text{He}$  being pumped to a minimum, the still will typically incorporate a system to suppress the superfluid film creep from the dilute phase.



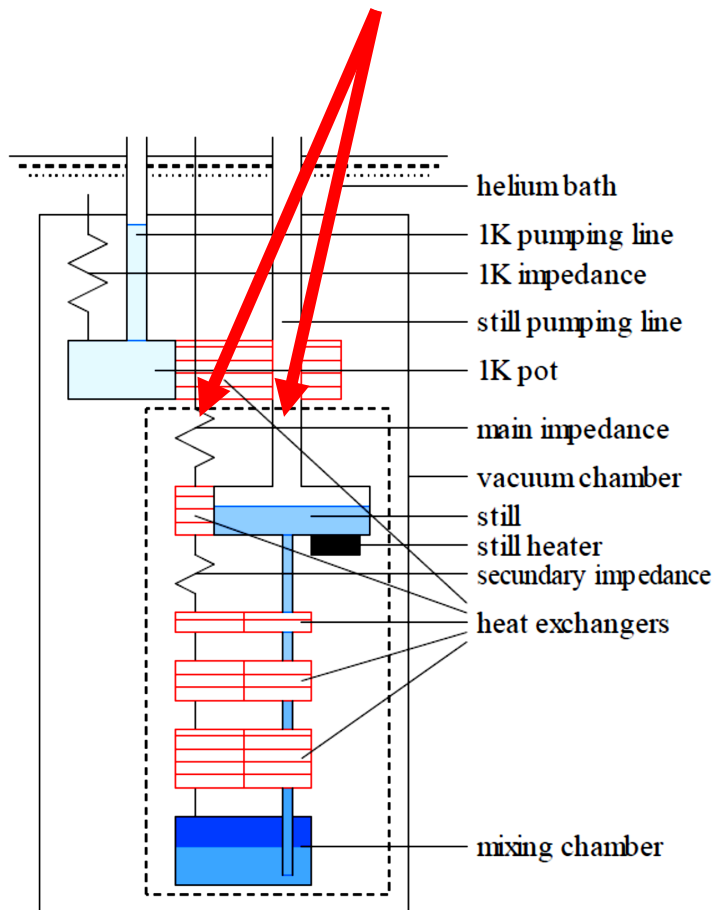
Pobell (2007)

# Why is the 1K pot needed?

A: Precooling the incoming  $^3\text{He}$  gas; Balance cooling power available in still for a stable continuous-flow operation.

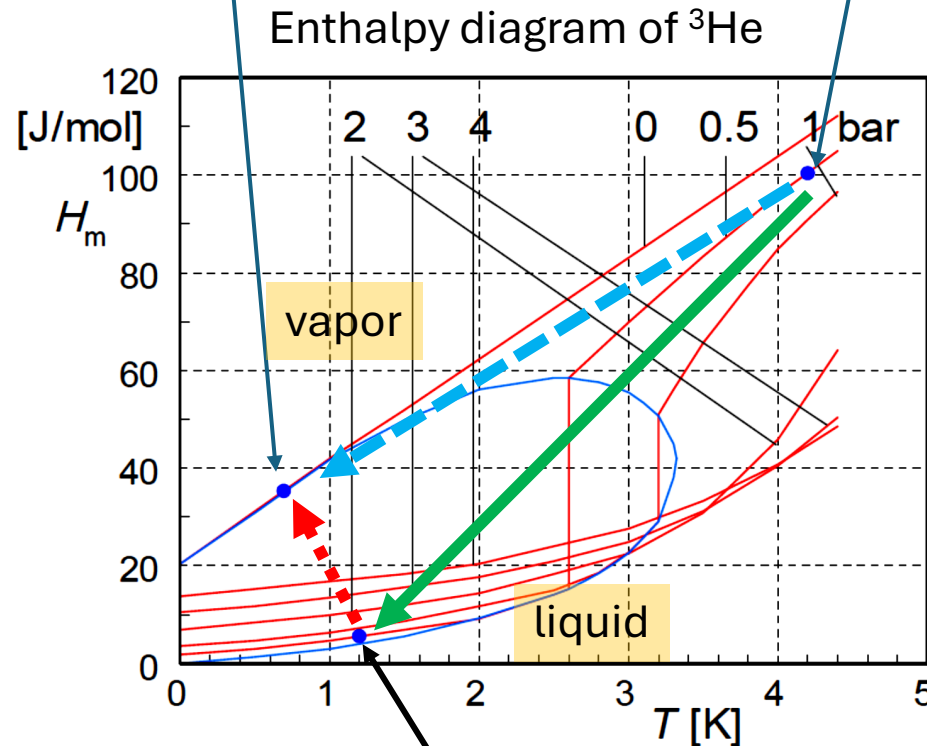
## Global enthalpy balance of DR:

Every incoming  $^3\text{He}$  atom can be cooled by an outgoing  $^3\text{He}$  atom in still:



$^3\text{He}$  leaves the still at 0.7 K and  $P \sim 0$ . Its enthalpy is 35 J/mol.

Incoming  $^3\text{He}$  at  $P=0.5$  bar and 4.2 K has a enthalpy of 99 J/mol



With a 1K pot at 1.2 K,  $^3\text{He}$  can be condensed to liquid and has enthalpy of 6 J/mol!

The deficit in enthalpy between the outgoing & incoming  $^3\text{He}$  is huge: 64 J/mol !

$$\dot{Q} = \dot{n}_3(35 - 99) = -64 \frac{\text{J}}{\text{mol}} \dot{n}_3.$$

Without 1K pot: *negative* balance, External *cooling* needed.

2. Using a 1K pot at 1.2 K:

$$\dot{Q}_S = \dot{n}_3(35 - 6) = 29 \frac{\text{J}}{\text{mol}} \dot{n}_3.$$

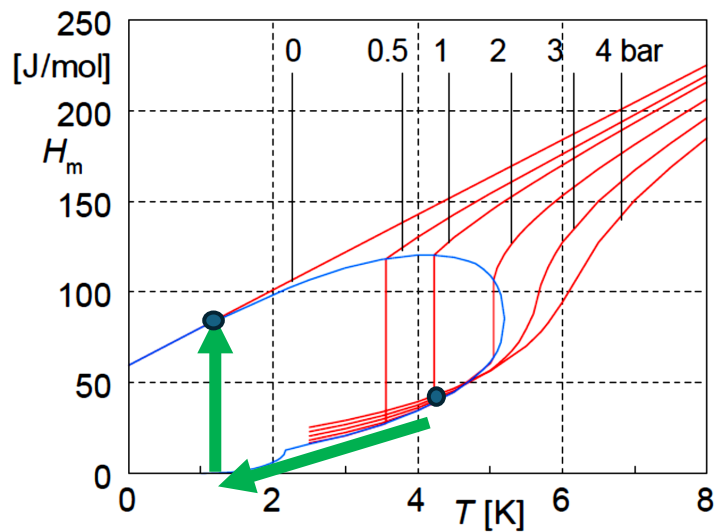
Incoming  $^3\text{He}$  is already liquefied and has a small enthalpy. A *positive* balance  $\rightarrow$  An external **heating** (usually at the still...) needed.



# Global enthalpy balance of DR

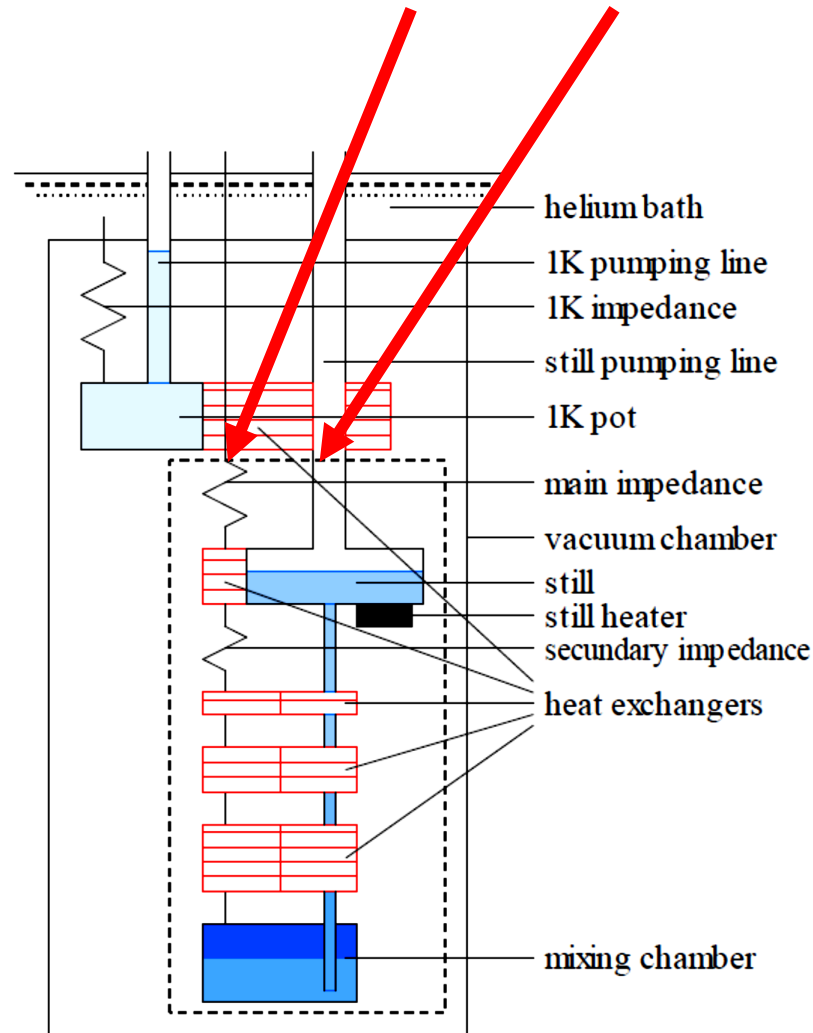
(only pure  $^3\text{He}$  flows in and out the sub-system)

Enthalpy diagram of  $^4\text{He}$

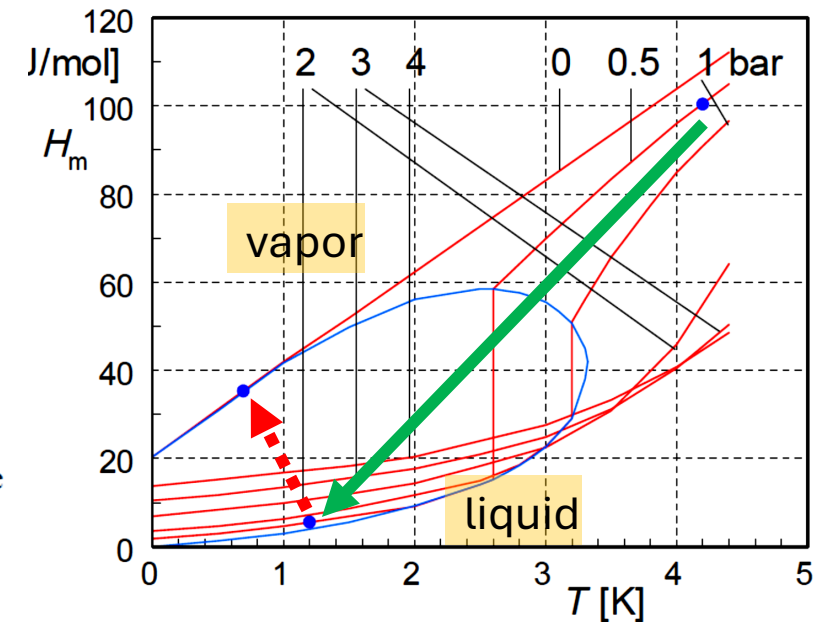


## 1K pot cooling power:

Cool liquid from 4.2K to 1.2K, then latent heat of liquid at 1.2 K



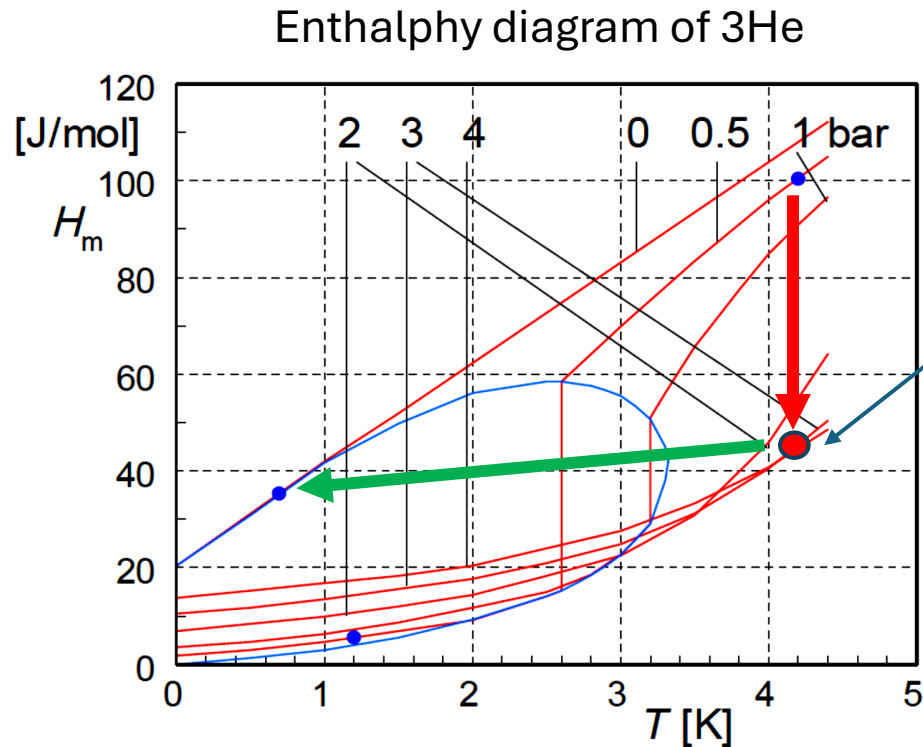
Enthalpy diagram of  $^3\text{He}$



**Cooling power needed** to change the  $^3\text{He}$  from gas at 0.5 bar and 4.2 K to liquid at 0.5 bar and 1.2 K, is

$$\dot{Q}_{1K} = \dot{n}_3 (99 - 6) = 93 \frac{\text{J}}{\text{mol}} \dot{n}_3$$

# Alternatively, precool the incoming $^3\text{He}$ at $\sim 3$ bars



At 4.2 K the minimum enthalpy (vs. Pressure) is  $H_m \approx 42$  J/mol near **3 bars**,

just above the enthalpy of the vapor at 0.7 K (35 J/mol).

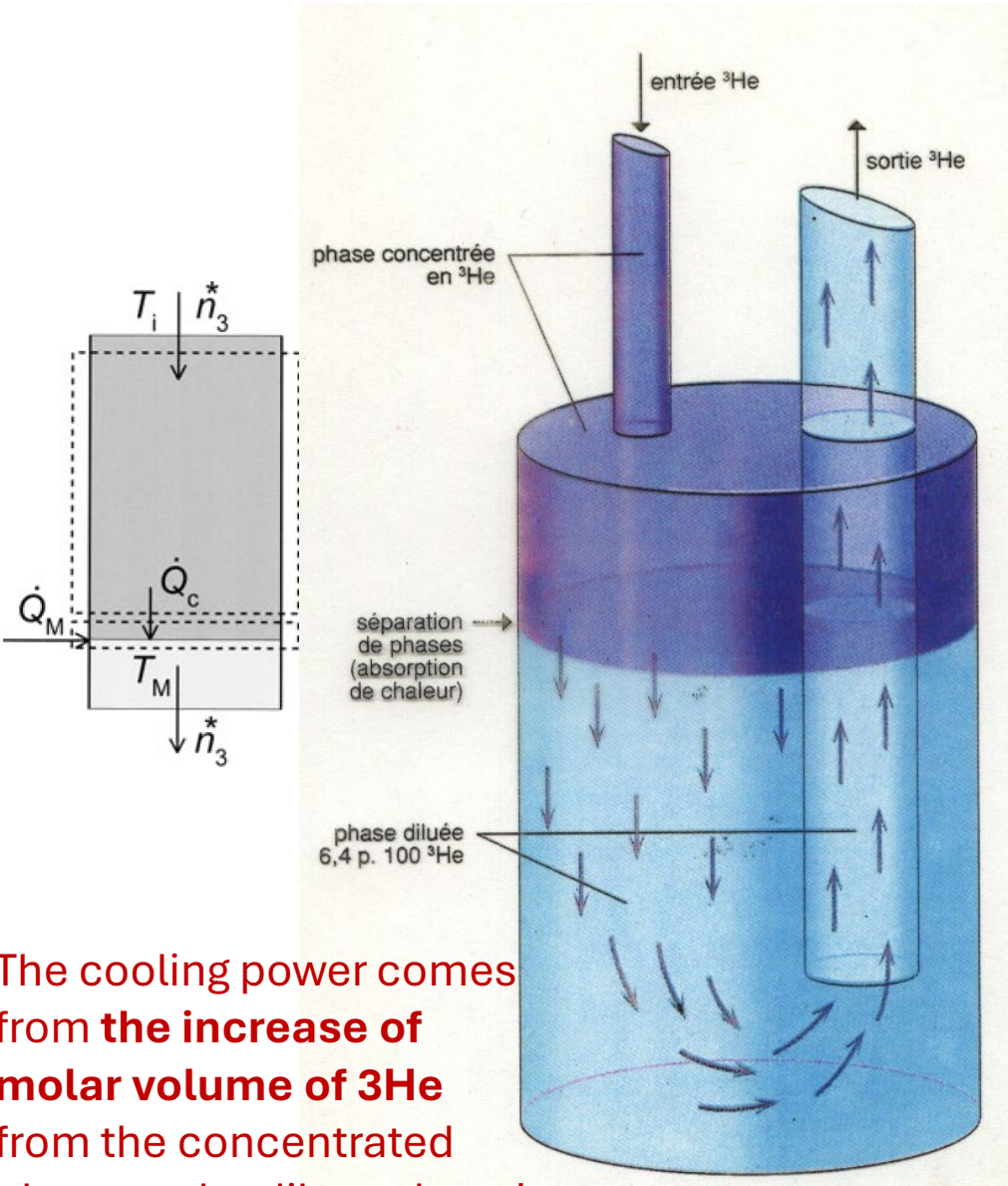
→ steady state is possible using a *heat exchanger* to precool the incoming  $^3\text{He}$  at 3 bars (gas) by outgoing  $^3\text{He}$  vapor from still at 0.7 K!!!

A compressor needed (essentially during initial condensation)

J. Kraus, Cryogenics **17**, 173 (1977)

A.T.A.M. de Waele, A.B. Reekers, H.M. Gijsman, Cryogenics **17**, 175 (1977)

# The Mixing Chamber: cooling is due to *expansion*, i.e., $^3\text{He}$ expanding from concentrated into dilute phase



second law, reversible cooling power

$$\dot{Q}_M = \dot{n}_3 T_M [S_3(T_M, x_s) - S_c(T_M)]$$

since

Entropy in  
dilute  $^3\text{He}$

Entropy in  
concentrated,  
pure  $^3\text{He}$  ( $x \approx 1$ )

Fermi liquid:  $S_F = \frac{\pi^2}{2} R \frac{T}{T_F(x)}$

so

$$\dot{Q}_M = \dot{n}_3 \frac{\pi^2}{2} R T_M^2 \left( \frac{1}{T_F(x_s)} - \frac{1}{T_F(1)} \right)$$

since

$$T_F(x) \sim \left[ \frac{N_A}{V_m(x)} \right]^{2/3}$$

we get

$$\dot{Q}_M = \dot{n}_3 (H_3 - H_{3c})$$

$$\dot{Q}_M \sim \dot{n}_3 T_M^2 (V_{ms}^{2/3} - V_{mc}^{2/3})$$

Molar volumes:

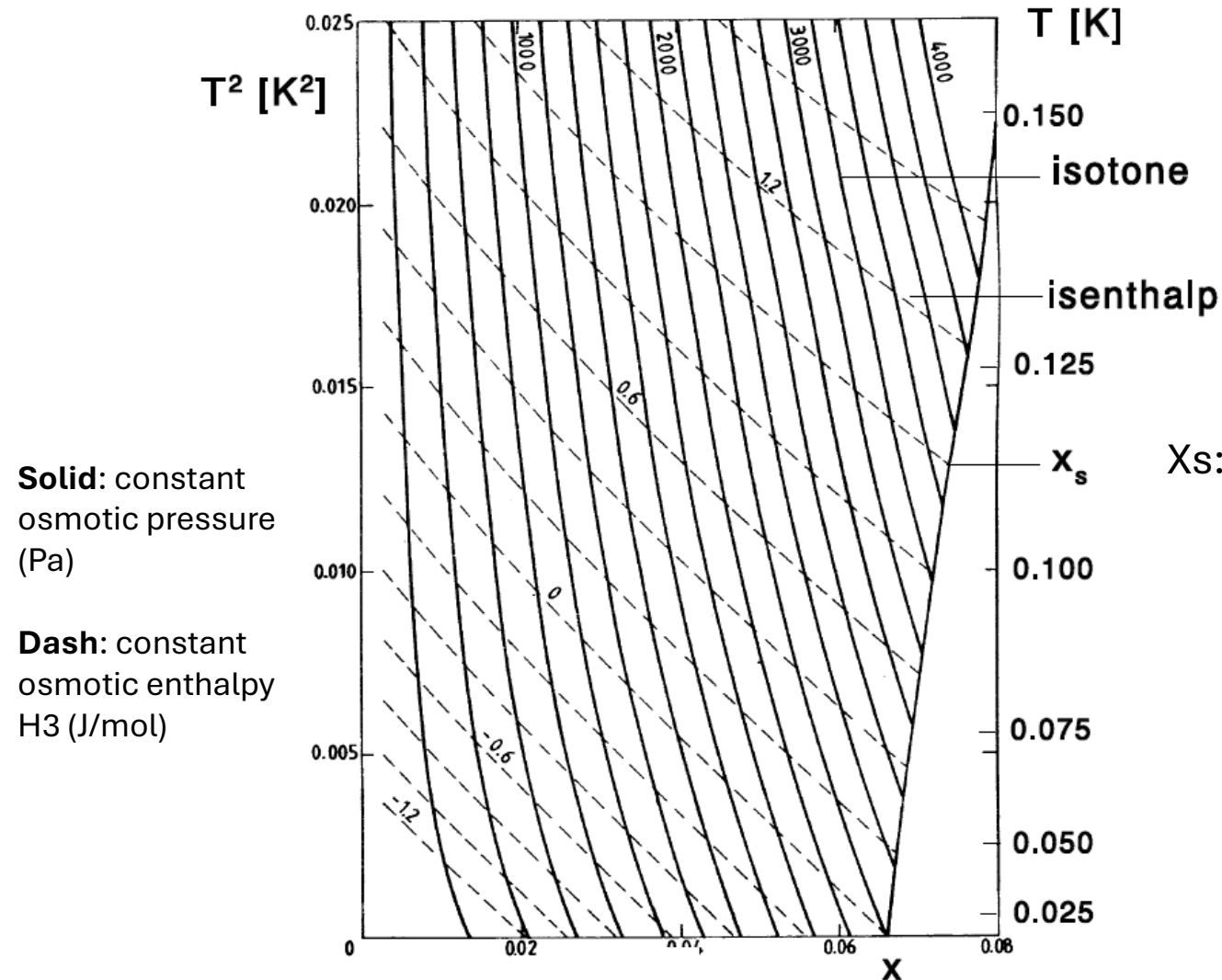
with  $V_{ms} = 426 \text{ cm}^3/\text{mol}$  and  $V_{mc} = 37 \text{ cm}^3/\text{mol}$

$$T_F(x_s) = 393 \text{ mK and } T_F(1) = 1.8 \text{ K}$$

The cooling power comes from **the increase of molar volume of  $^3\text{He}$**  from the concentrated phase to the dilute phase!



# Osmotic enthalpy of $^3\text{He}$ mixtures



special cases

	approximate $H_3$
saturated concentration $x = x_s(T)$	$96T^2$
constant concentration $x = x_s(0)$	$84T^2$
constant osmotic pressure $\Pi = \Pi_s(0)$	$54T^2$
pure $^3\text{He}$	$12T^2$

$x_s$ : the saturation concentration of the dilute phase.

# Cooling power at the mixing chamber (MC)

The relation for the molar enthalpy of pure  $^3\text{He}$  as a function of temperature is

$$H_{3c} = 12T^2. \quad (28)$$

It should be noted that the numbers in the expressions (25)-(28) are only approximate.

In terms of the osmotic enthalpy the cooling power of the mixing chamber can be written as

$$\dot{Q}_M = \dot{n}_3 [H_{3s}(T_M, x_s) - H_{3c}(T_i)] . \quad (29)$$

Now it is no longer necessary to limit the expression to reversible mixing. The temperature  $T_i$  is the temperature of the  $^3\text{He}$  entering the mixing chamber in the concentrated phase. With Eqs.(26) and (28) Eq.(29) gives

$$\dot{Q}_M = \dot{n}_3 (96T_M^2 - 12T_i^2) . \quad (30)$$

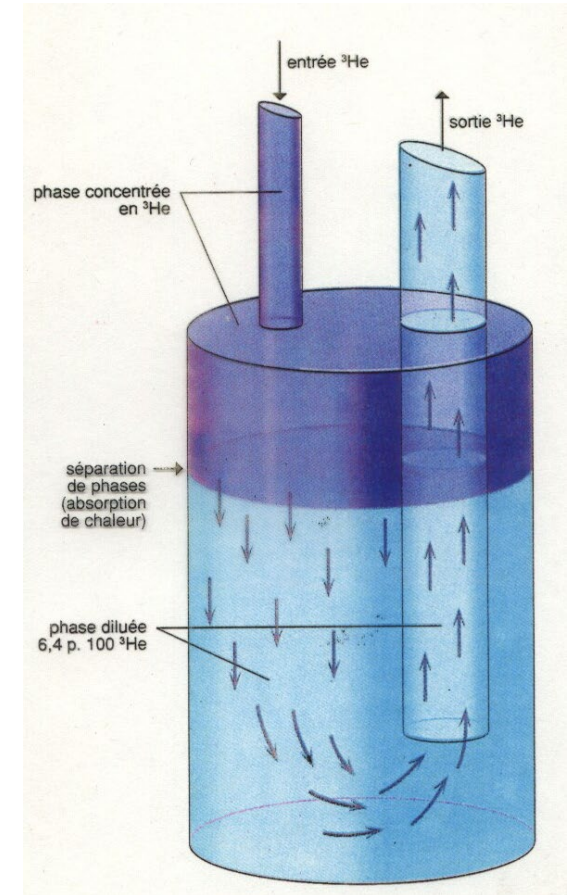
If  $\dot{Q}_m = 0$ . Using Eqs.(30) gives the famous relation

$$T_M = \frac{1}{\sqrt{8}} T_i \approx \frac{T_i}{2.8} \quad (31)$$

which states that there is a fixed ratio between the mixing chamber temperature and the temperature of the incoming fluid. This property shows the importance of the precooling of the incoming  $^3\text{He}$ . In this case  $T_M \neq T_i$  so the mixing process is not reversible and Eq.(8) does not hold.

c: pure

s: saturated



Suggested reading:

<https://courses.physics.illinois.edu/phys523/fa2025/CryogenicsFiles/MinifridgeJaakkoKoivuniemi1998.pdf>

# Homework - due 12/2

In the dipstick DR, we will condense about 2 liters of STP  $^3\text{He}$  gas into liquid. The pump will allow a circulation rate of 35 micro-mole/sec.

Estimate the following:

1. The amount of  $^3\text{He}$  in moles.
2. The volume of liquid  $^3\text{He}$  after being condensed in the mixing chamber.
3. The cooling power of the mixing chamber at 100 mK. The cooling power depends on the incoming temperature of  $^3\text{He}$  entering the mixing chamber.

Estimate the cooling power of the MC at 100 mK for the following incoming  $^3\text{He}$  temperature:

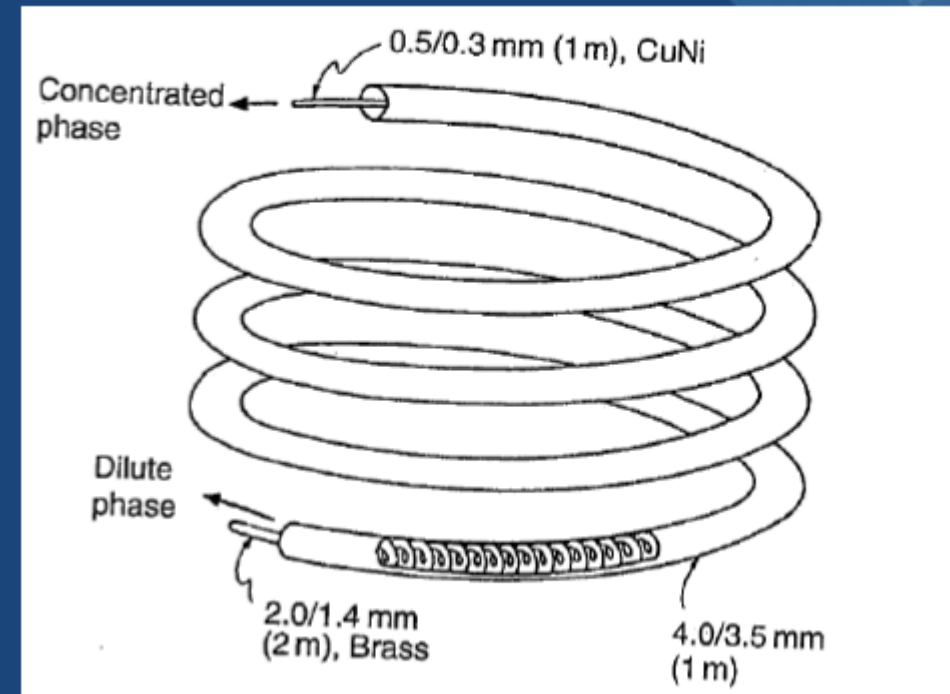
Ti	500 mK	400 mK	300 mK	200 mK
Cooling power (micro-W)				

4. How cold does the incoming  $^3\text{He}$  has to be for the MC to reach 100 mK?

Supplemental slides - reference

# Heat exchanger design above 50 mK

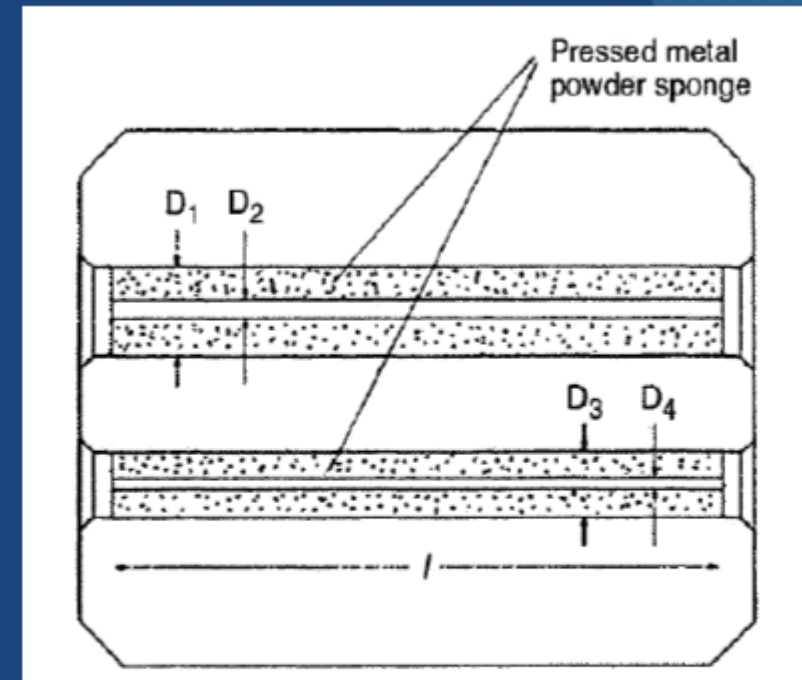
- At higher temperatures, continuous heat exchangers work well. These are typically of the concentric (or coil-in-tube) configuration.
- Heat is transferred from the dilute phase in the annular space and the concentrated phase in the coiled capillary.
- Heat transfer along the fluid and the capillary are negligible.



Pobell (2007)

# Heat exchanger design below 50 mK

- Designs using continuous heat exchangers only can reach  $\sim 30$  mK.
- For lower temperatures or very high flow rates, larger surface area heat exchangers are needed to overcome the increasing Kapitza resistance.
- Simple form is a sintered metal block to give **large surface area**, with a machined channel to **reduce flow impedance** (and hence viscous heating, see later).



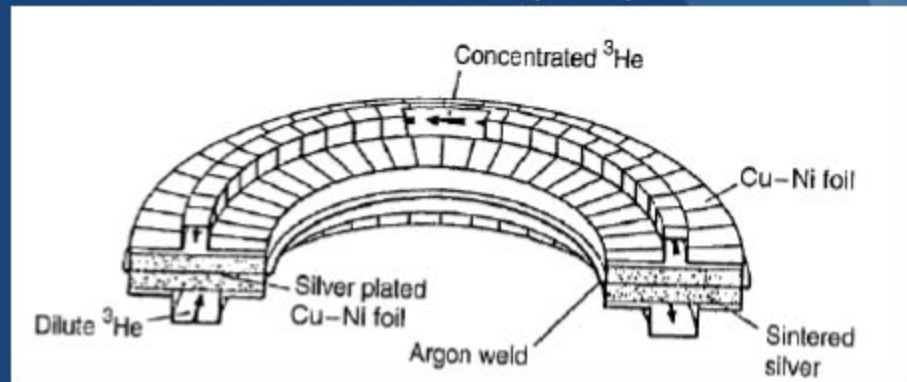
General discrete heat exchanger layout (from Pobell, 2007)



# Heat exchanger design below 50 mK

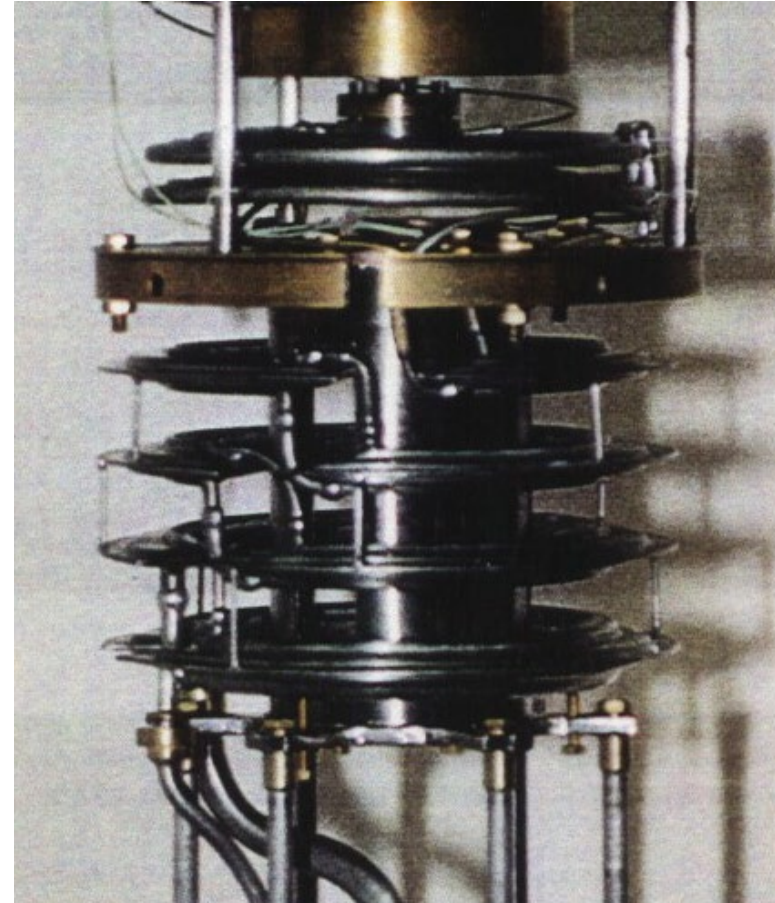
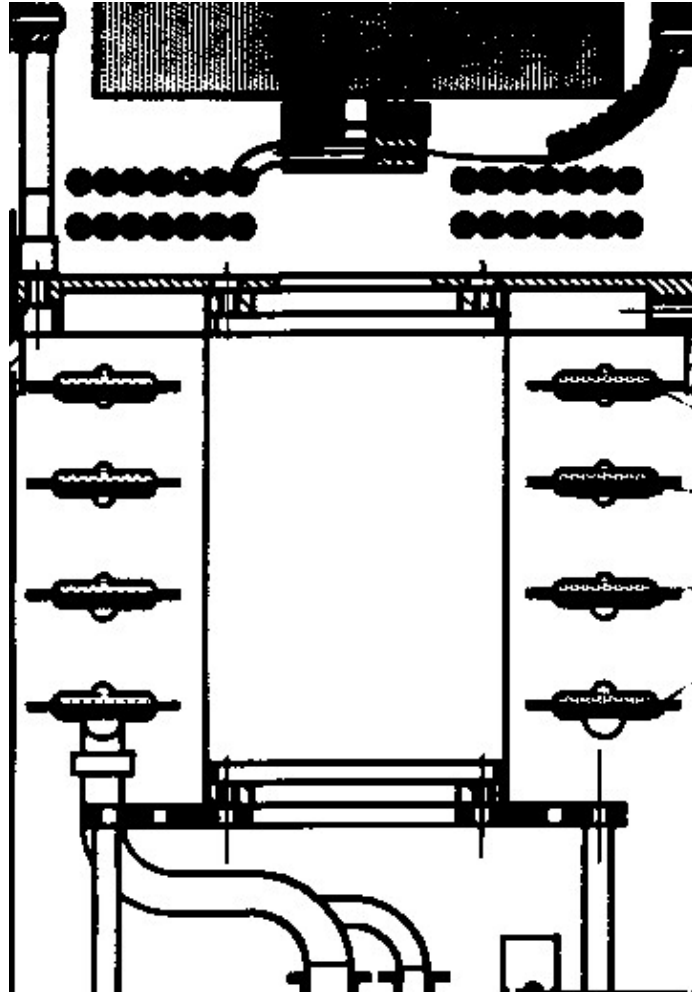
- To minimize axial heat flow, several discrete exchangers are connected in series – “step” heat exchangers at progressively lower temperature.
- Common design in commercial fridges similar to that shown right (from Pobell (2007), adapted from Frossati (1992)).

Pobell (2007)



Set of 5 step exchangers on a commercial dilution unit

# Heat exchangers & cold plate





# Sintered silver heat exchangers

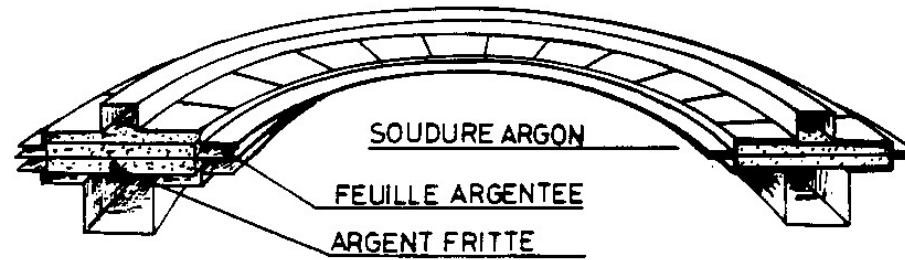


Fig. 20a

Coupe d'un échangeur continu utilisant de la poudre d'argent frittée

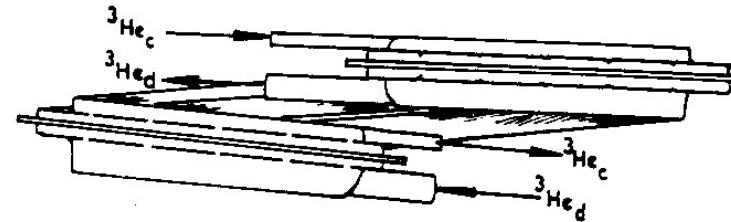
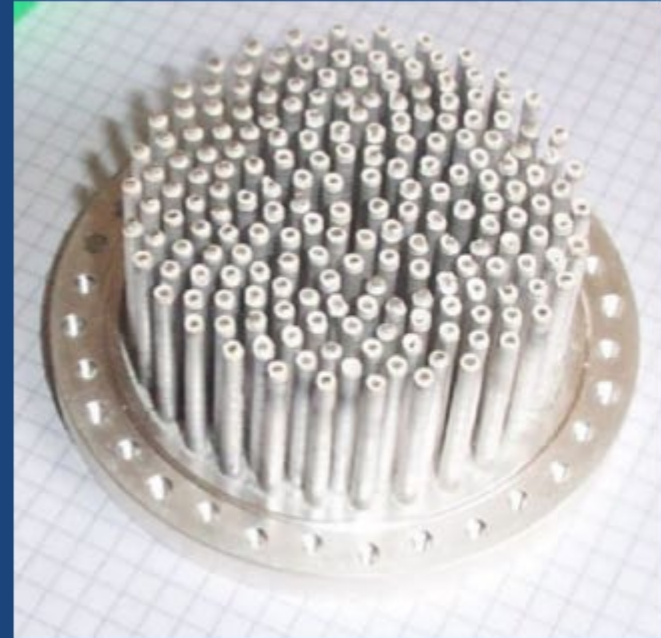


Fig. 20b

La forme spirale facilite la connexion avec les autres échangeurs et empêche la formation de points bas côté concentré, où la phase diluée pourrait s'accumuler

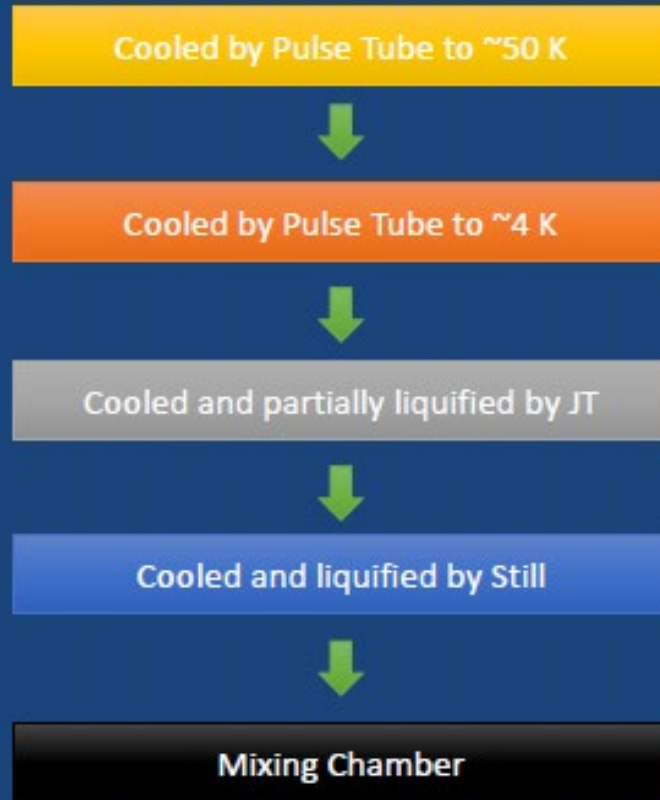
# Mixing chamber

- The mixing chamber is a relatively simple. Major requirement is for a large contact area between the helium phases and the metal.
- This is typically achieved through sintered structures, such as the silver sponge example shown right.
- In this design, the diameter of the mixing chamber is approximately 100 mm and each sponge has a surface area of 10 m<sup>2</sup>.



Mixing chamber construction  
example from Uhlig (2015)

# “Dry” vs “Wet” Refrigerators

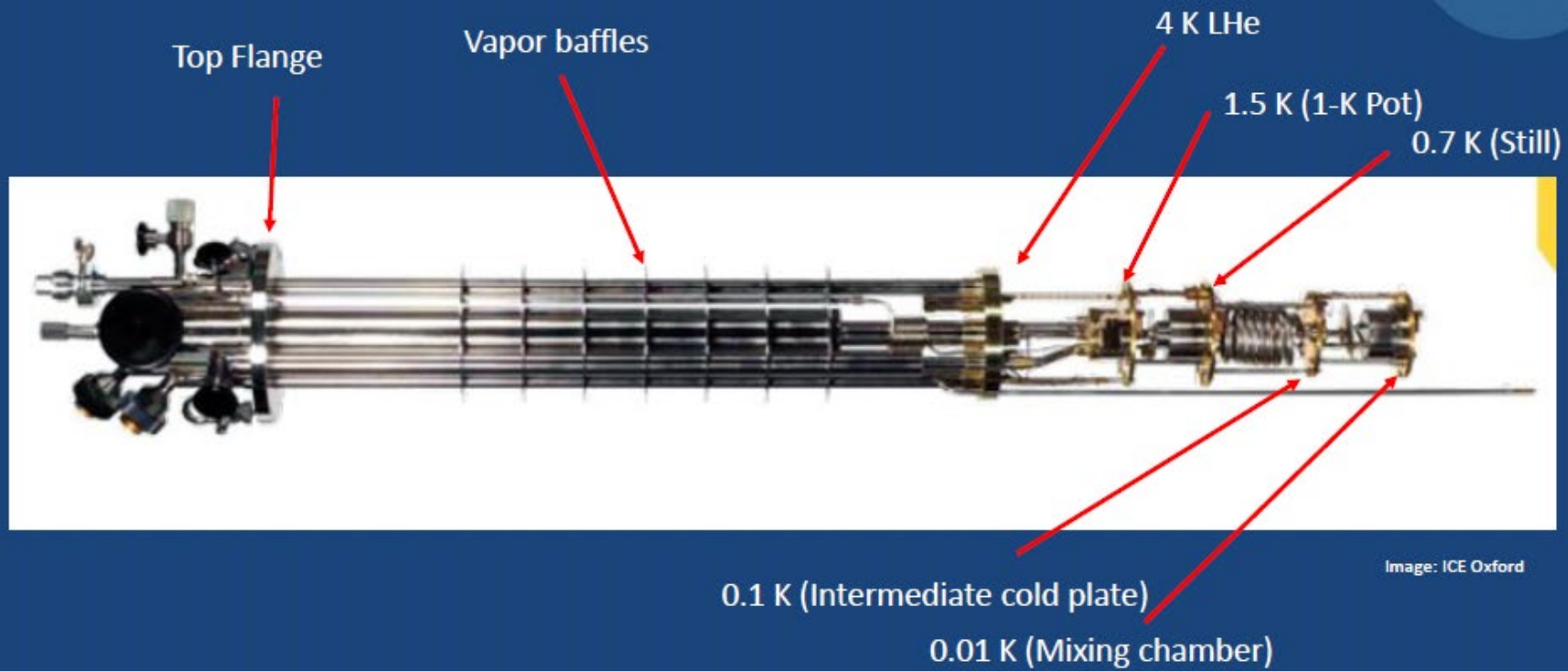


“Dry” Process



“Wet” Process

# “Wet” Layout





# "Cryogen Free" Refrigerator Layout

2 stage pulse tube cooler

Additional heat exchanger

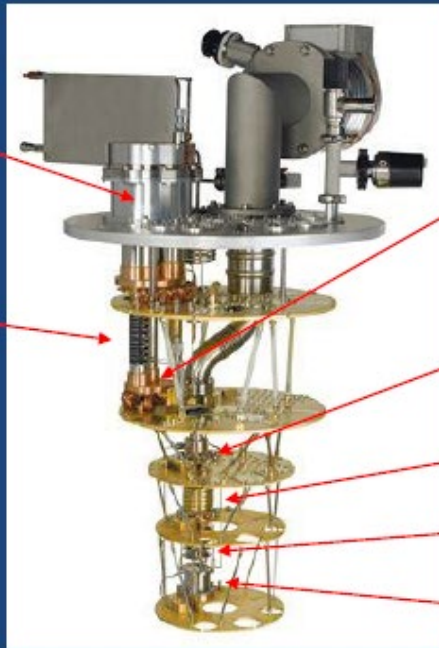
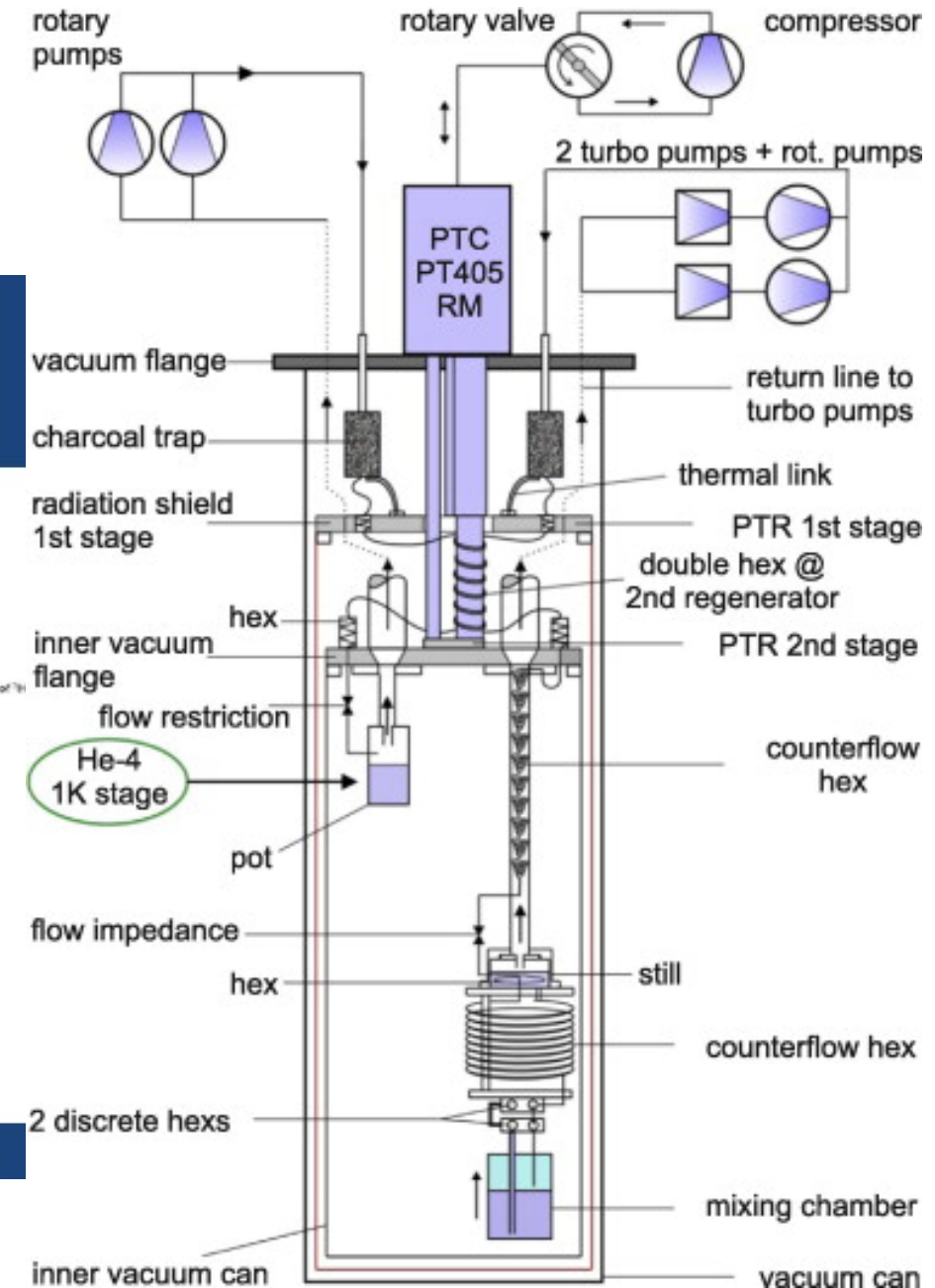
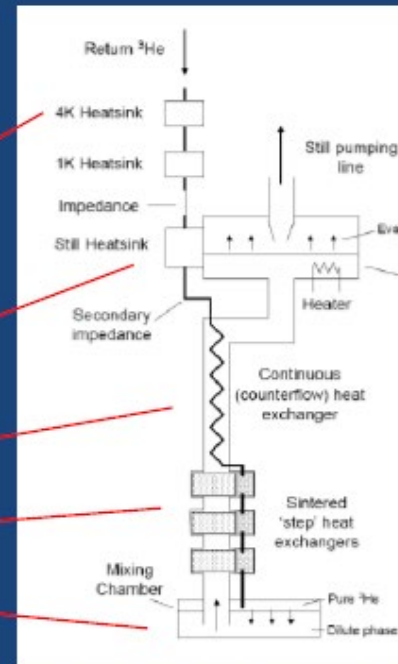
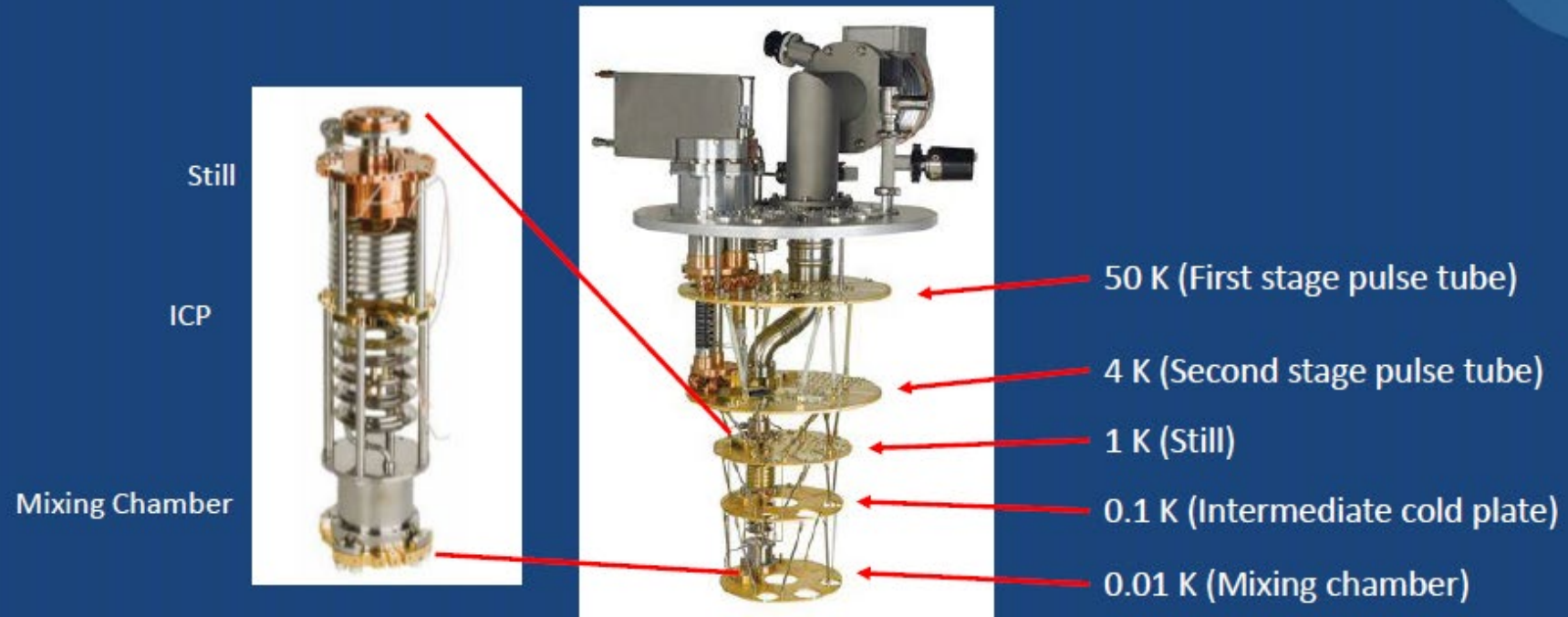


Image: Oxford Instruments



# Cryogen Free Layout – Temperature Stages



Images: Oxford Instruments

# Improvements to the precooler design

- An interesting property of the pulse tube cooler was measured in the early 2000s in that excess cooling capacity on the regenerator between the two stages exists.
- Some heat can be dissipated on the regenerator with little impact on the 4 K cooling performance.
- Adding a coil to the regenerator provides a useful additional heat sink for the inflowing  $^3\text{He}$ , reducing the cooling requirements at the 4-K stage.



Additional heat exchanger (Uhlig, personal communication)

Well, you decided to have a DR...  
(or you had no choice!)

- There are many kinds of dilution refrigerators:

- Classical, Helium (and nitrogen?) bath: Ultralow temperatures, measurements with superconducting coils, special samples...). Several manufacturers: Air Liquide, Cryoconcept, Leiden Cryogenics, Oxford Instruments, etc.
- With partial cryogenic bath and shields (DRILL, Sionludi) (neutrons, X-rays, etc...) "Table cryostat" Air Liquide, Oxford Instruments,...
- Pulse-tube Dilution refrigerators: Air Liquide, Leiden Cryogenics, BlueFors Cryogenics, Oxford Instruments, Cryoconcept, Vericold,...
- "top loading" : fast measurements with small samples, Oxford Instruments,...
- Inserts (Neumeier, Garching), ILL
- Horizontal (Niinikovskii, CERN)
- $^4\text{He}$  circulation (de Waele, Eindhoven; Schumacher, Grenoble)



# Some choices...

Precooling stage:

- With 1K pot?
- Joule-Thompson stage?

Pumping: a rotary pump and...?

- diffusion pump?
  - Roots?
  - Turbo-molecular pump?
  - Adsorption pump?

# Design considerations

**Obtaining low temperatures and high cooling power ( $dn/dt > 100 \mu\text{moles/sec}$ ):**

1) optimisation by a careful calculation:

- heat exchangers (sintered silver powder step exchangers).
- conduction and viscous heating in the low temperature parts.
- pressure drop in the pumping lines.
- condensation of the mixture

2) Optimisation of the mechanical parts:

- vibrations
- viscous heating

3) Empirical optimisation (know-how!):

- circulation of  $4\text{He}$
- interaction of the dilute  $3\text{He}$  on the excitations of  $4\text{He}$

4) Optimisation of the reliability:

- simple design
- modularity
- foresee leak testing by elements

5) Training the operators!

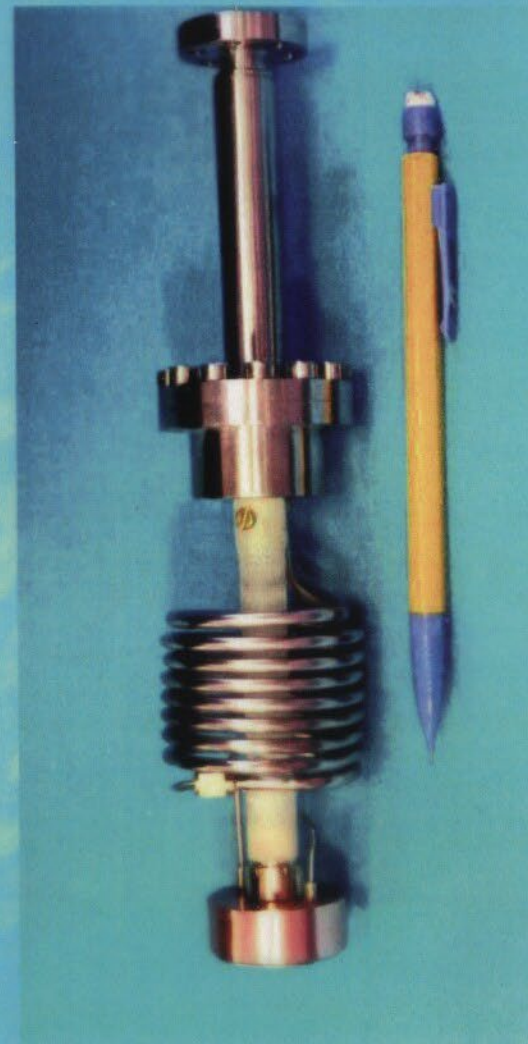
# Where do I put my DR now...???

- Check-list:

- Space available: area, height...
  - Door width (can the DR Dewar enter the room??)
  - Do I need to make holes in the floor or ceiling?
  - Dewar, pumping lines...
  - Room for the helium and nitrogen storage dewars?
  - Room for the pumps?
  - Electrical power? Water? Compressed air?
  - Helium recovery lines?
  - Anti-vibration system?
  - Shielded room?
- 
- Adapt the design to the needs.
  - Adapt the design to the constraints



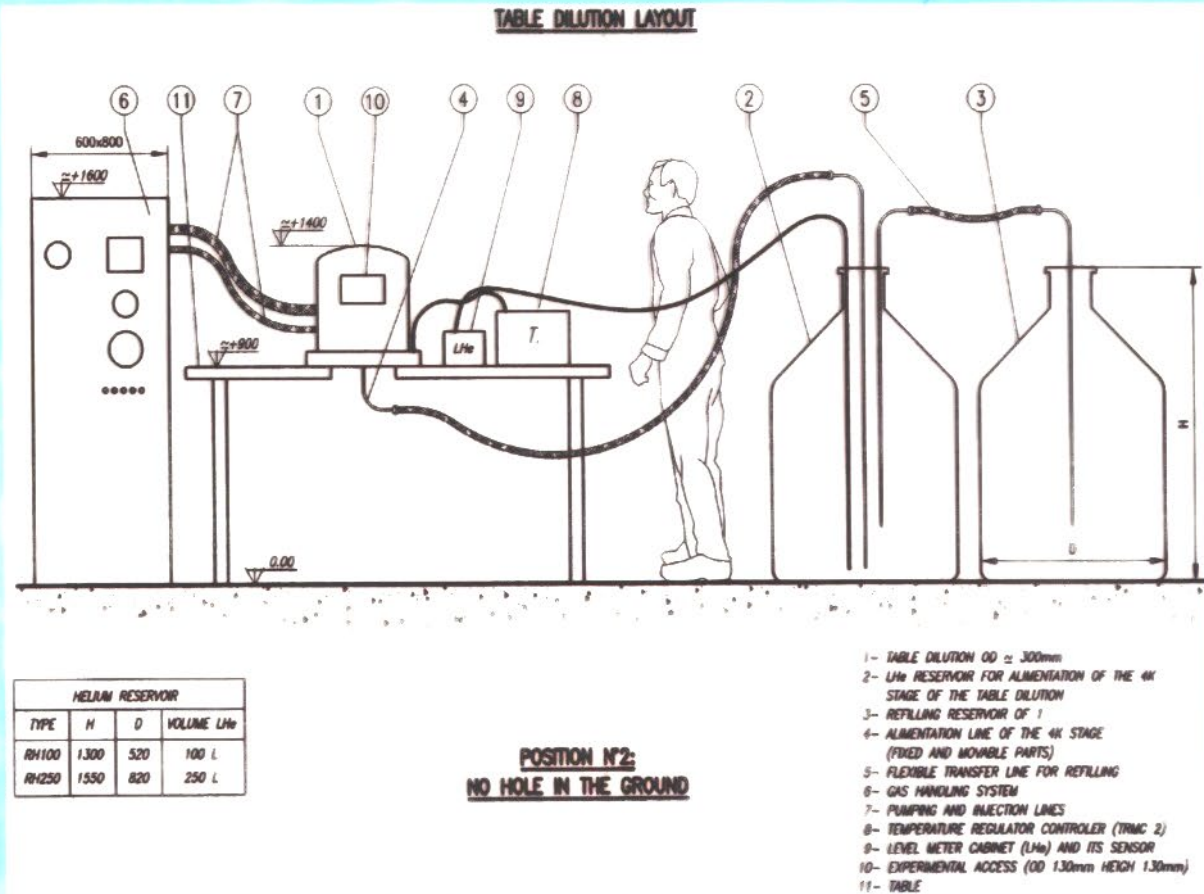
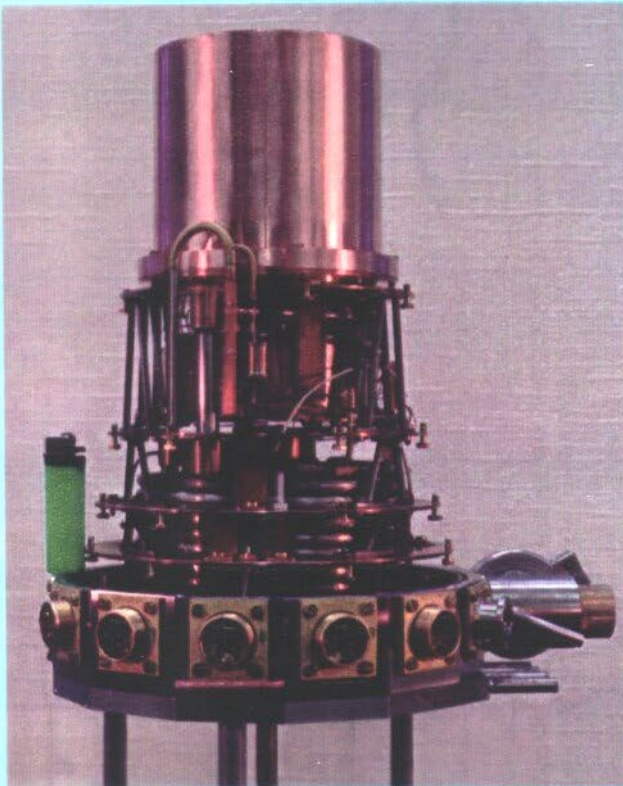
**Standard MINIDIL and detail of a lower part**





# DTA : Dilution Cryostats (Table Dilution)

2/3

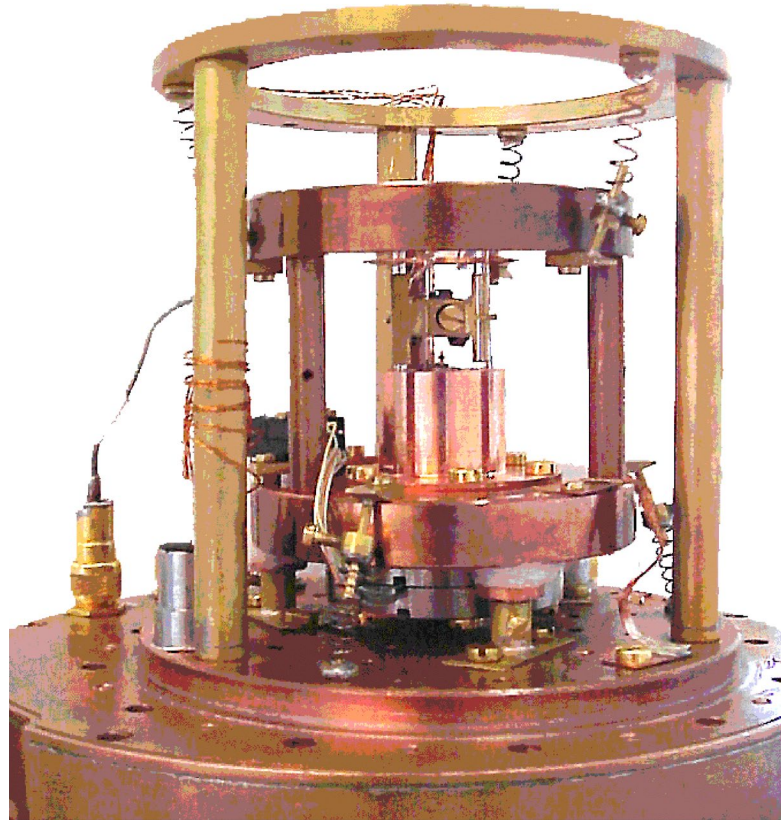


**TABLE DILUTION & Lay OUT of this NEW system**

# Table-top Dilution Refrigerator 50 mK

CNRS – Air Liquide

A. Benoit



STM, AFM, optical access...





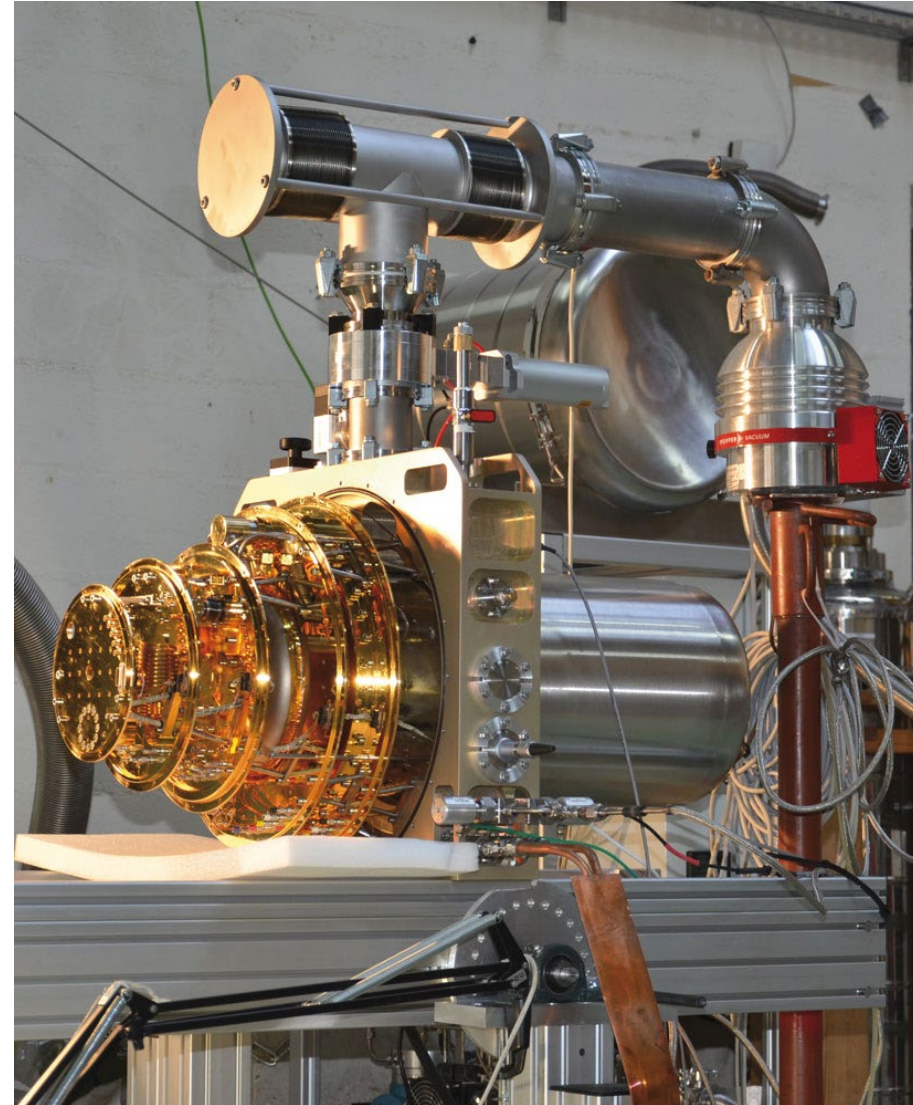
Base temperature < 30mK

Stability @ 30mK 0,1 mK

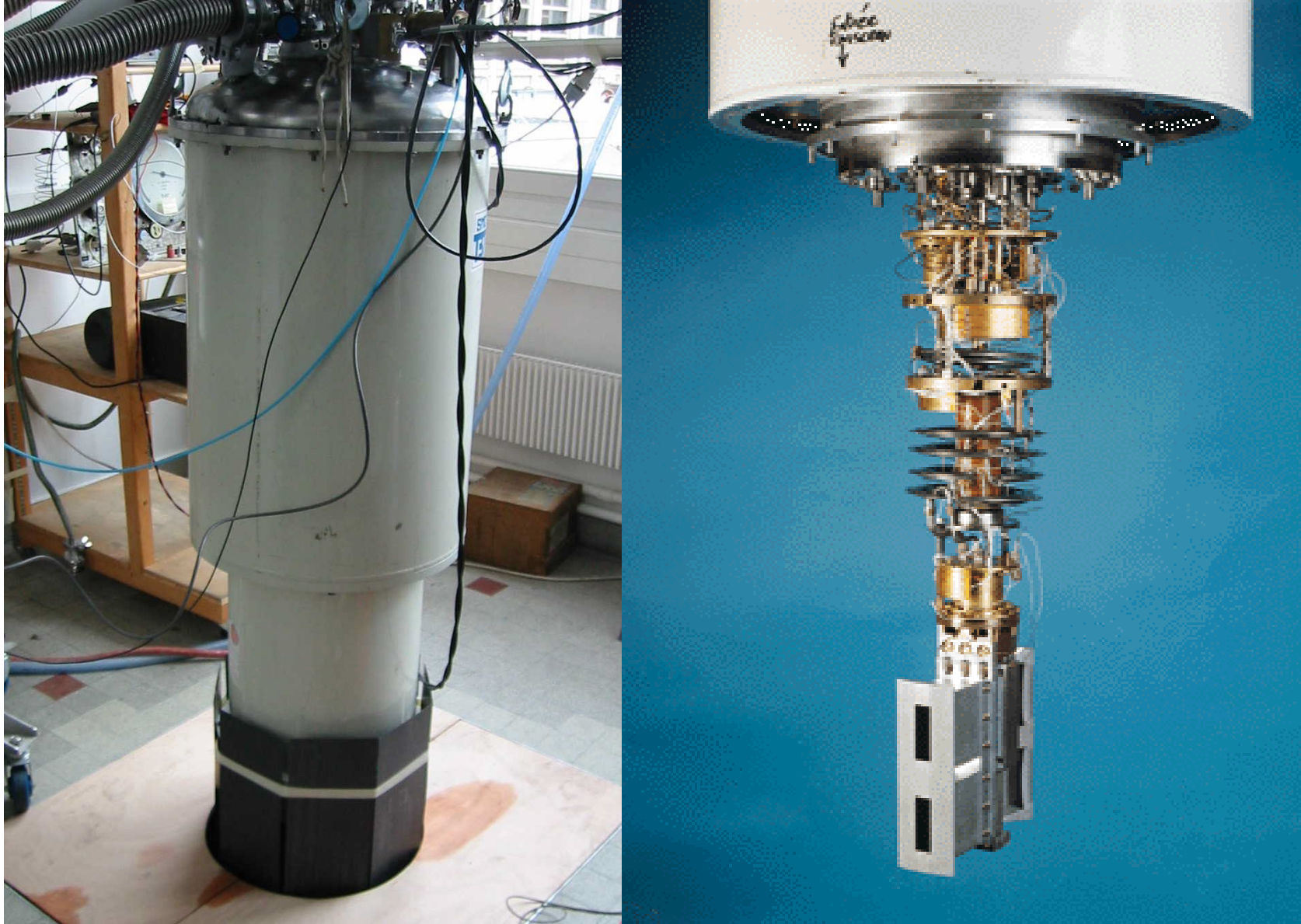
250 $\mu$ w @ 100mK

Less than 16 hours for 300K-30mK

Liquid helium hold time 48 hours  
(up to 10 days with automatic refill)

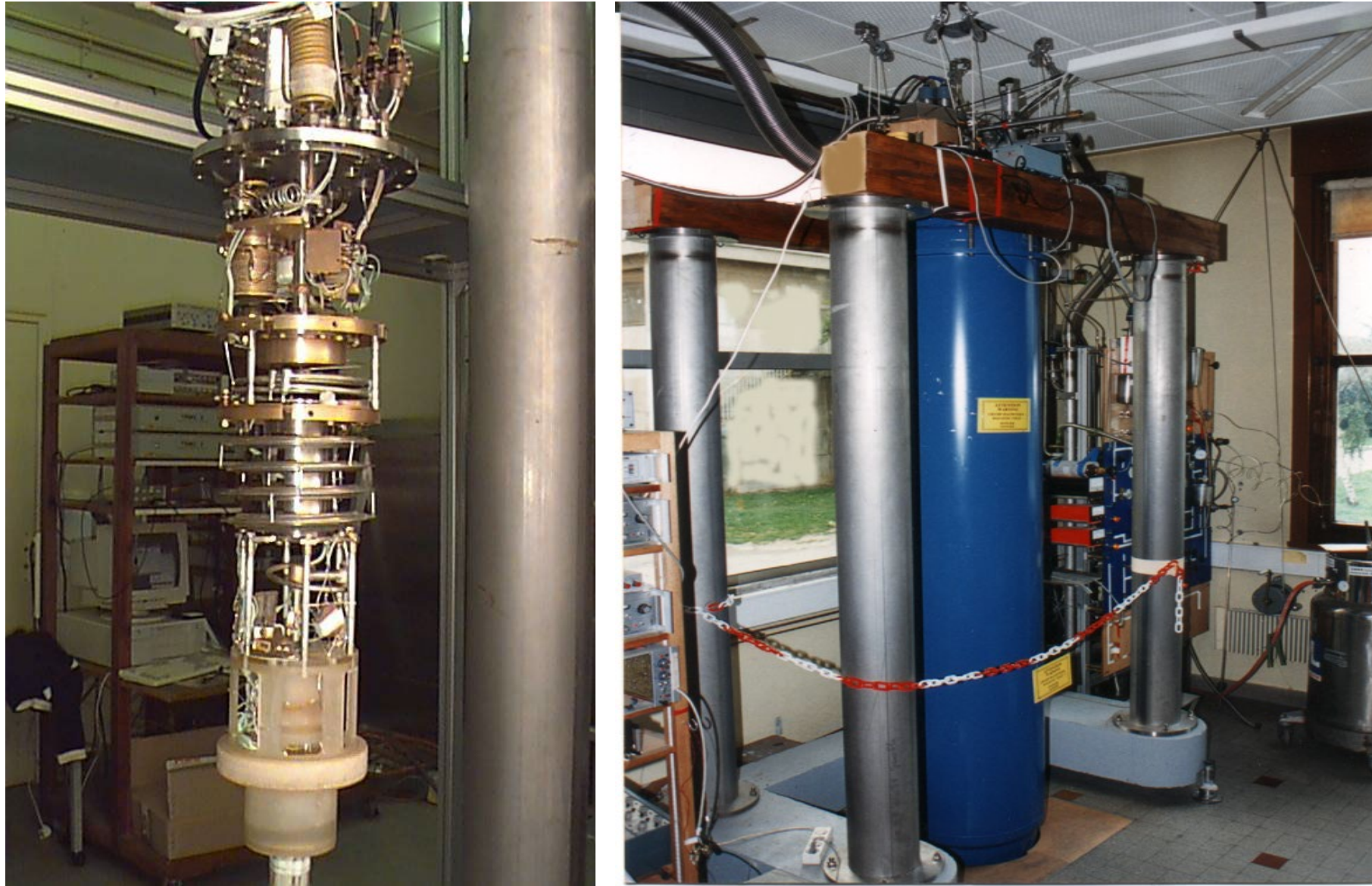


## DRILL – 5 mK DR for neutron scattering





# DN1: Dilution refrigerator for ultra-low temperatures

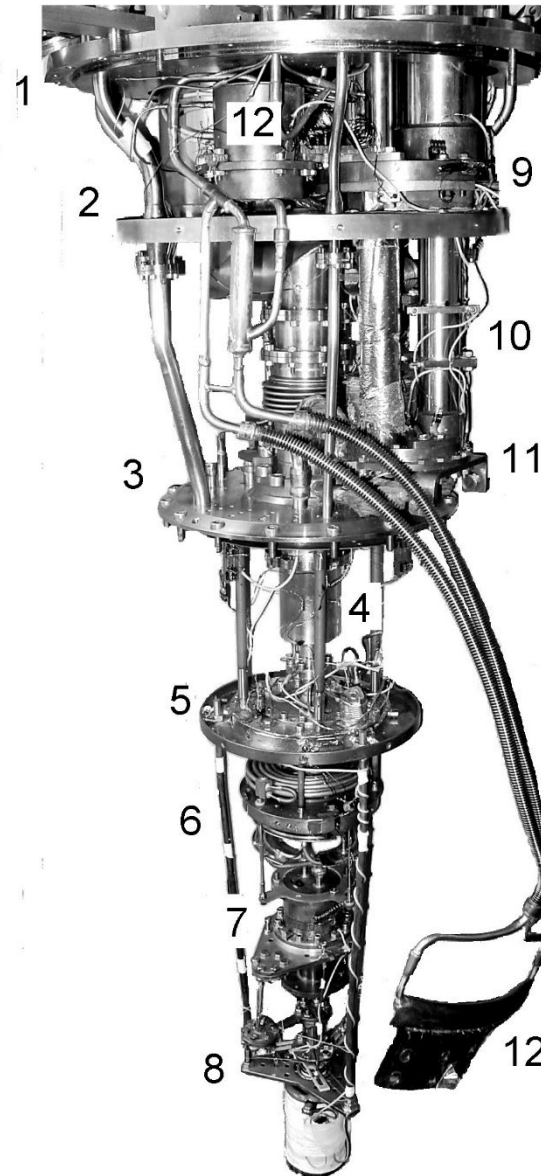


# PT-DR1 (1999) and PT-DR2

(Commercial unit delivered 2003)



H. Godfrin, S. Triqueneaux (CNRS & Air Liquide)



Th. Prouvé, S. Triqueneaux, H. Godfrin



# Pulse-Tube dilution refrigerator PT-DR2

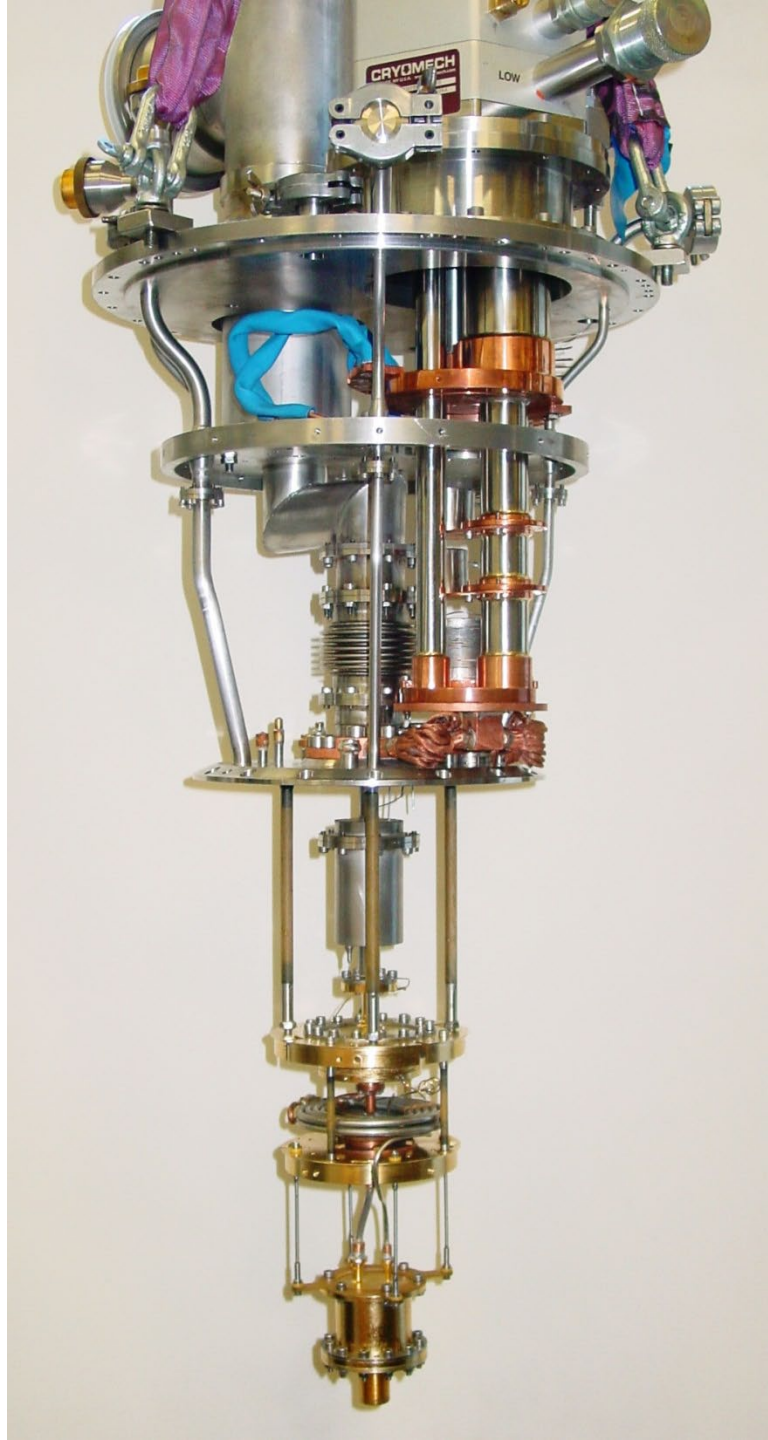
(industrial collaboration with l'Air Liquide)



Commercial unit

PT-DR3

CNRS - Air Liquide





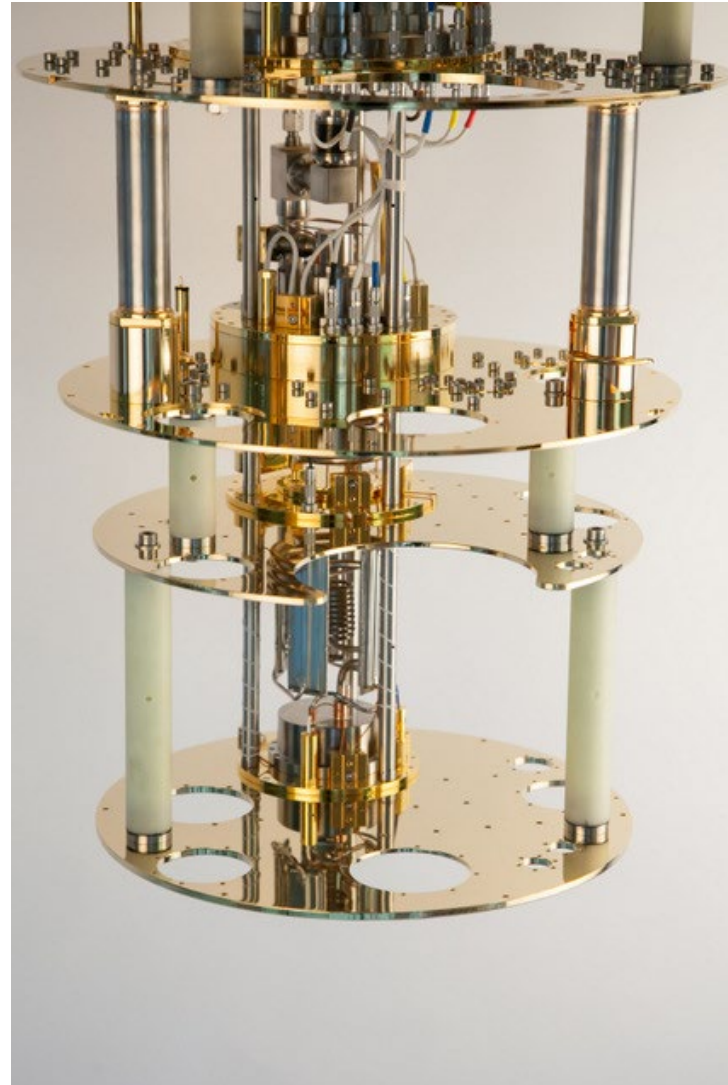
Base temperature < 10mK

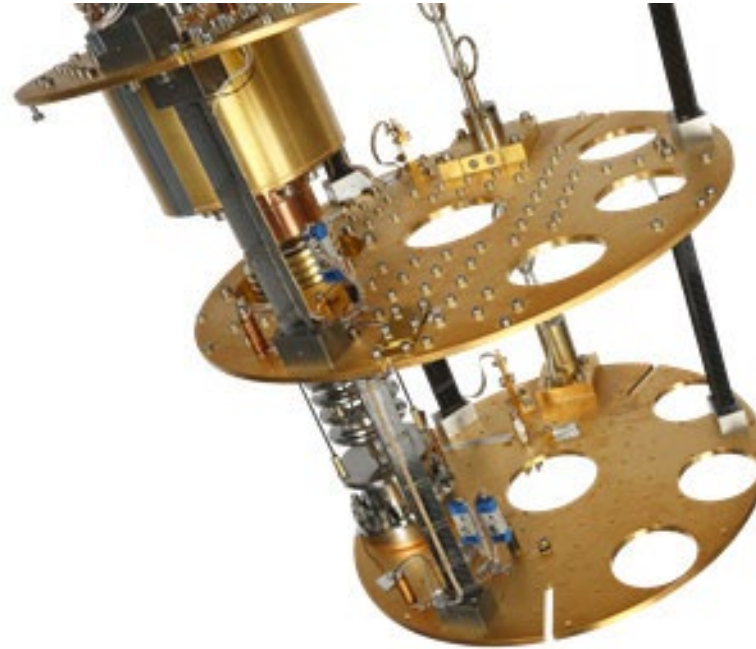
400 $\mu$ w @ 100mK

Stability @ 20mK 0,1 mK

10  $\mu$ w @ 20 mK

High temperature 30K





	With DU7-500 dilution unit	With DU7-300 dilution unit
<b>Base T:</b>	8 mK typical, < 10 mK guaranteed	8 mK typical, < 10 mK guaranteed
<b>At 100 mK:</b>	500 $\mu$ W typical, 450 $\mu$ W guaranteed	300 $\mu$ W typical, 250 $\mu$ W guaranteed
<b>At 20 mK:</b>	15 $\mu$ W typical, 12 $\mu$ W guaranteed	10 $\mu$ W typical, 8 $\mu$ W guaranteed
<b><math>^3\text{He}</math> requirement:</b>	18 litres STP	11 litres STP



# BlueFors



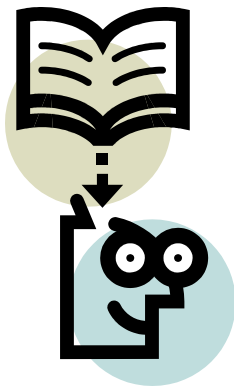
High Technology at Low Temperatures.





## Playing with the mixture...

- Calculate all the volumes of all circuits inside the RD, and also in pumps, traps, lines, etc...
- Calculate the amount of  $^3\text{He}$  and  $^4\text{He}$  gas needed
- Remember that the still has less than 1% concentration of  $^3\text{He}$ ...
- Mixing Chamber should be large for stability...
- Start with minimum amount of mixture!
- Best operation with small amount of mixture in the still, and small amount of concentrated phase in mixing chamber



# Condensation of the mixture

---

- Pre-cooling of cryostat at 4 K
- Pump the exchange gas (pump or adsorption)
- Check tanks pressure
- Check LN2 (eventually LHe) traps
- Circulate mixture through the traps.
- Long condensation process (hours)
- Condensation temperature must be low to avoid additional delays.
- Start circulation. With tricky cryostats, start with high flow rates (4He low points)

# Operating the RD...

---

- Flow rate is determined by the still power, not by the pumps!
- This is NOT true for PT-DRs (no 1K pot, large heat of condensation)
- Choose adequate flow rate (high power, or  $T_{\min}$  ?)
- With high circulation rates,  $^4\text{He}$  poisoning of the RD.
- High temperature (  $T > 1 \text{ K}$  ) operation: remove mixture, use only the mixture of the pump.

# Warm-up

---

- Removing the mixture takes a long time
- Heating the MC and Still: not very efficient!
- Exchange gas? Caution! Unstable, keep  $P < 10^{-4}$  mBar
- Close tanks, put rest of the mixture in the pump, close the pump.
- Traps: throw away (pumping) what was not pumped when the nitrogen trap was cold.
- Do not allow water condensation on RD...

# Troubleshooting

- 1 K pot hot, pressure is low. Filling capillary blocked. Remove LHe so that the bath level is below the intake, keep  $^4\text{He}$  pressure in Pot above bath pressure. Heater on capillary...
- High inlet pressure. Air? Hydrogen? Water?
- Low still pressure. Still empty? Check T vs. P!
- No cooling power. Interface level in MC? Heat leak to MC? Apply heat and check cooling power at higher temperatures.  $^3\text{He}/^4\text{He}$  ratio OK?
- Heating spikes, oscillations. Superfluid leak to Vacuum can?



# Good luck!

- Building a dilution refrigerator is NOT easy
- Repairing a dilution refrigerator is even worse.

We told you....!



## Pulse-tube $^3\text{He}/^4\text{He}$ Dilution refrigerators



# References

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