CRYO COURSE EUROPE

Cryocourse 2011

Grenoble & Chichilianne

Dilution refrigerators: design and operation

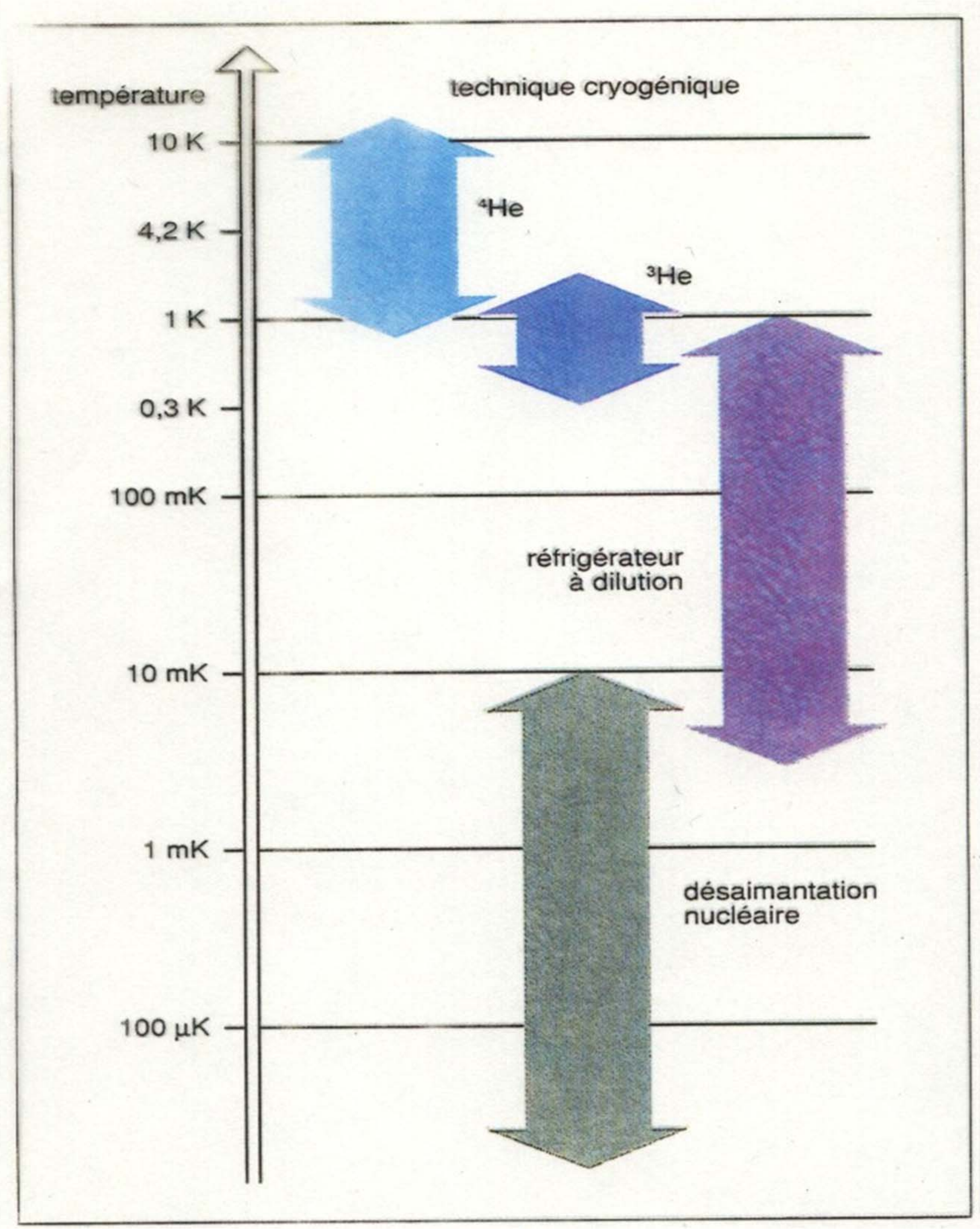
Henri GODFRIN

CNRS/IN/MCBT – Grenoble

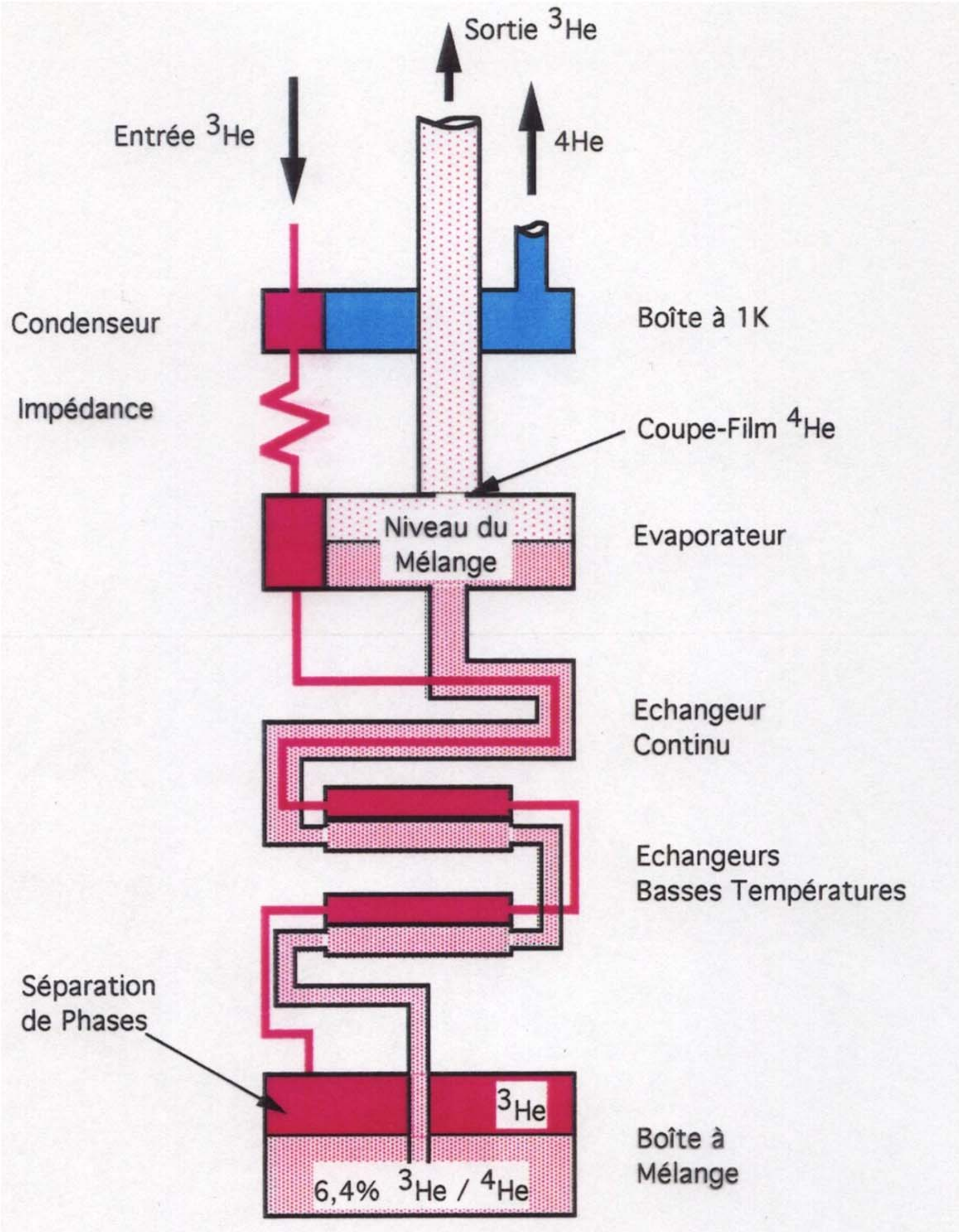
cnrs NÉEL institut UNIVERSITE JOSEPH FOURIER SCIENCES.TECHNOLOGIE.SANTÉ Grenoble INP

Refrigeration:

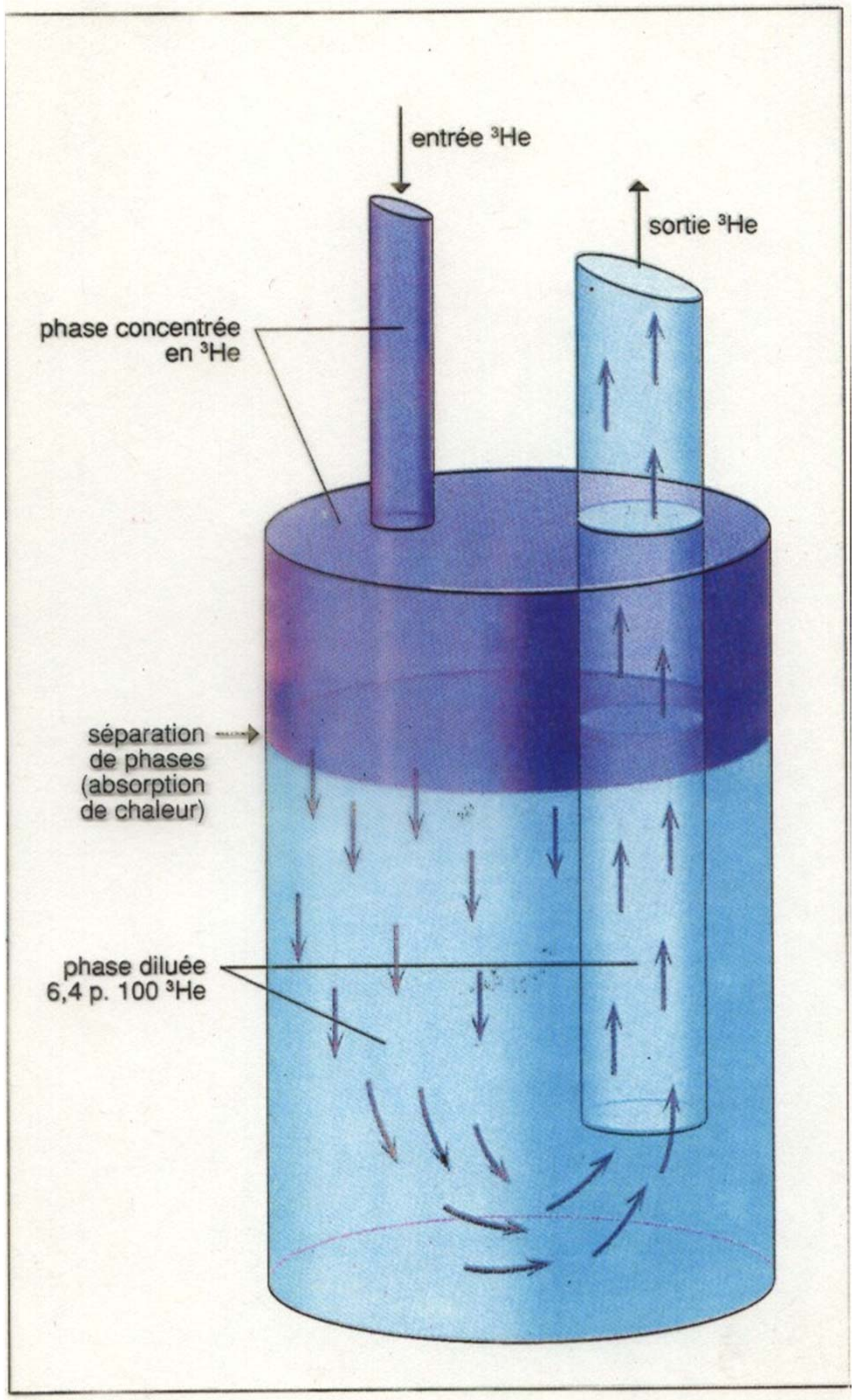
Temperature range



Schematic view of a DR



The mixing chamber



General considerations

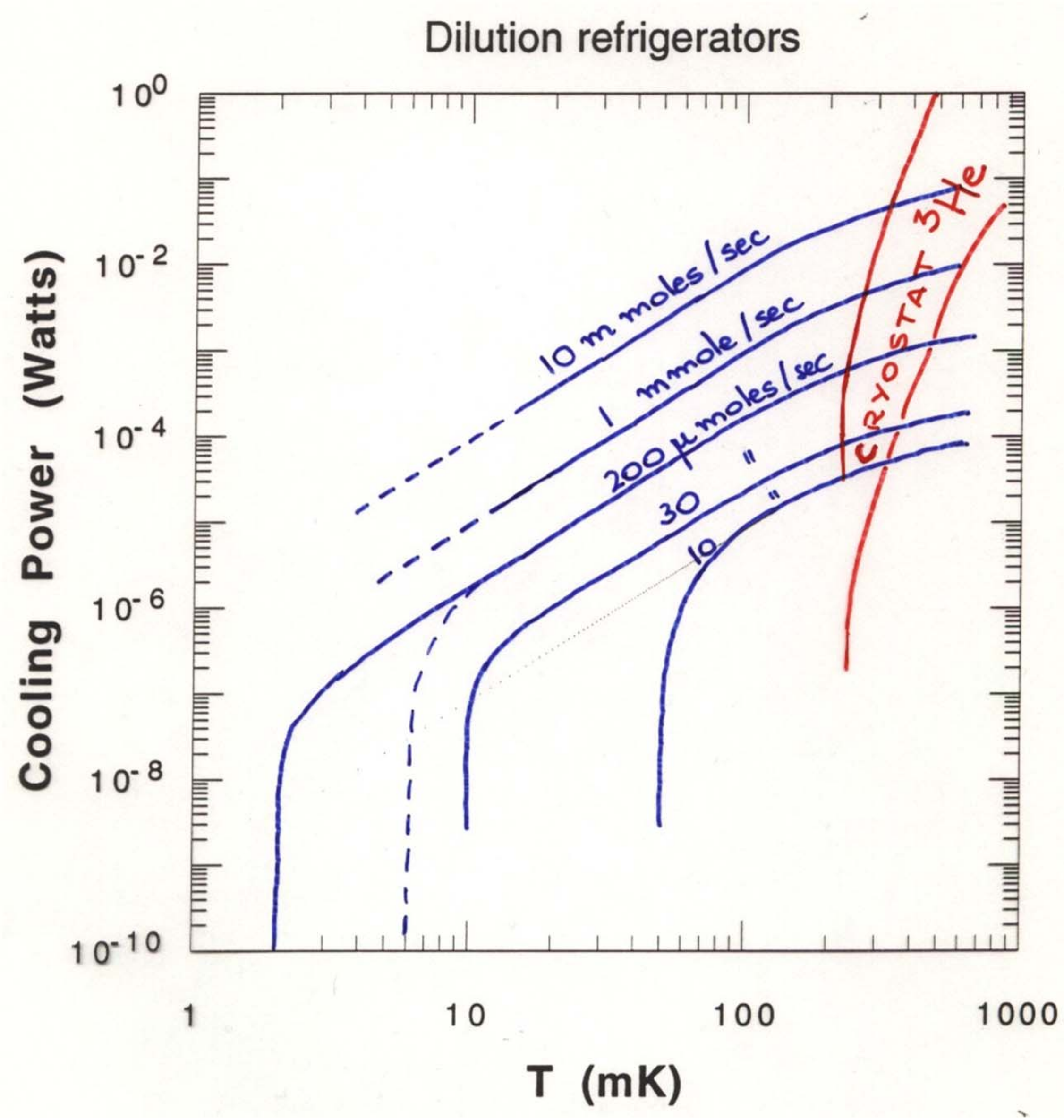
Dilution Refrigerators

- Minimum temperature T=2 mK in CONTINUOUS operation.
- Cooling power = 82 dn/dt T^2, for T>3 T_{min}
- Sophisticated machines! Do you really want to have a DR???

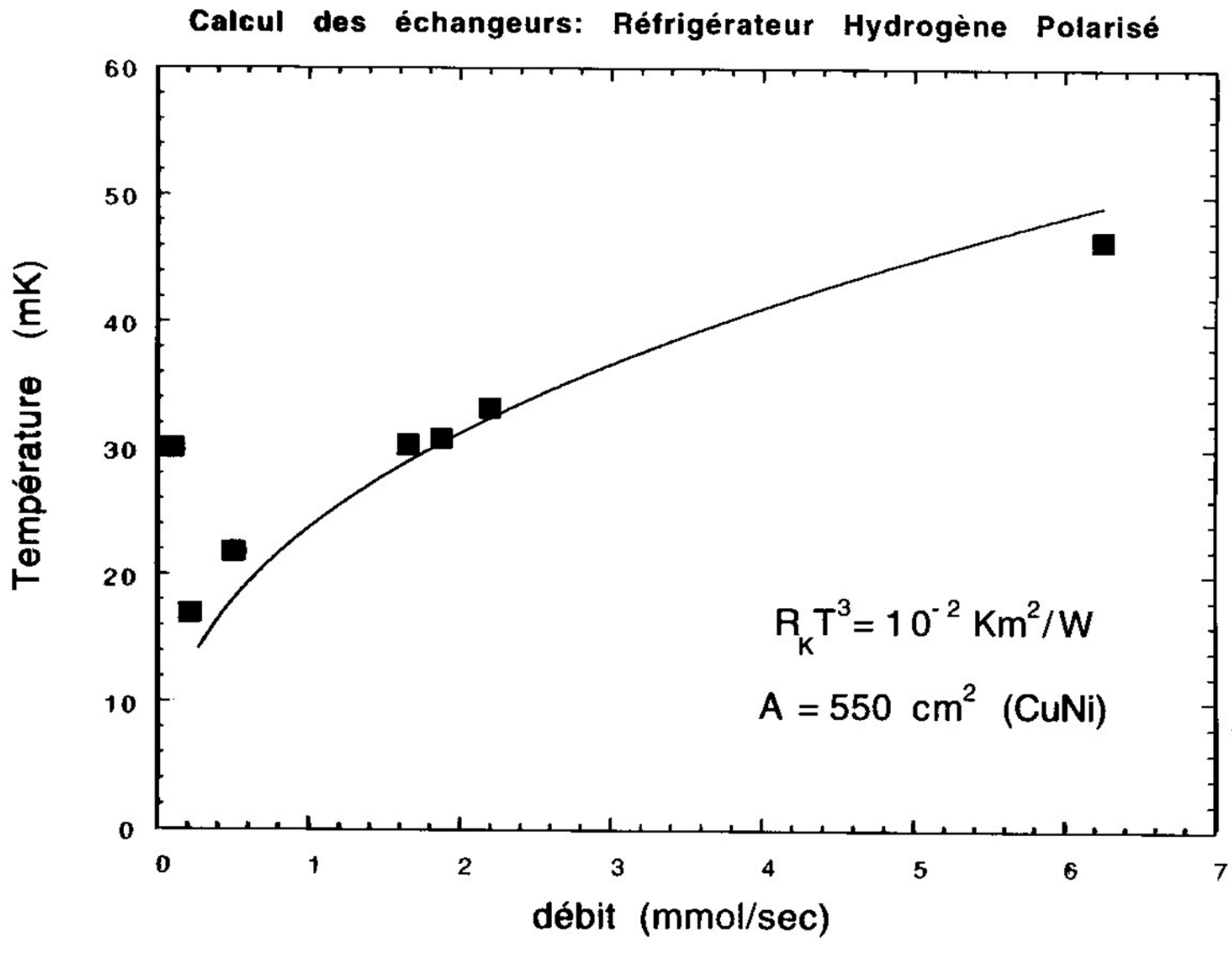
- Use a ^{3}He cryostat if you do NOT need temperatures below 0.3 K!

- Advantages of a ^{3}He cryostat :
  - Higher cooling power
  - Simplicity of operation
  - Gain in operation time
  - Reduced risk of failures
  - Troubleshooting is easier
  - Reduced initial and maintenance costs
  - Saves laboratory space

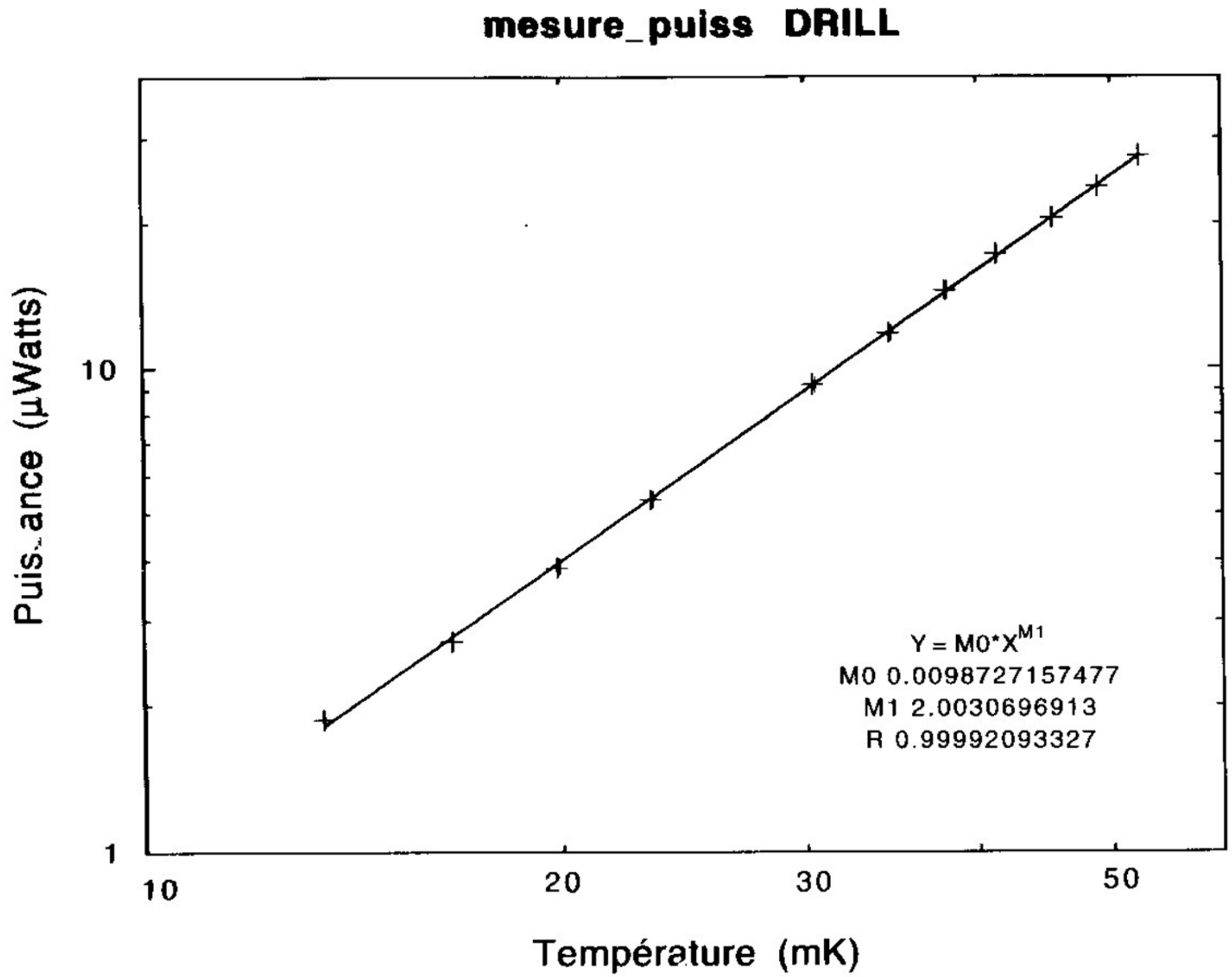
Cooling power



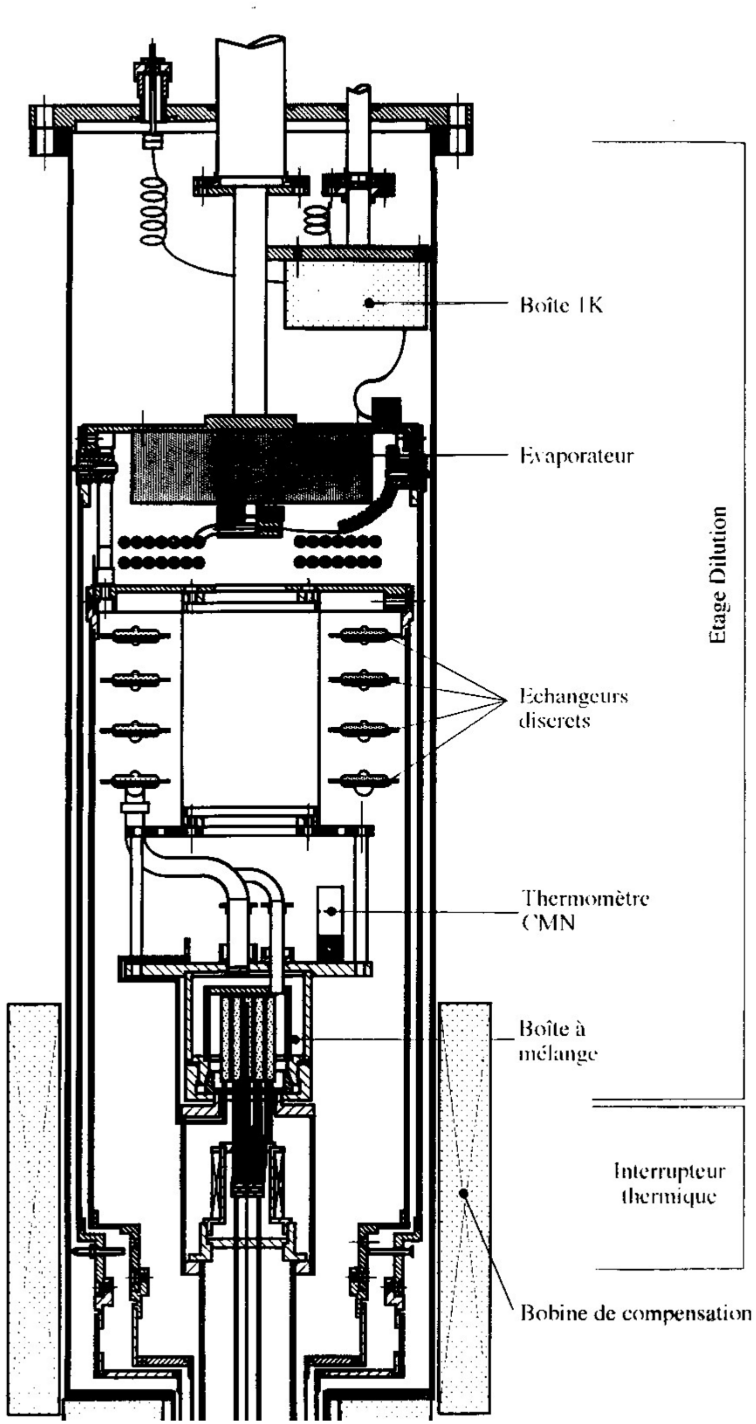
Cooling power of a DR as a function of the flow rate



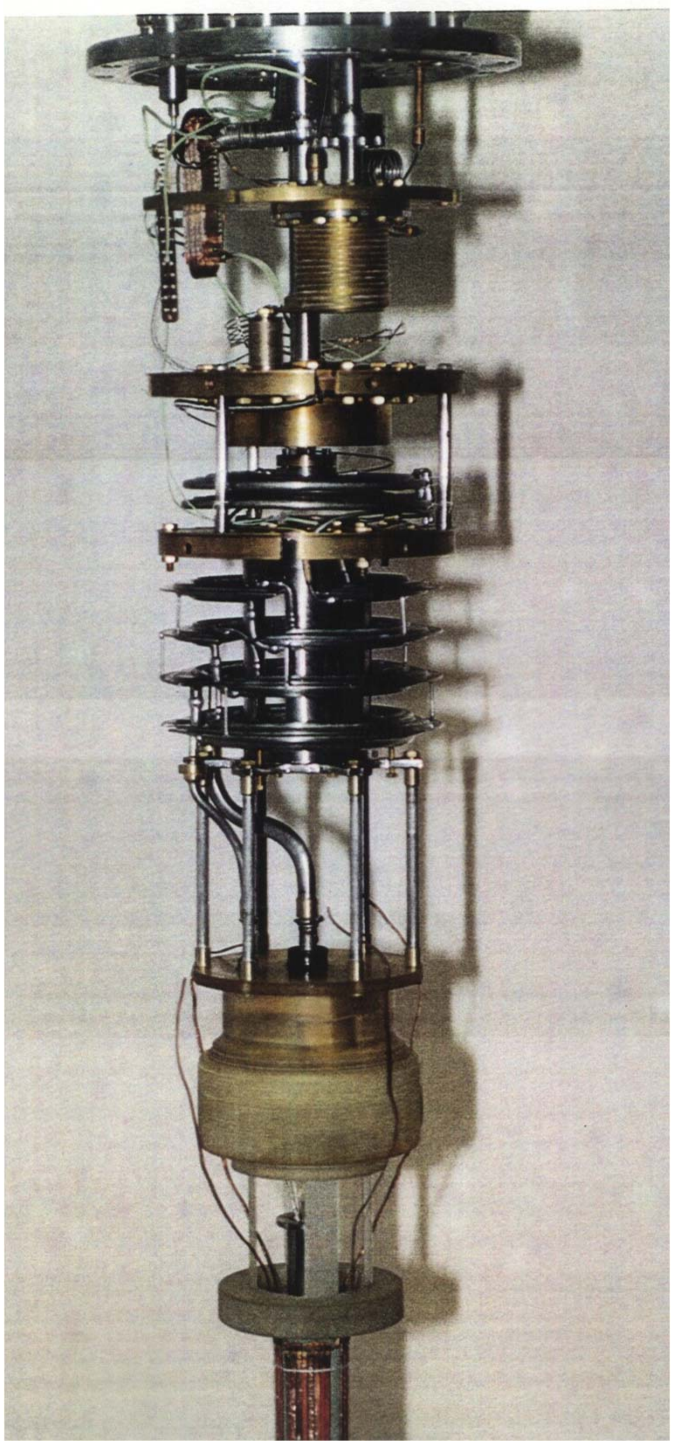
Cooling power as a function of temperature (Tmin off this DR = 5 mK)



The real drawing!



The real thing!



Sintered silver heat exchangers

Fig. 20a
Coupe d'un échangeur continu utilisant de la poudre d'argent frittée

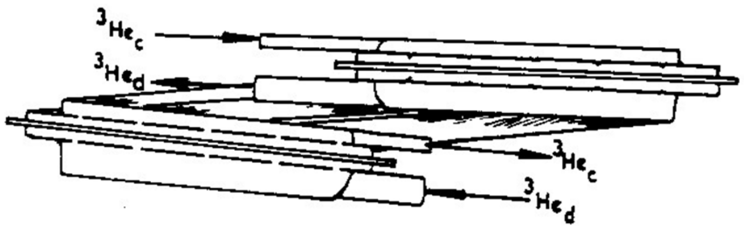
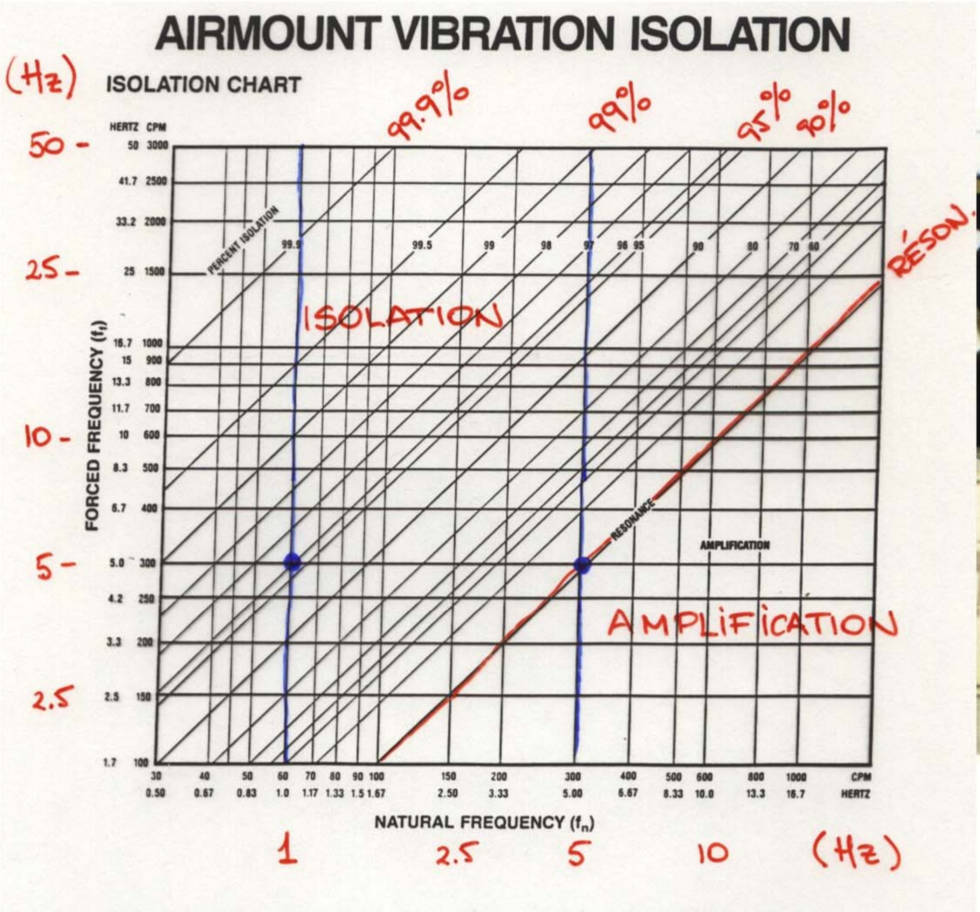
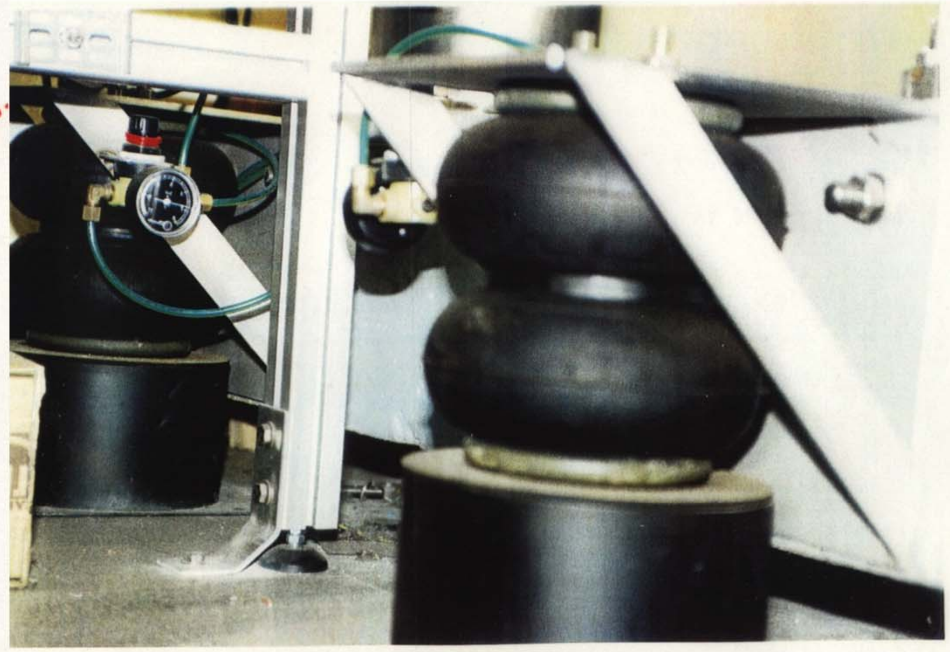


Fig. 20b
La forme spirale facilite la connection avec les autres échangeurs et empêche la formation de points bas côté concentré, où la phase diluée pourrait s'accumuler

Vibration isolation





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}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 µmoles/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 4He
   - interaction of the dilute 3He on the excitations of 4He

4) Optimisation of the reliability:
   - simple design
   - modularity
   - foresee leak testing by elements

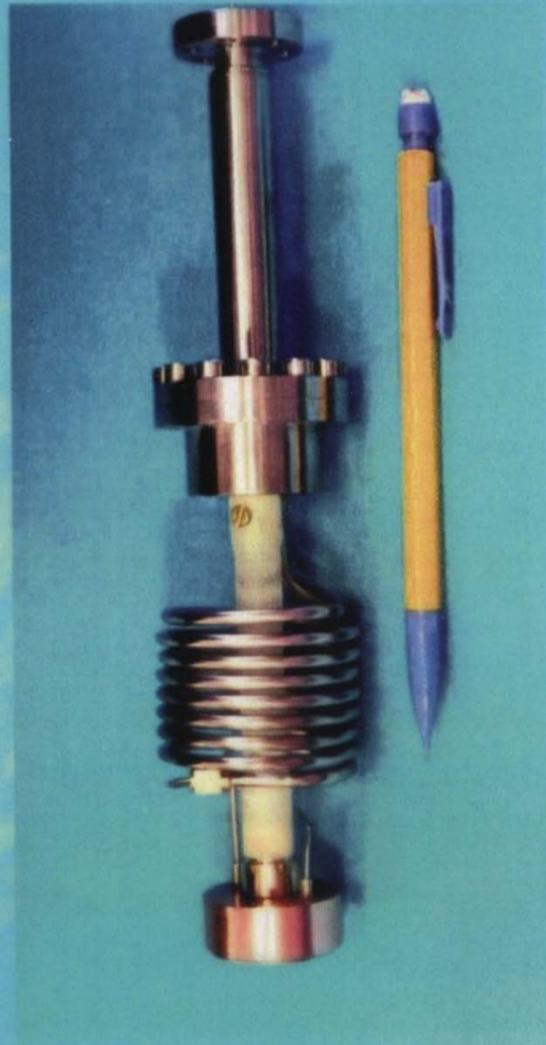
5) Training the operators!

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

DTA : Dilution Cryostats MINIDIL OD70 & OD45



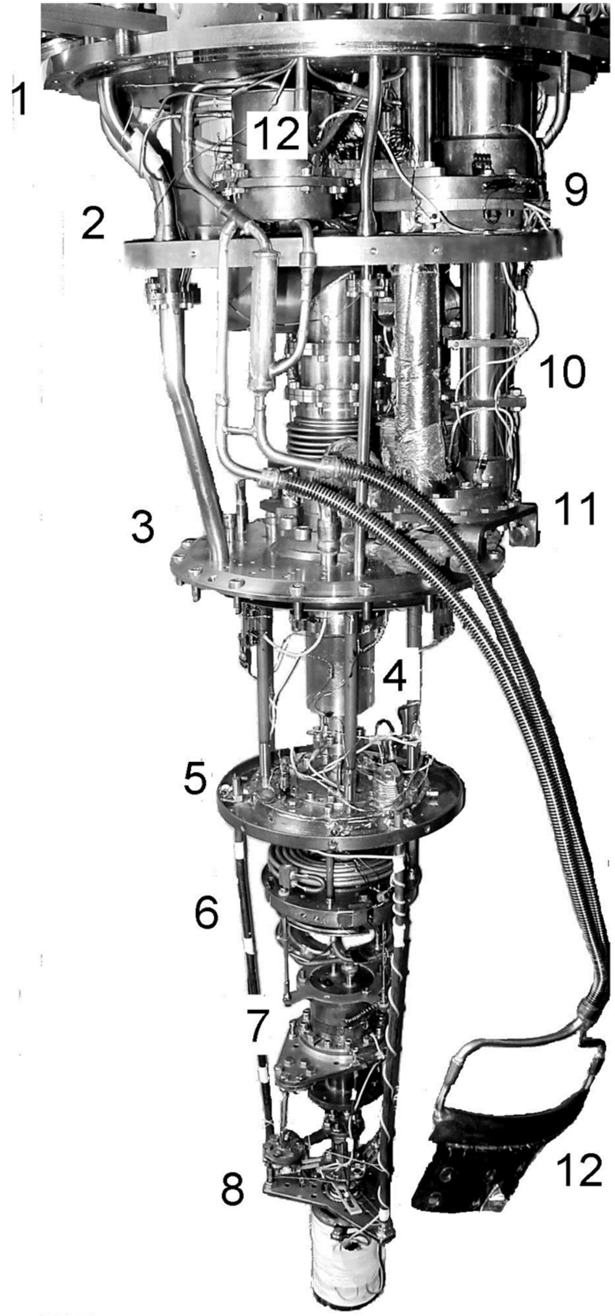
Standard MINIDIL and detail of a lower part

Pulse-tube $^3He/^4He$ Dilution refrigerators

Pulse-Tube dilution refrigerator PT-DR2

(industrial collaboration with l'Air Liquide)

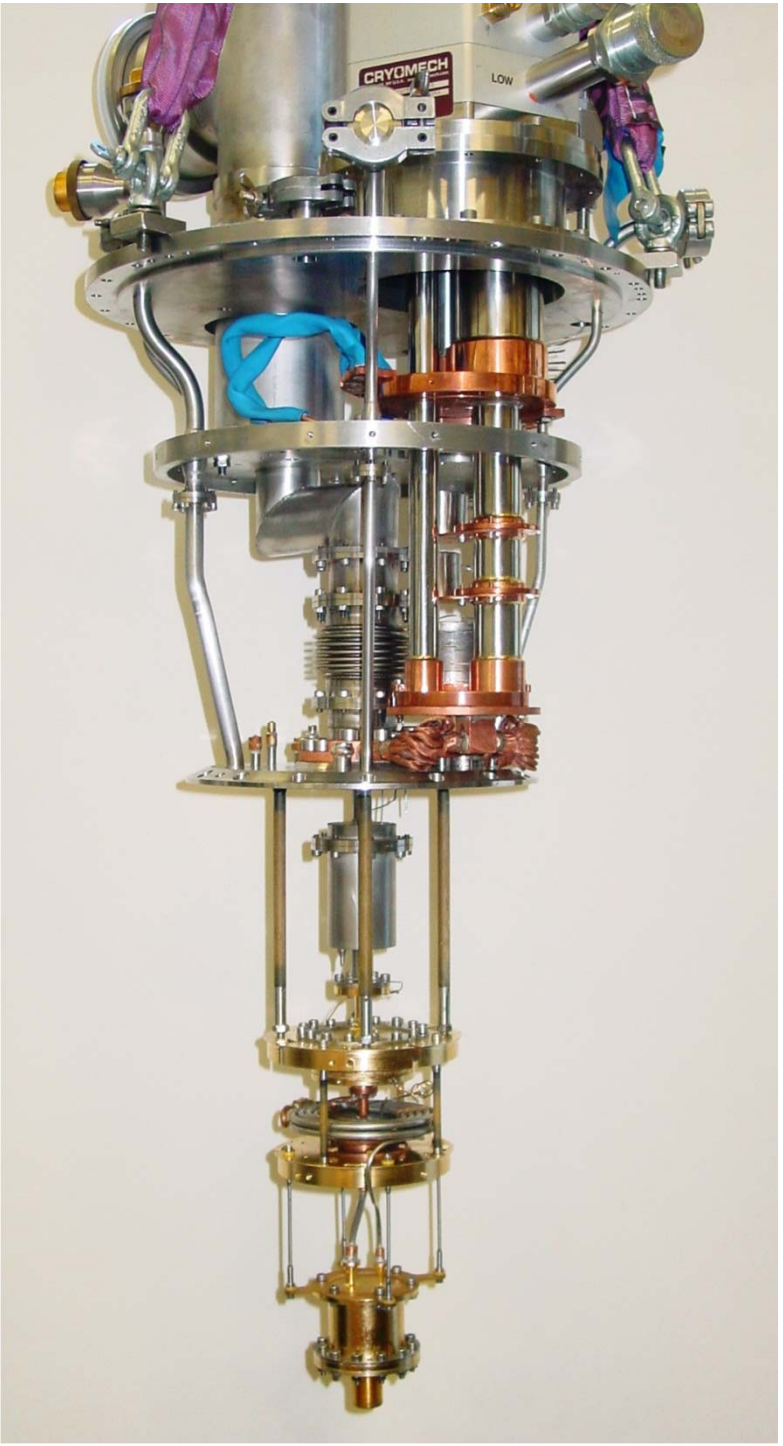
PT-DRs…



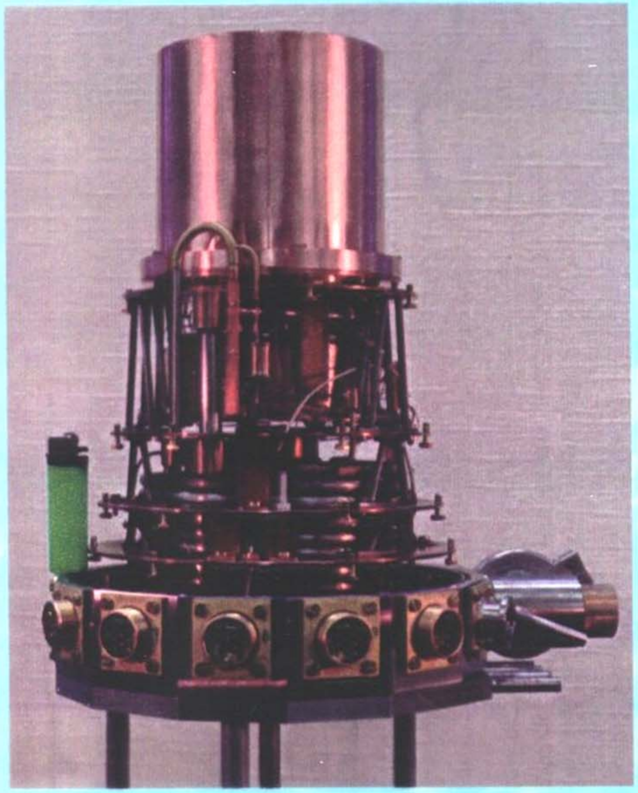
Commercial unit

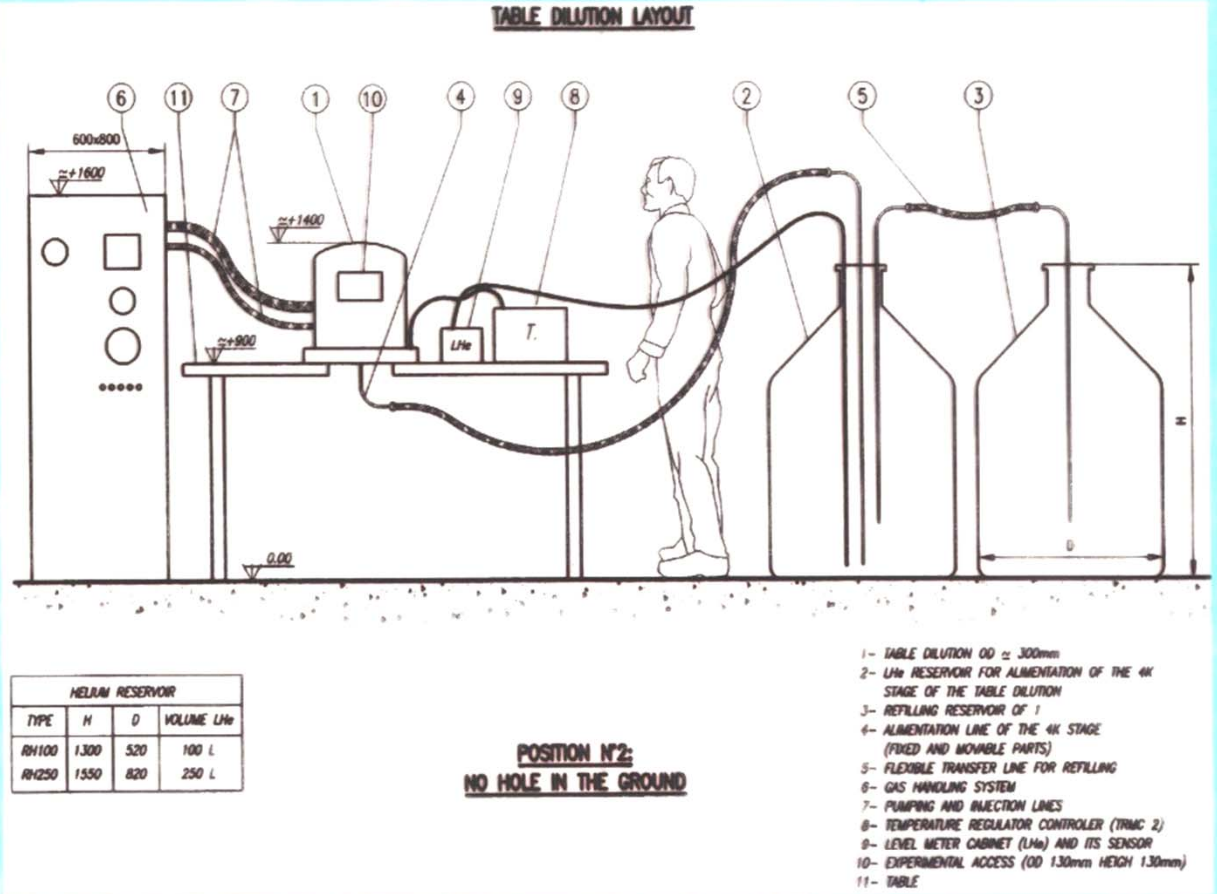
PT-DR3

CNRS - Air Liquide



DTA : Dilution Cryostats (Table Dilution) 2/3





HELIUM RESERVOIR

TYPE	H	D	VOLUME LHe
RH100	1300	520	100 L
RH250	1550	820	250 L

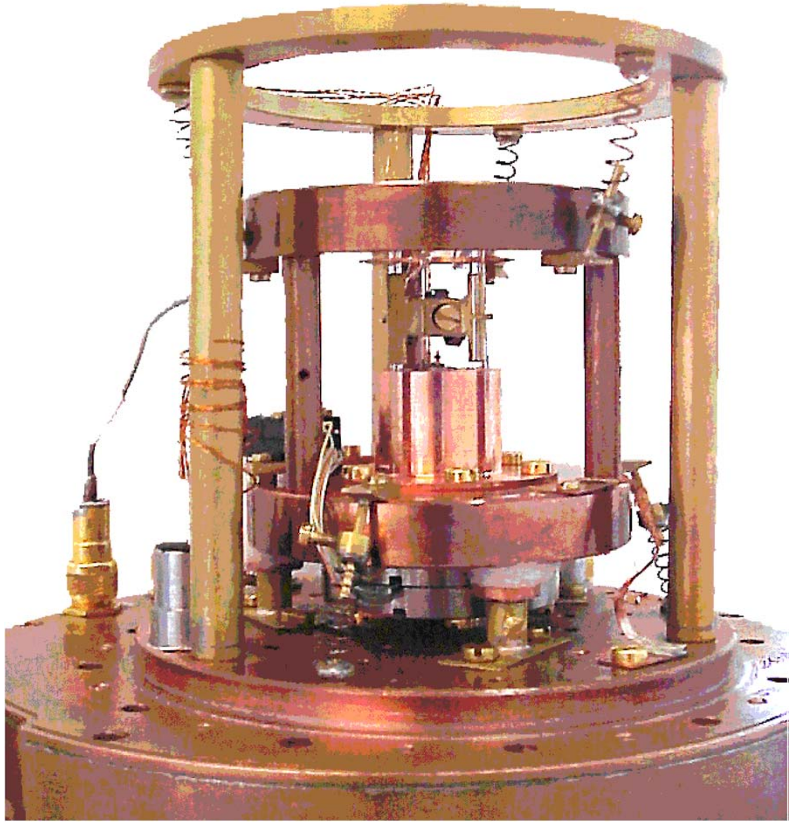
TABLE DILUTION & Lay OUT of this NEW system

AIR LIQUIDE

Table-top Dilution Refrigerator 50 mK

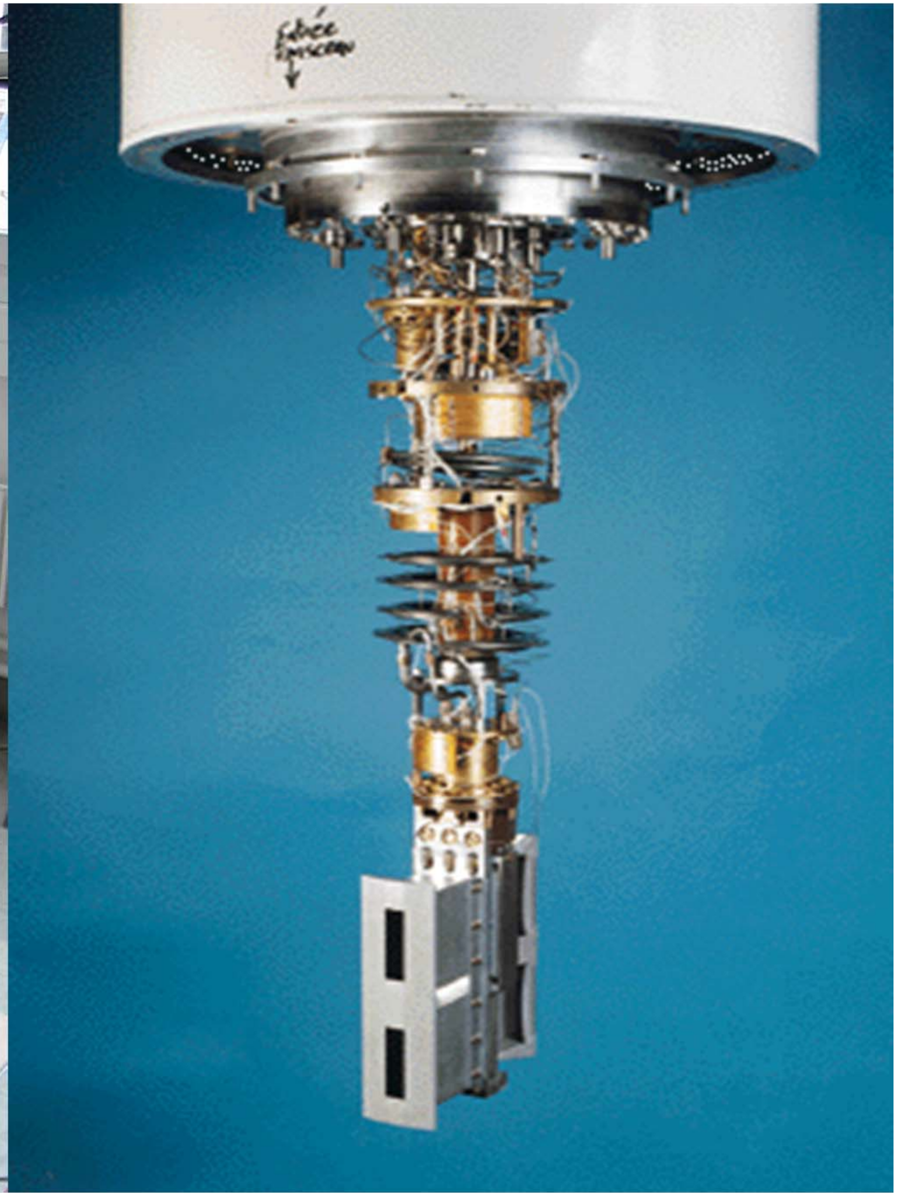
CNRS – Air Liquide

A. Benoit

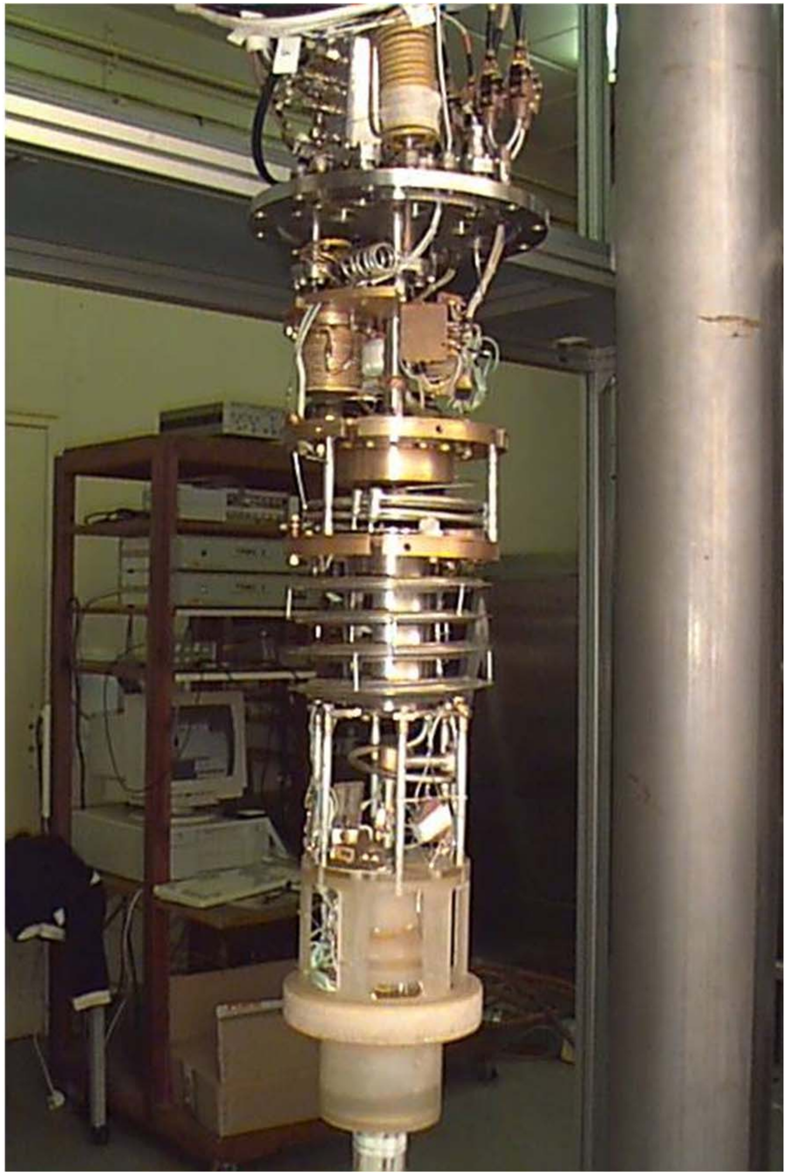


STM, AFM, optical access…

DRILL – 5 mK DR for neutron scattering



DN1: Dilution refrigerator for ultra-low temperatures



Playing with the mixture…

•Calculate all the volumes of all circuits inside the RD, and also in pumps, traps, lines, etc…

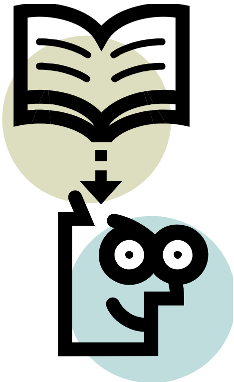
•Calculate the amount of 3He and 4He gas needed

•Remember that the still has less than 1% concentration of 3He…

•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}He poisoning of the RD.
- High temperature (T> 1 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 4He 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. $^3He/^4He$ 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….!