CRYOGENICS 2

Going below 1 K

Welcome to the quantum world!

590B

Makariy A. Tanatar

- History
- He3 systems
- Properties of He-3 and He-4
- Dilution refrigerator
- Demagnetization refrigeration

Short history of temperatures below 1K

1908 LHe liquefaction by **Kamerlingh Onnes**

1926 The idea of demagnetization cooling by Debye

1927-31 Realization of demagnetization cooling, <100mK

1945 He-3 liquefaction

1950 The idea of Pomeranchuk refrigerator (cooling by adiabatic solidification of He-3)

1962 The idea of dilution process (Heinz London) with G. R. Clarke, E. Mendoza

1965-66 First dilution refrigerators build in Leiden, Dubna and Manchester reaching 25 mK

1971 Superfluidity of He-3

The kid of the 20th century, parallels space rocket and nuclear studies

Similar to space rockets Tsiolkovskiy train generation of low temperatures uses several stages

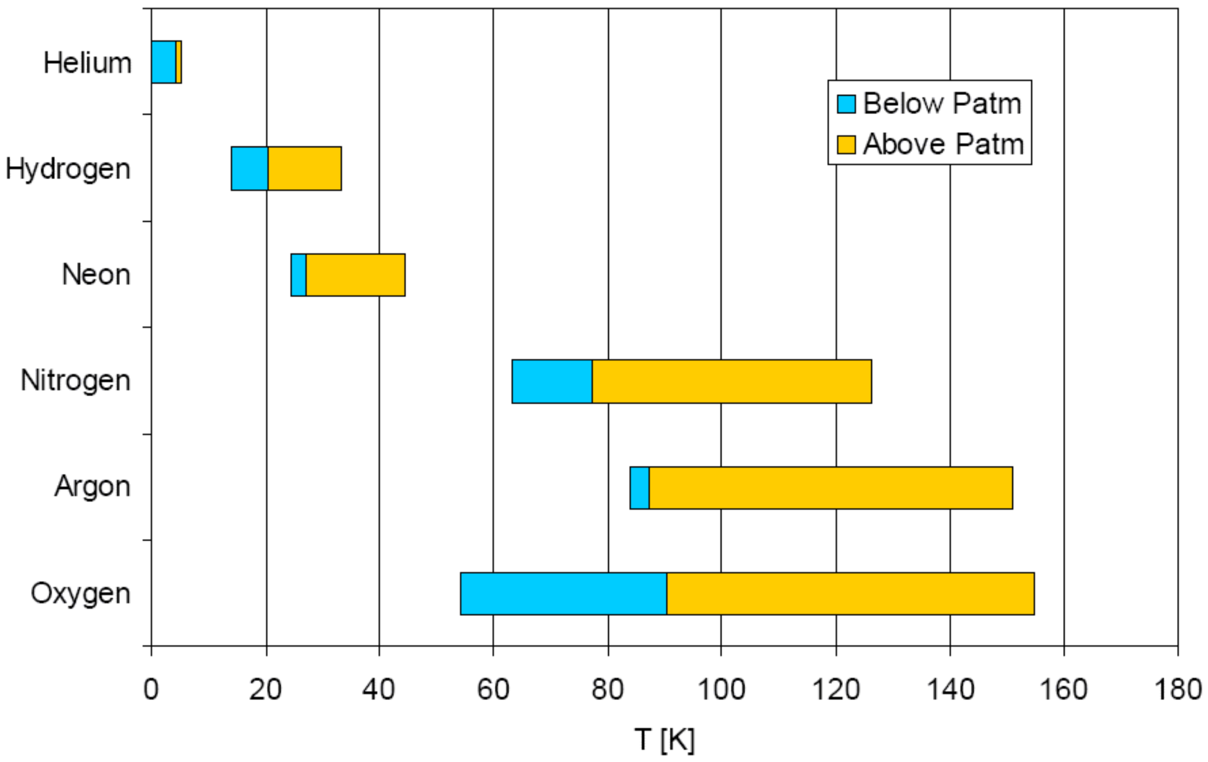
- 4K stage, usually referred to as He bath or main bath
- 1K stage, or 1K pot
- low temperature unit

The cooling power rapidly decreases with each stage,
stages are activated in sequence,
functioning of each stage impossible before full activation of the preceding

Important:
To reduce cryogen liquid consumption use to a maximum extent cooling power of the first stages

Cooling with cryogenic liquids

Useful range of cryogens



The lighter the better!
The lightest is stable He-3 isotope

Operation below 4.2 K completely relies on vacuum pumping
Pumping oil creates characteristic smell of low-temperature laboratories!

Wikipedia

Natural He contains 0.000137% of He-3.
Thousands of liters of He-3 are used annually in cryogenic applications

He-3 is produced, not mined!
Main source: nuclear fusion

Earth gravity is not strong enough to keep He in atmosphere
Once released, He leaves to space

Proposals as exotic as mining He-3 on Moon surface, where it accumulates due to exposure to Solar wind

http://www.webelements.com/helium/isotopes.html

Isotope	Atomic mass (m_a/u)	Natural abundance (atom %)	Nuclear spin (I)	Magnetic moment (μ/μ_N)
^{3}He	3.016 029 309 7(9)	0.000137 (3)	$^1/_2$	-2.127624
^{4}He	4.002 603 2497(10)	99.999863 (3)	0	0

Physical properties

	Helium-3	Helium-4
Boiling (1atm)	3.19 K	4.23 K
Critical point	3.35 K	5.19 K.
Density of liquid (at boiling point, 1atm)	0.059 g/ml	0.12473 g/ml
Latent heat of vaporization	0.026 kJ/mol	0.0829 kJ/mol

Cooling power of evaporating cryogenic liquid

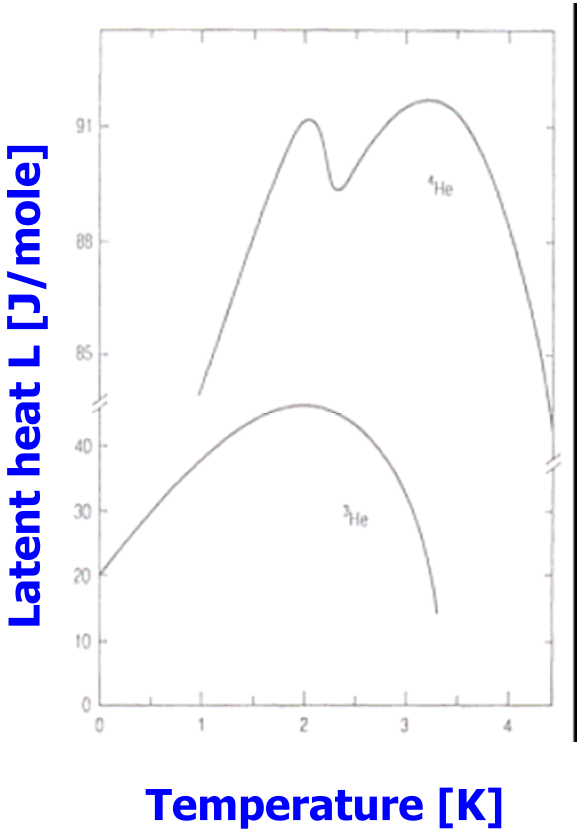
$Q=n\Delta H=nL$,

Q cooling power
n rate of evaporation, molecules/time
ΔH enthalpy of evaporation
L latent heat of evaporation

For a pump with constant volume rate V

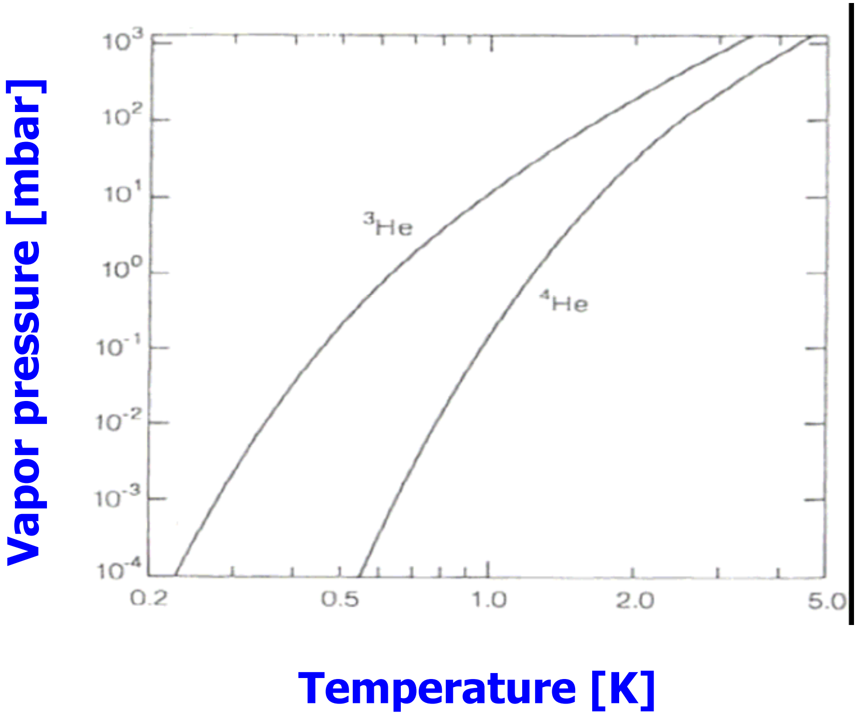
$Q=VP(T)L$

L approximately constant



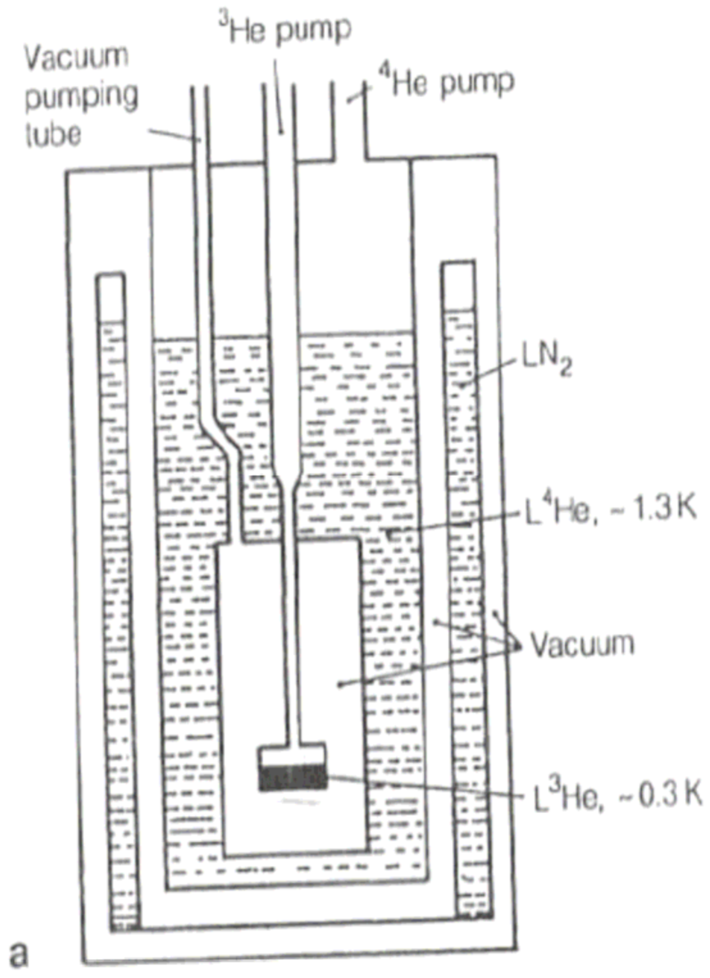
Cooling power proportional to vapor pressure
Q~P(T)~exp(-1/T)

Exponentially small at low T
We can get by pumping on
He-4 T~1K
He-3 T~0.26 K



Evaporative cooling is used in
1K pot
He-3 cryostat

He-3 refrigerator

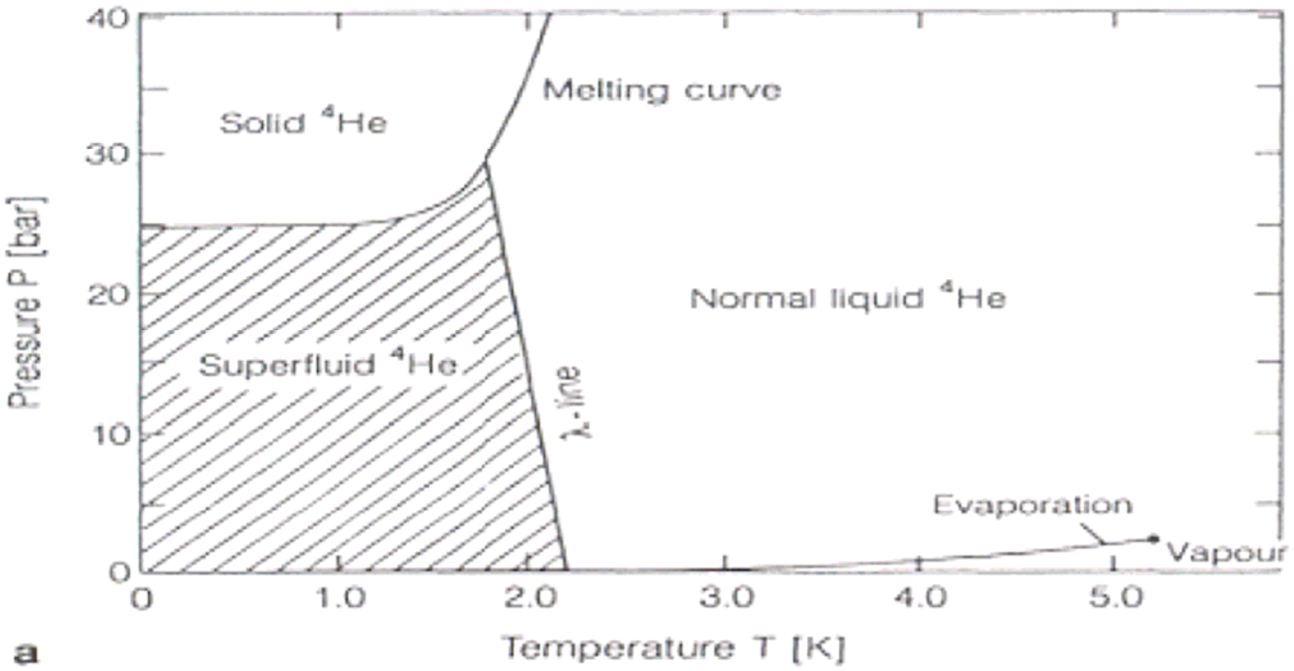


Typical features:
Sample in vacuum
(rarely sample in He-3 liquid)
One shot mode of operation
Hold time 10-60 hours

Operation sequence:
Release He-3 from cryopump
Condense by heat exchange with 1K pot
Cool condensate to 1.5K
Start cryopumping to reach base temperature

He-3 is stored in a sealed space to avoid loss
He-3 pump is called Sorb, uses cryopumping

He-4 nucleus has no spin, Boson



No solid phase due to:

weak van der Waals inter-atomic interactions, E_{pot} is low

Large quantum mechanical zero-point energy $E_0=h^2/8ma^2$ due to small mass, E_{kin} is high

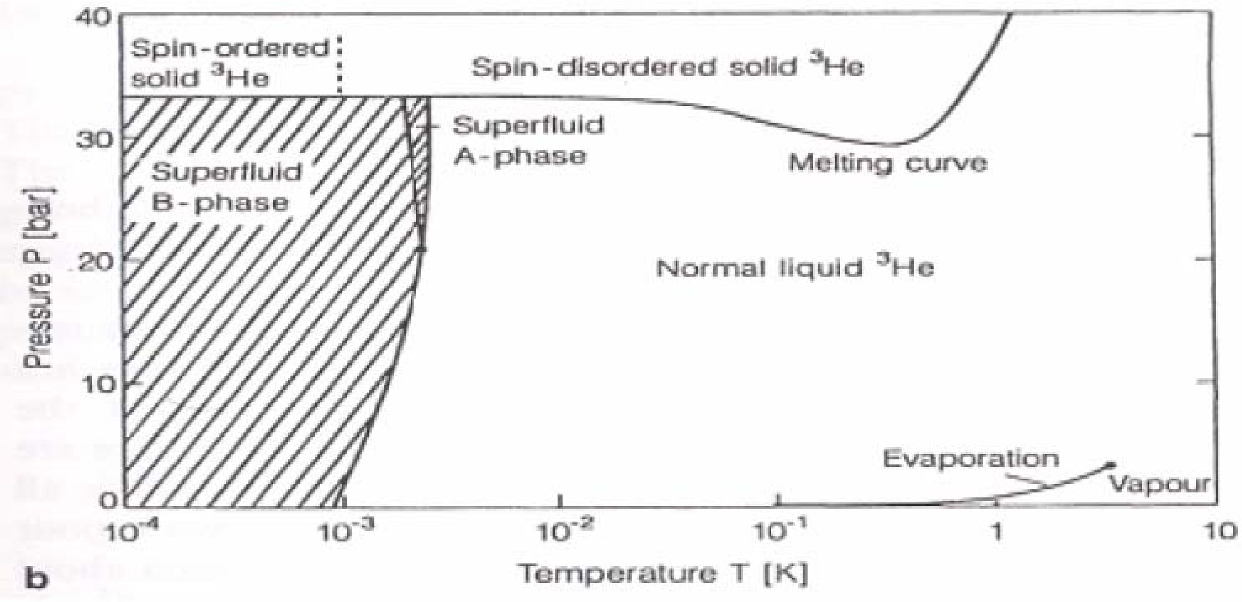
Bose-Einstein condensate instead of a solid

Quantum liquids, ratio $\lambda=E_{kin}/E_{pot}$ He4 $\lambda=2.64$ He3 $\lambda=3.05$

Amplitude of vibrations about 1/3 of interatomic space

He-3 nucleus has spin 1/2, Fermion

Additional spin entropy

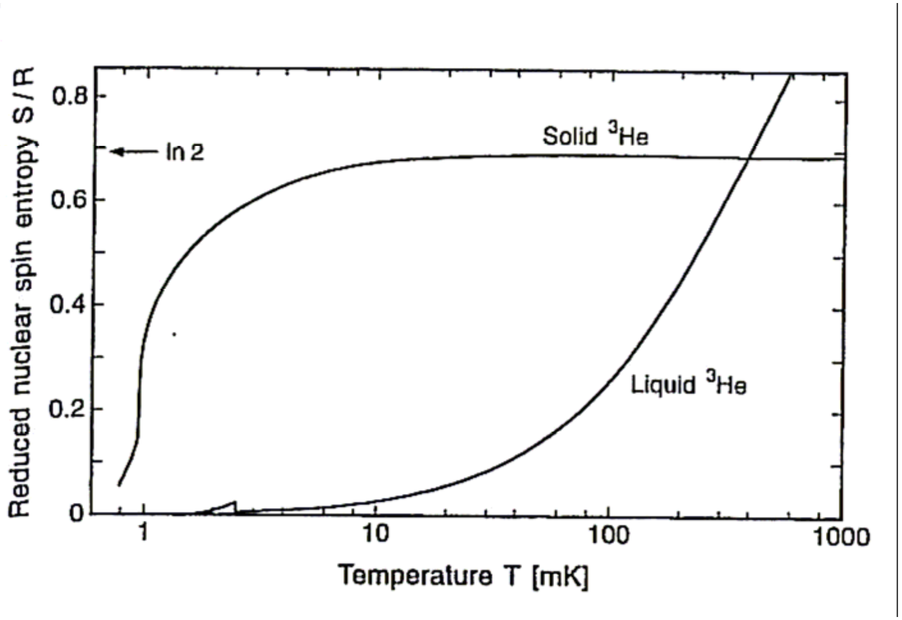


Bose-Einstein condensate of pairs, several superfluid phases

Special feature: Below T_F spins in the liquid phase are spatially indistinguishable.
Therefore they start obeying Fermi statistics and are more ordered than in the paramagnetic solid phase!

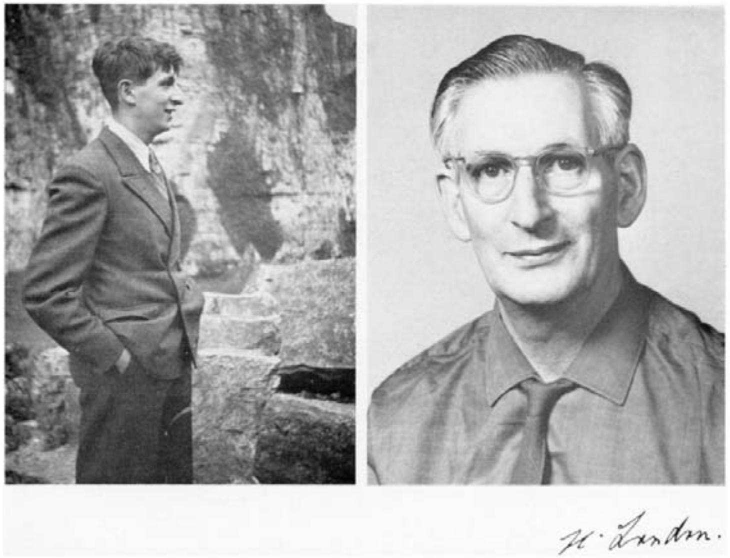
Pomeranchuk cooling (1950)

Isentropic compression of He-3 below 200 mK leads to cooling



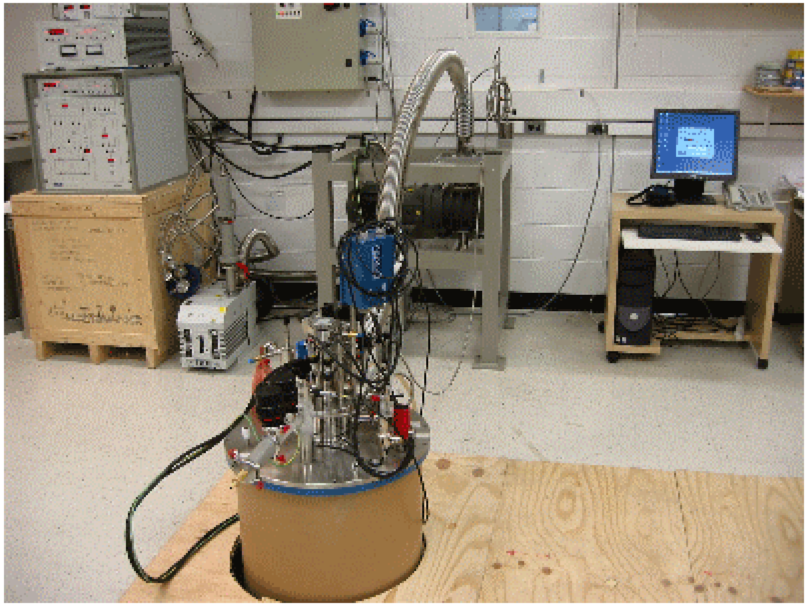
Isaak Pomeranchuk

Pomeranchuk cooling was used to discover superfluid phases in He-3



H. London.

Heinz London



Top-loading Dilution Refrigerator

In dilution process of He-3 into He3-He-4 mixture
ΔH is enthalpy of mixing
$\Delta H = \int \Delta C\, dT$

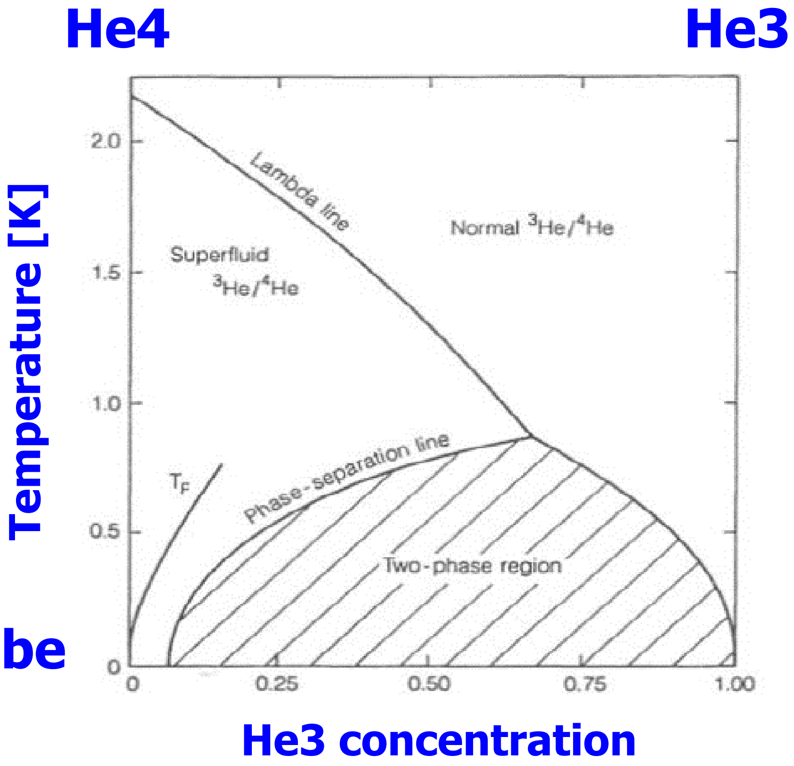
Mixture of He3 and He-4

Phase separation of the mixture into He3 rich and He3 poor phases, but not pure He3 and He4

Pure quantum effect
classical liquids should separate into pure components to obey 3rd law of thermodynamics, S=0

In case of He3-He4 mixture, S=0 can be for finite concentration because of the Fermi statistics for He3 and Bose statistics for He4

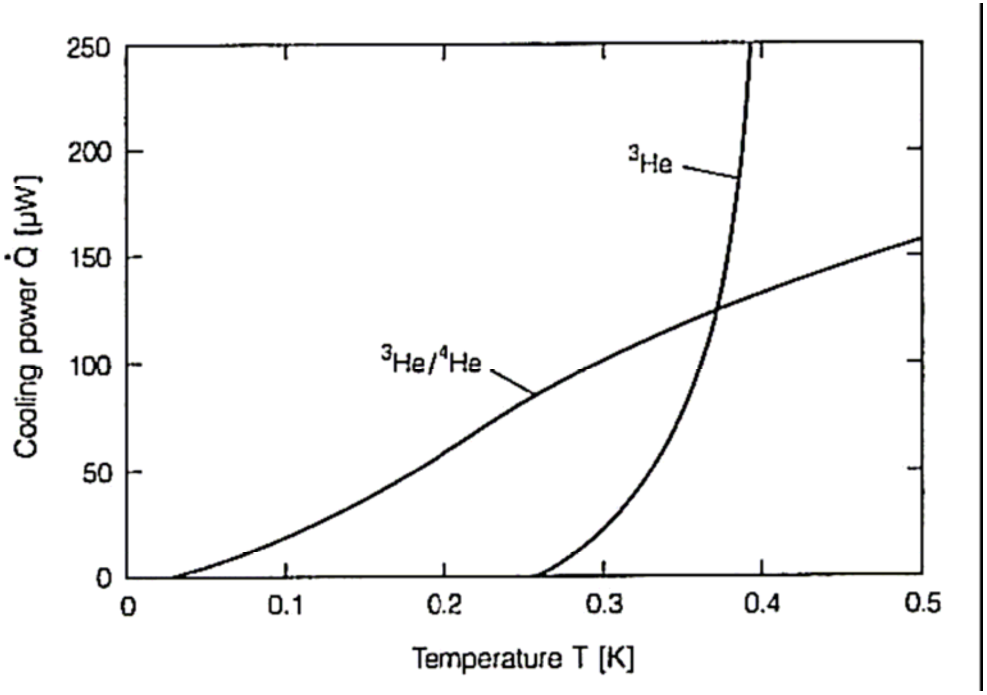
Phase separation starts below T=0.867 K (max at X=0.675)



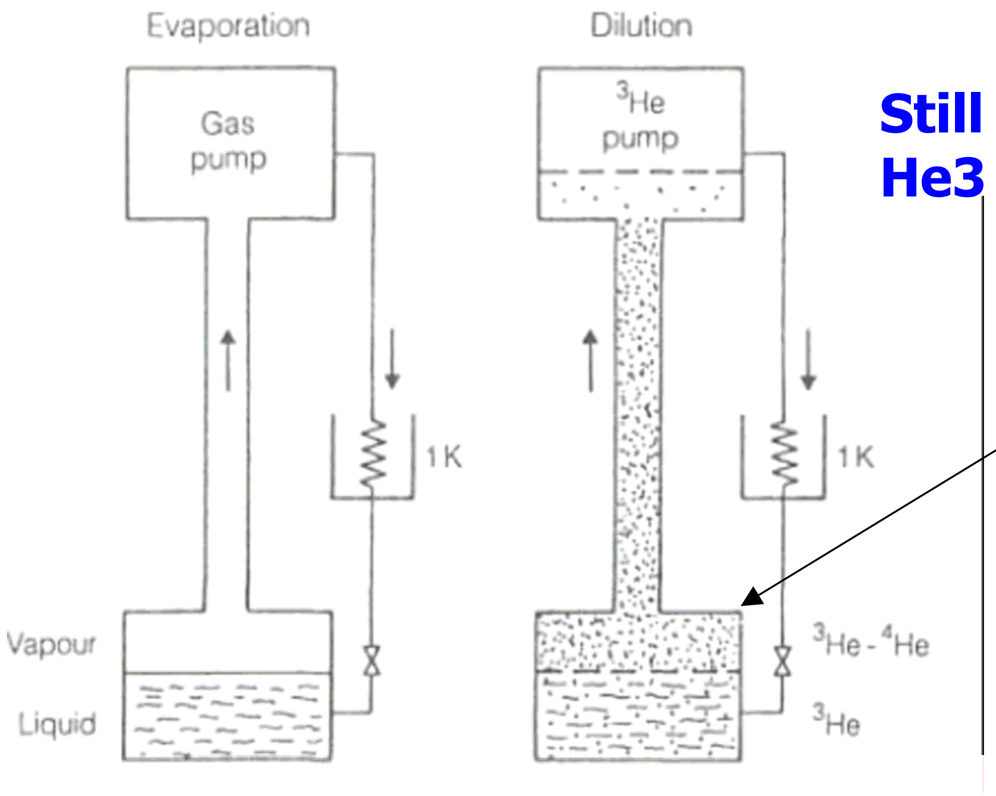
Cooling power:
Power law decrease
Instead of exponential decrease

Enthalpy of mixing uses the difference
In specific heat of two phases

$\Delta H = \int \Delta C\, dT$
$Q \sim x\, \Delta H \sim T^2$



Evaporative cooling **Dilution**

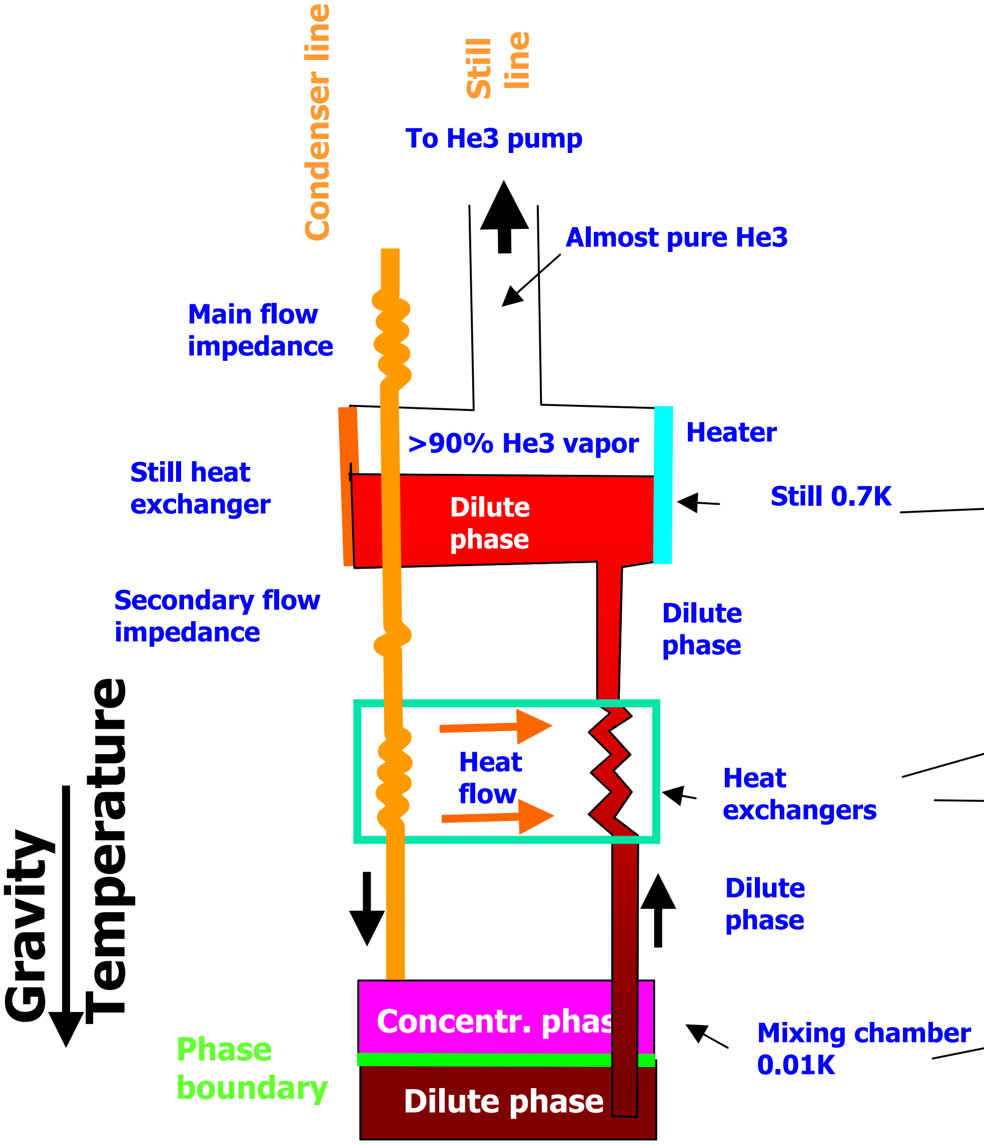


Still evaporates He3 from mixture

Mixing chamber

Phase separation line

Dilution Refrigerator: more details



Condenser line

Still line

To He3 pump

Almost pure He3

Main flow impedance

>90% He3 vapor

Heater

Still heat exchanger

Dilute phase

Still 0.7K

Secondary flow impedance

Dilute phase

Heat flow

Heat exchangers

Gravity

Temperature

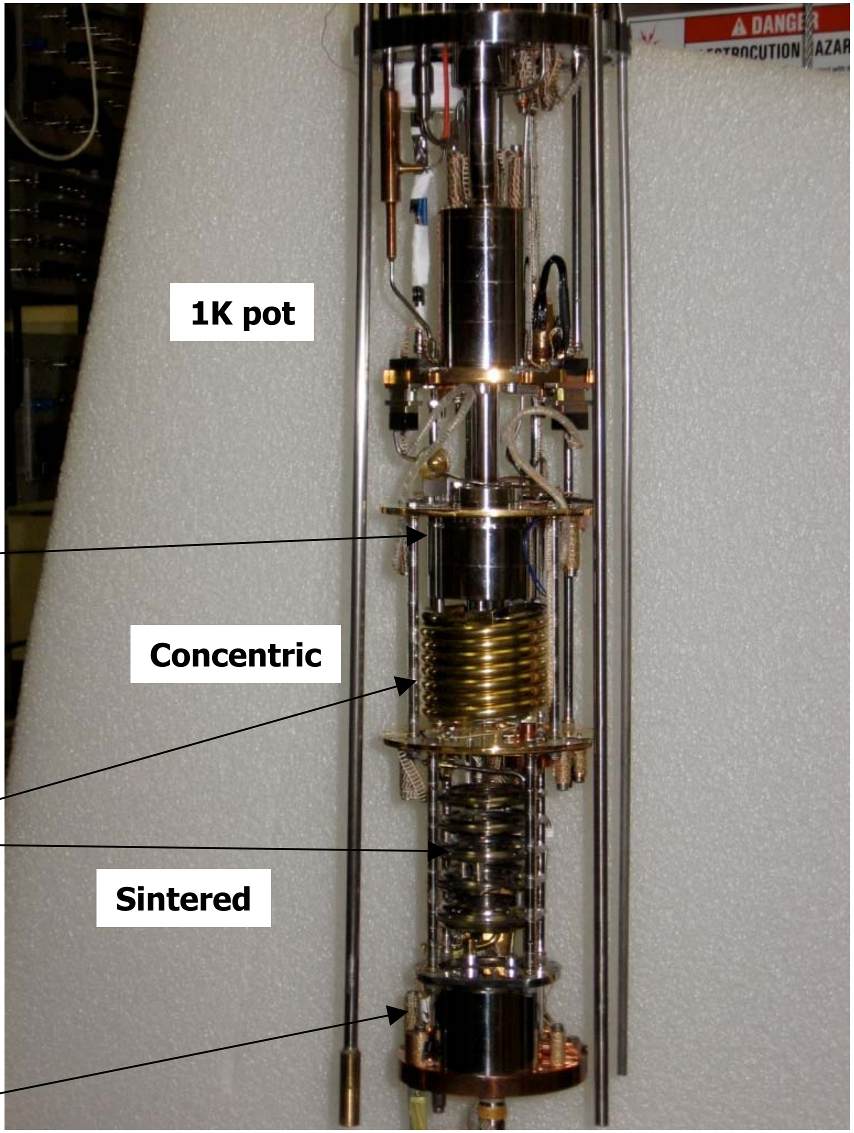
Dilute phase

Concentr. phas

Mixing chamber 0.01K

Phase boundary

Dilute phase



1K pot

Concentric

Sintered

Kapitza resistance

A discontinuity in temperature across the interface of two materials through which heat current is flowing

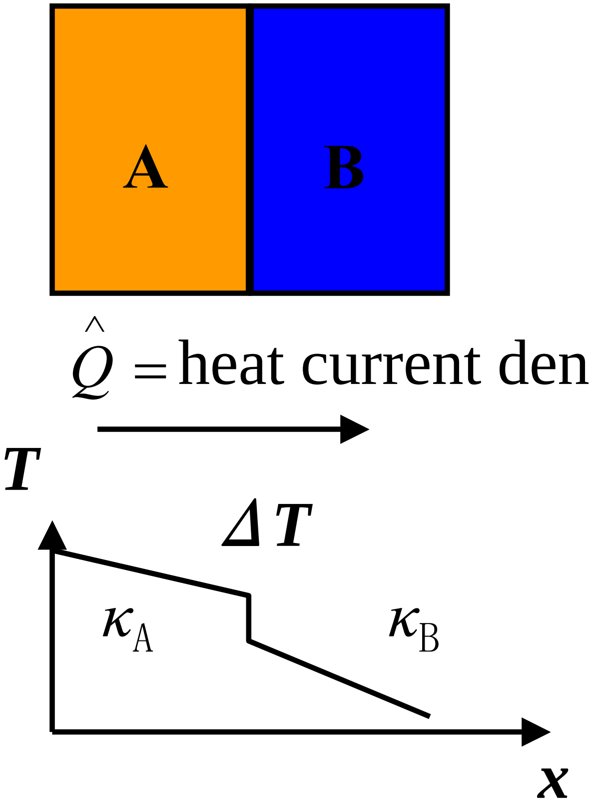
acoustic impedance mismatch at a
boundary of two substances
phonons have probability to be reflected

Kapitza resistance, $\sim T^{3}$

Important effect as *T* tends to 0

1K vs 10 mK

6 orders of magnitude change!



$\hat{Q}$ = heat current density

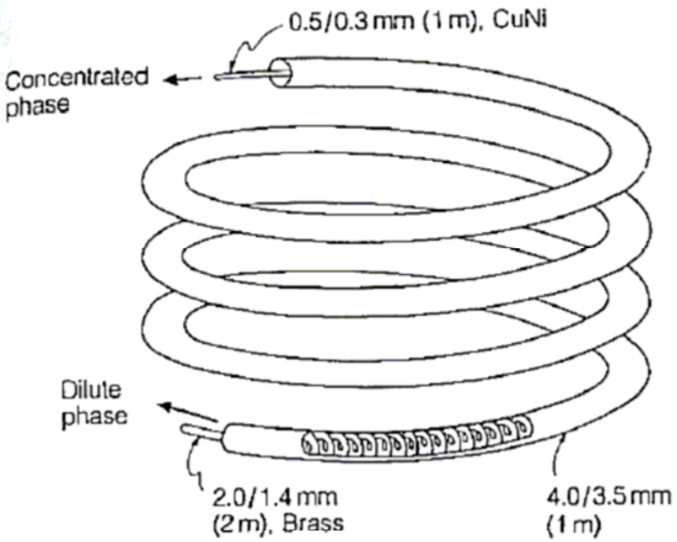
$$\hat{Q} = \kappa_K \Delta T$$

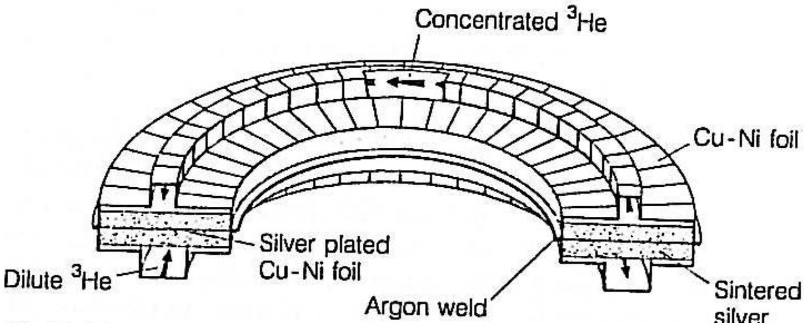
κ_K – Kapitza conductance

Good Thermal contact at low temperatures needs conduction electrons

Dilution refrigerator: heat exchanging

Need big surface area contacts!





Concentric heat exchanger
High temperatures

Welded Cu-Ni foil
Sintered submicron silver powder
Close to mixing chamber

Dilution refrigerator: Experiment cooling

Do not rely on insulating contacts!

Vibration

RF heating

Dilution refrigerator: gas handling at room temperature

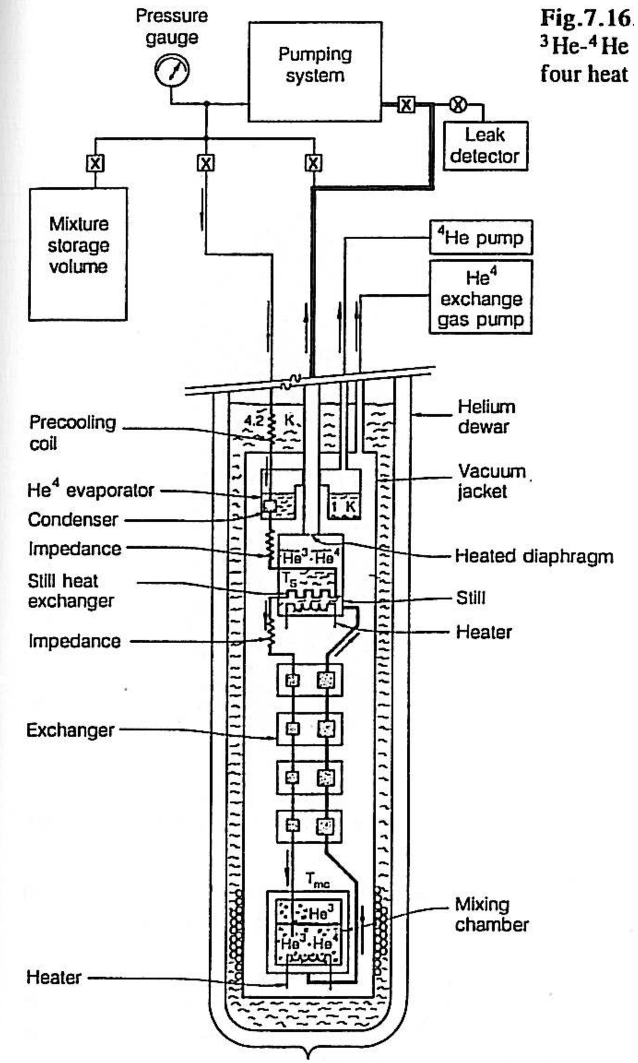


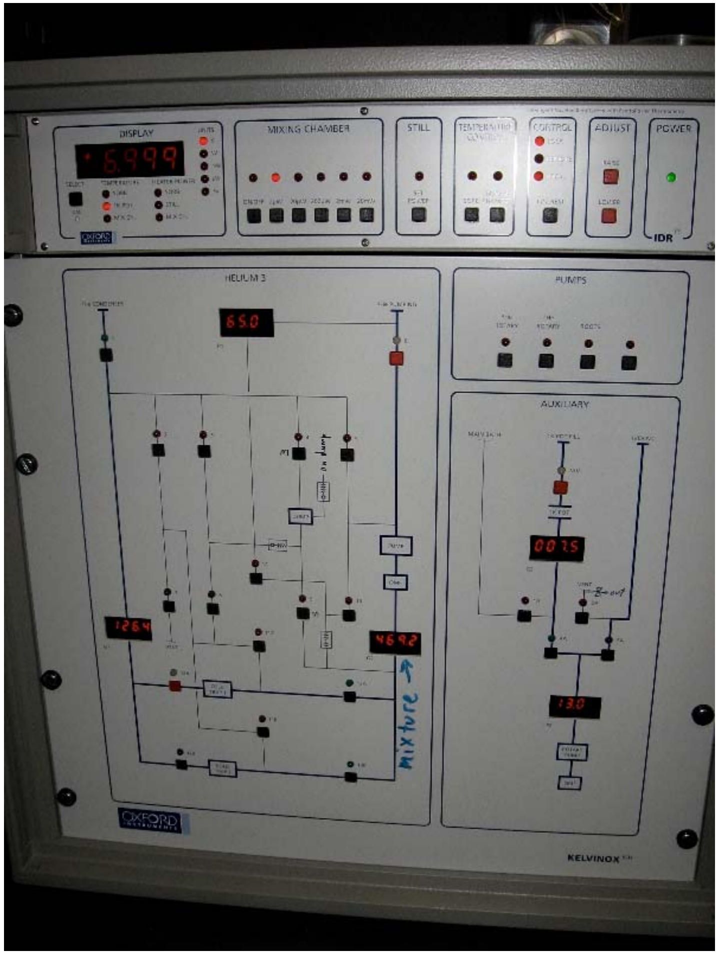
Fig.7.16 ^{3}He-^{4}He four heat

Key elements:
He3-He4 Gas storage "Dump"
Vacuum pump for 1K pot
Vacuum pump for He3 circulation
Roots (booster) pump for Still line pumping
Cold traps for mixture cleaning

Very demanding to vacuum leaks
To avoid loss of mixture,
all operation goes at $P<P_{atm}$
Leaks in, not out!

Dilution refrigerator: gas handling system

Front view



He3 **He4**

Back view

Dilution refrigerator: pumps

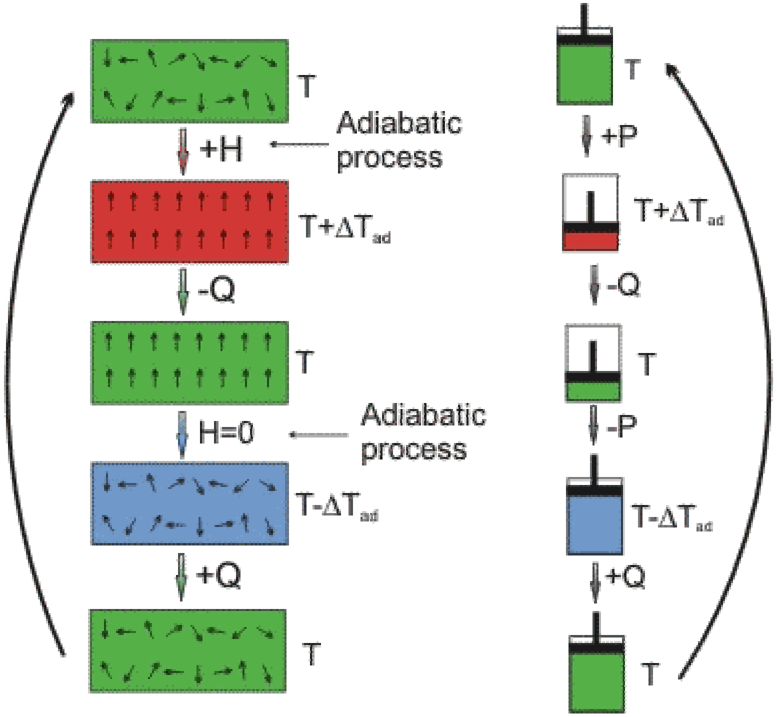
Still line

Roots Booster pump

He3 pump

He4 pump for 1K pot

WIKIPEDIA

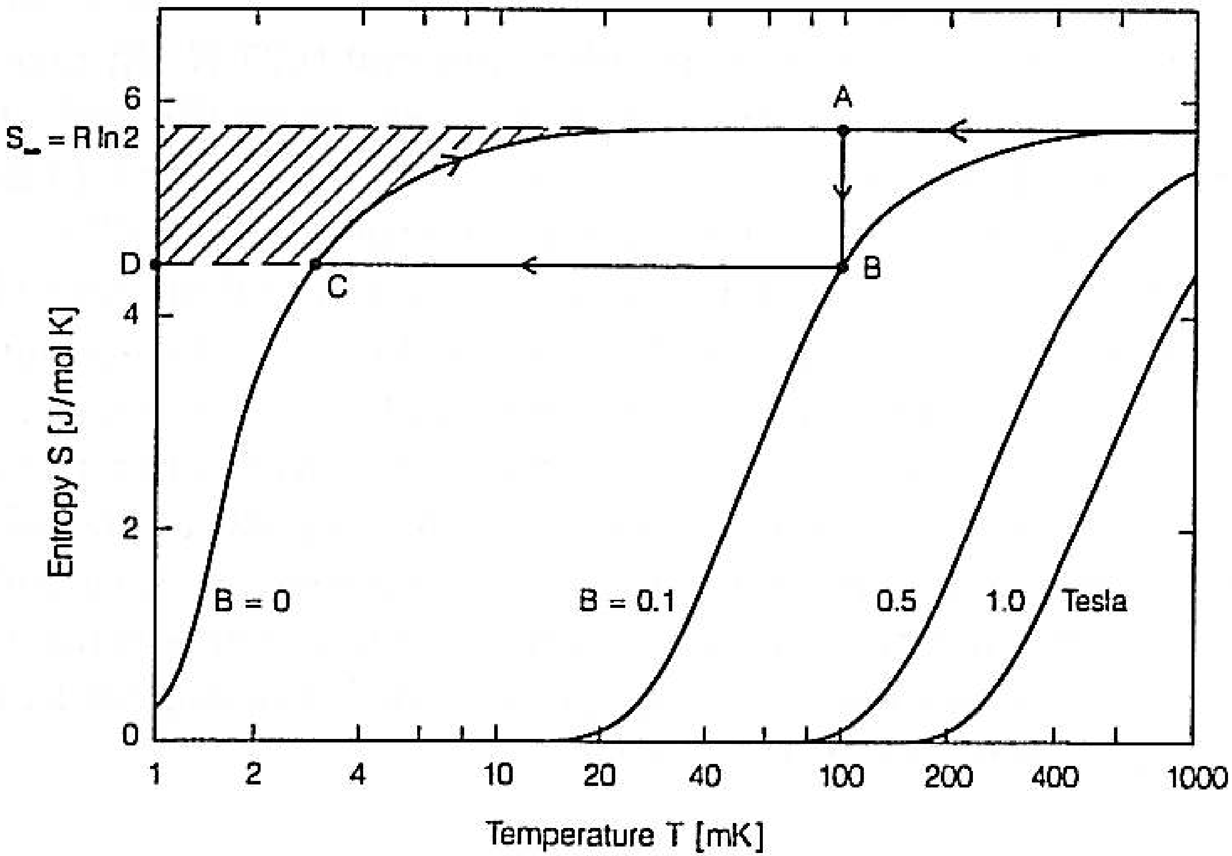


Magnetic refrigeration Vapor cycle refrigeration

Adiabatic magnetization
Isomagnetic enthalpic transfer
Adiabatic demagnetization
Isomagnetic entropic transfer

Peter Joseph William Debye

Weakest point: needs heat switch working at 1K


6
4
2
0
$S_\infty = R \ln 2$
A
B
C
D
B = 0
B = 0.1
0.5
1.0
Tesla
Entropy S [J/mol K]
1
2
4
10
20
40
100
200
400
1000
Temperature T [mK]

Demagnetization fridge

"High"-temperature salts:

MAS: $Mn^{2+}SO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$; $T_c \simeq 0.17\ K$

FAA: $Fe_2{}^{3+}(SO_4)_3 \cdot (NH_4)_2SO_4 \cdot 24H_2O$; $T_c \simeq 0.03\ K$

"Low"-temperature salts:

CPA: $Cr_2{}^{3+}(SO_4)_3 \cdot K_2SO_4 \cdot 24H_20$; $T_c \simeq 0.01\ K$

CMN: $2Ce^{3+}(NO_3)_3 \cdot 3Mg(NO_3)_2 \cdot 24H_2O$; $Tc \simeq 0.002\ K$.

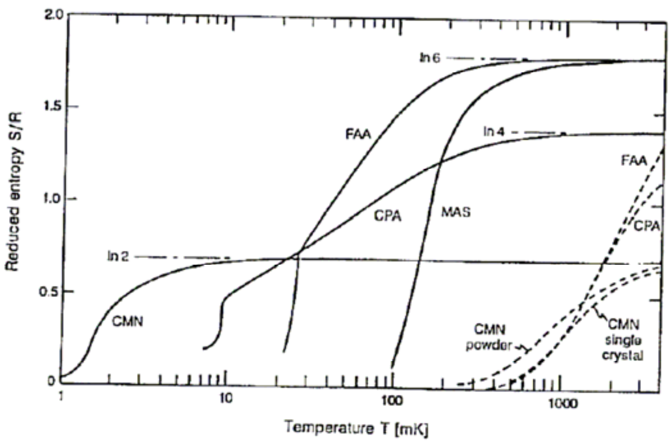
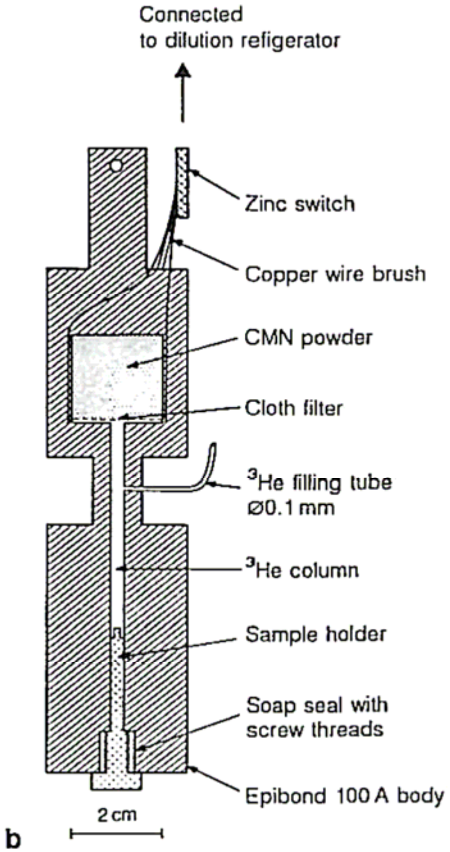
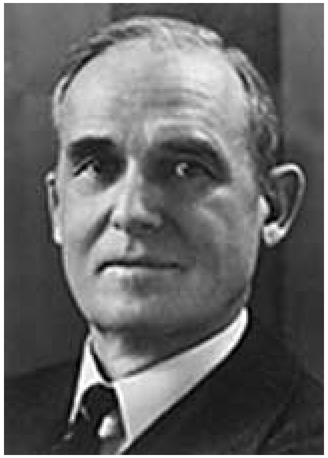


Fig.9.2 Entropies S (divided by the gas constant R) of four salts suitable for paramagnetic demagnetization as a function of temperature in zero field (*full lines*) and in 2 T (*dashed lines*). (For the chemical formula of the salts see the text)



Experimental realization, T<100 mK
Experimental verification of 3rd law
of thermodynamics, S→ 0 when T → 0

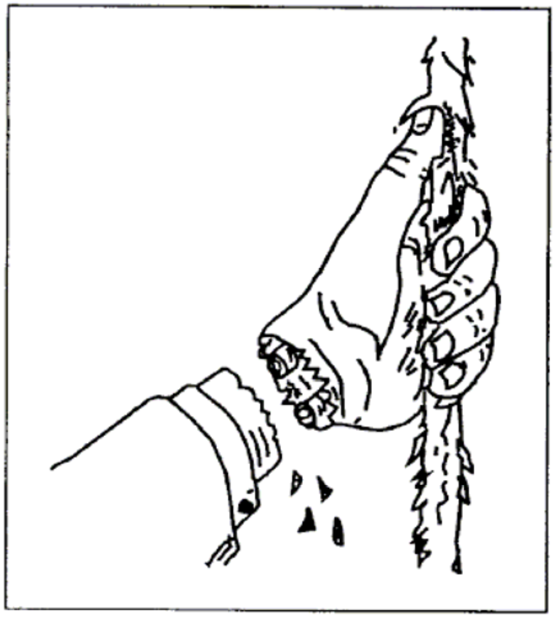


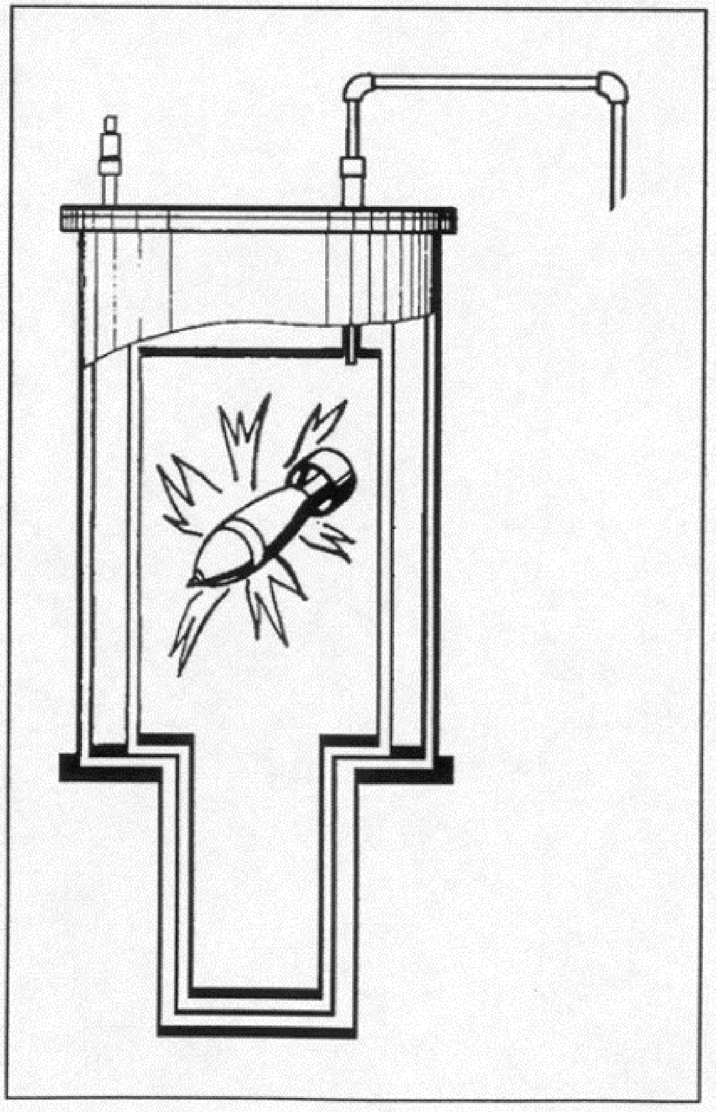
William F. Giauque

Commercially available from Janis, CMR, Dryogenics
~60mK
Short experiment time, residual magnetic fields
Relatively inexpensive

Safety

Flesh may freeze and stick to cold surfaces





DON'T PANIC

Ref. [6]

Safety

Temperature

Gas Pressure

Energy (SC magnets)

Damage to expensive equipment

Loss of mixture

Do not let it happen here!

Known cases in my experience

Kyoto: 15T magnet on energization attracted rotary pump, which destroyed vacuum Dewar
Loss of the magnet

Kyoto: due to an air leak to cryogenic center
Liquefier was damaged, 0.5 mln $ loss

Sherbrooke: quench due to He magnet bath exhaust damaged magnet
Critical field reduced from 15T to 0.7T

Sherbrooke: due to a leak, dipper accumulated He liquid inside and exploded on warming, fortunately no one was injured

Cornell: closed 1K pot with He liquid inside and relieve valve frozen, destroyed DF

Bristol: on a day like today, student opened OVC vacuum valve
1 million worth of equipment

Reading materials

Matter and methods at low temperatures
Author: Frank Pobell; Springer, 2007

Experimental techniques in low-temperature physics
Author: Guy K. White; Clarendon Press, 1979

Experimental low-temperature physics
Author: Anthony Kent; American Institute of Physics, 1993

Experimental techniques in condensed matter physics at low temperatures
Author: Robert C Richardson; Eric N Smith Addison-Wesley Pub. Co., 1988

Experimental techniques for low-temperature measurements : cryostat design, material properties, and superconductor critical-current testing
Author: J. W. Ekin; Oxford University Press, 2006

Hitchhiker's guide to dilution refrigerators,
Nathaniel Creig and Ted Lester