How to get down to microkelvin

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HighRR Seminar, 17.05.2017

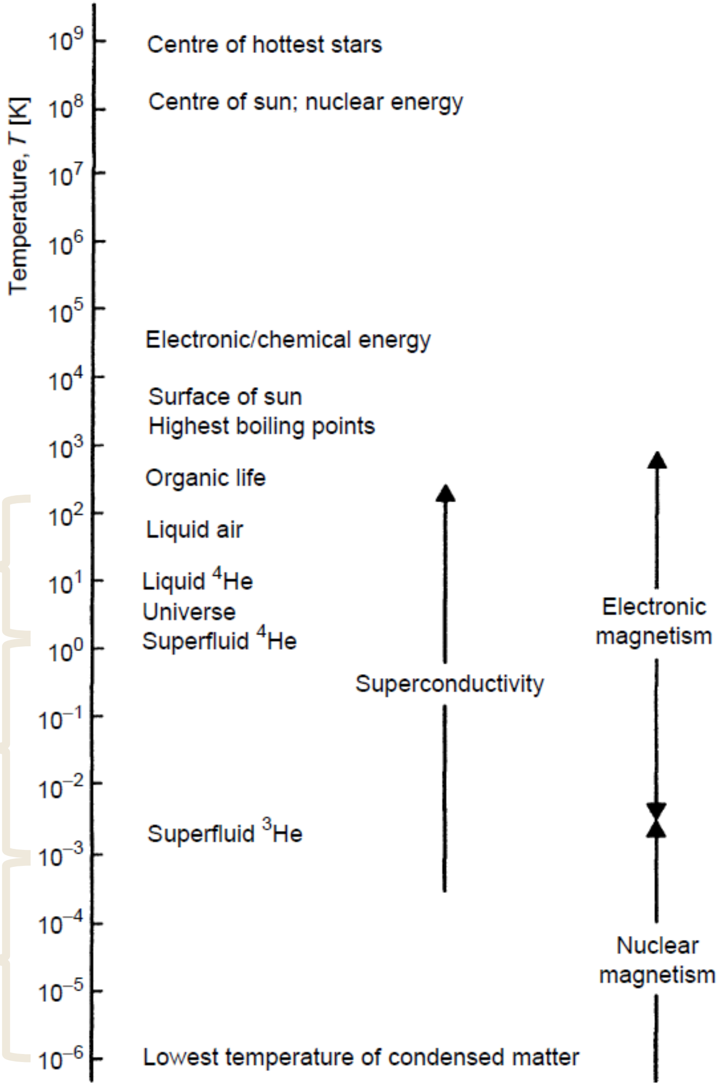
Contents

- History
- Liquification of Cryoliquids

- ^{4}He Cryostat (wet)
- Pulse Tube Cooler (dry)

- ^{3}He/^{4}He Dilution Refrigerator
- Adiabatic Demagnetization

- Nuclear Demagnetization



History

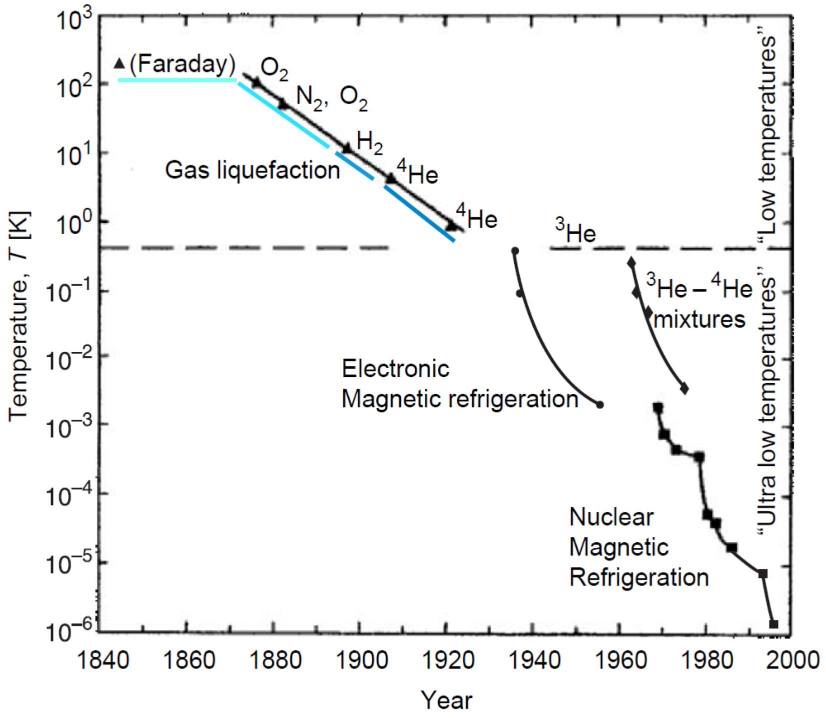
Historic Introduction

1845: Faraday successfully liquified most known gases, except for six, which became known as the permanent gases: oxygen, hydrogen, nitrogen, carbon monoxide, methane and nitric oxide (helium not found as gas yet).

1877: Oxygen and nitrogen were liquified by Louis Cailletet (France) and Raoul Pictet (Switzerland), 80 K reached.

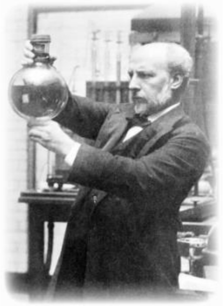
1898: Liquefaction and solidification of hydrogen by James Dewar (13 K reached), Cambridge.

1908: Last permanent gas, helium, was liquified by Kamerlingh Onnes, Leiden. He reached 0.83 K in 1922.



Michael Farady (1791 – 1867)

Raoul Pictet
(1846 – 1929)



James Dewar
(1842 – 1923)

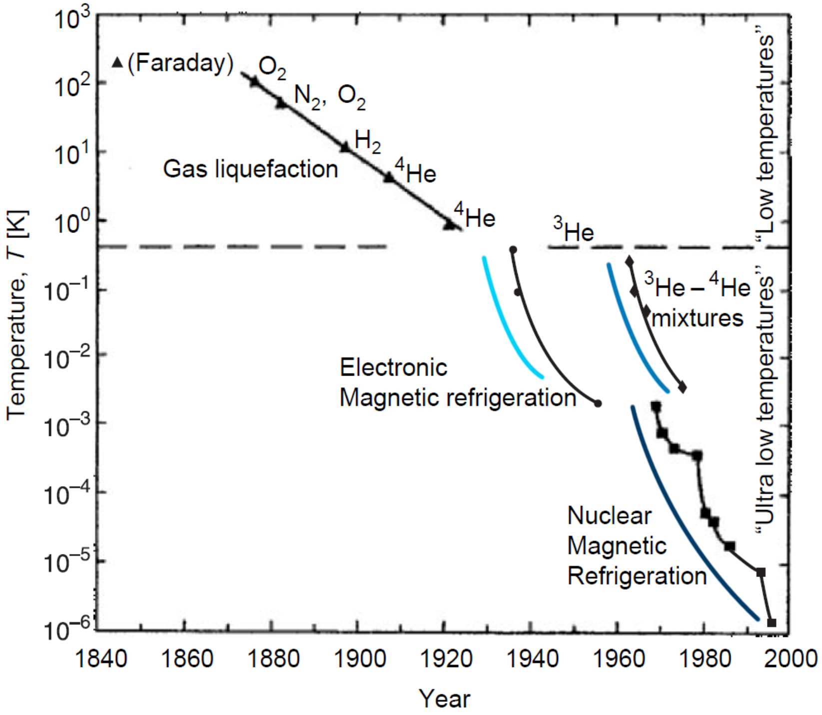
Heike Kamerlingh Onnes
(1853 – 1926)

Historic Introduction

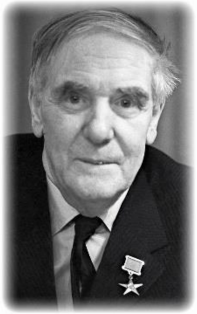
1930‘s: Adiabatic Demagnetization opens up lower millikelvin range for experiments
(single shot cooling)
Today: $T = 5 - 30$ mK (record: 1 mK)

1960‘s: Development of Dilution Refrigerators which allow continueous cooling to millikelvin.
Today: $T = 5 - 20$ mK (record: 2 mK)

1970‘s: Nuclear Demagnetization makes microkelvin temperatures available for experiments
(single shot cooling)
Today: $T < 100$ µK (record 1.5 µK)

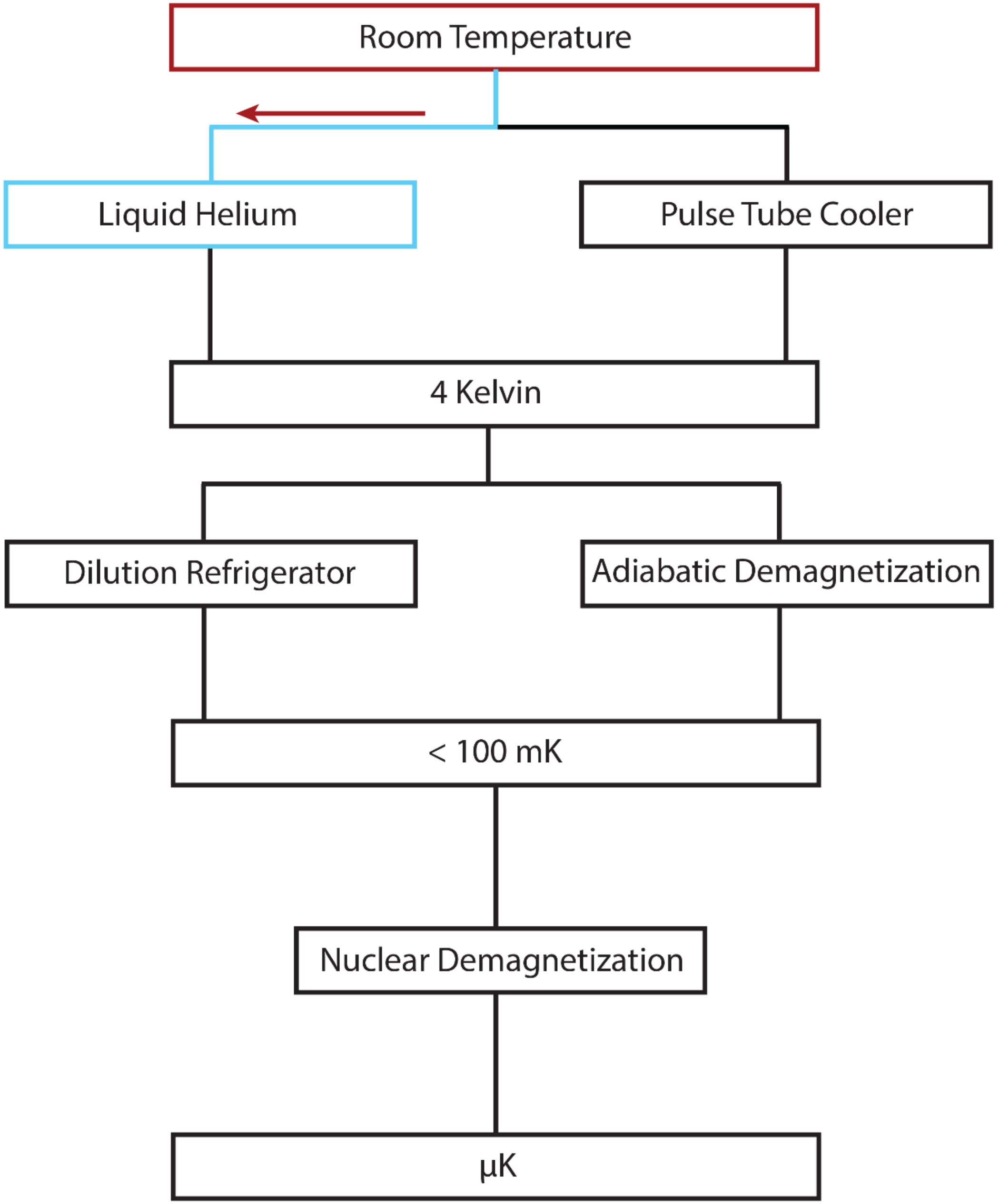


Peter Debye
(1884 – 1966)



Pjotr L. Kapitza
(1894 – 1994)

Heinz London
(1907 – 1970)



Room Temperature

Liquid Helium

Pulse Tube Cooler

4 Kelvin

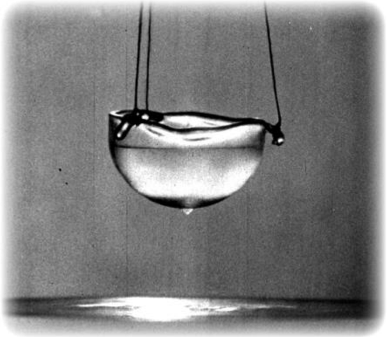
Dilution Refrigerator

Adiabatic Demagnetization

< 100 mK

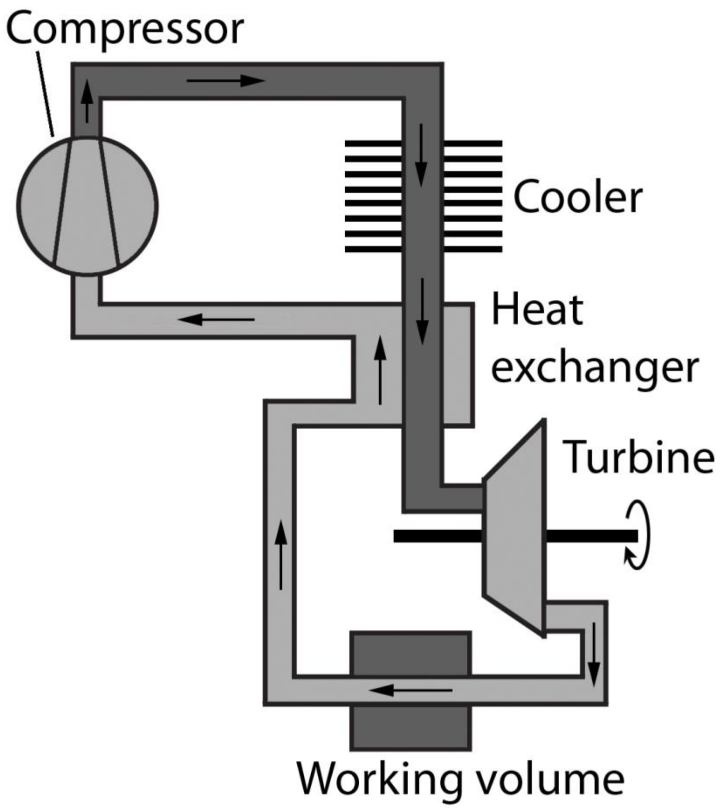
Nuclear Demagnetization

µK



Liquification of Cryoliquids

Expansion Cooler



- Compression at room temperature
- Removal of excess heat at cooler
- Further cooling at heat exchanger
- Adiabatic expansion at turbine:

$$T_2 = T_1 \left(\frac{p_1}{p_2}\right)^{\frac{1-\gamma}{\gamma}} \quad \text{with} \quad \gamma = c_p/c_v$$

- Expansion: gas performs work (ideal case)

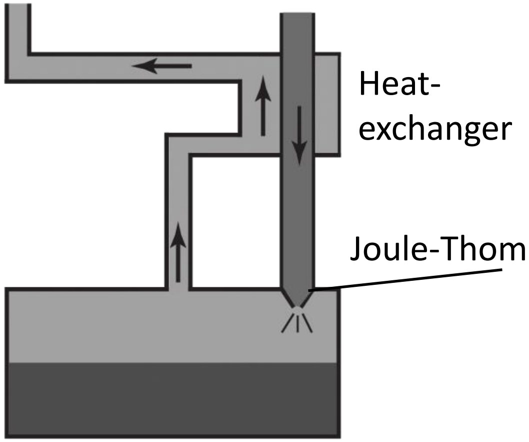
$$W = H_1 - H_2 = (U_1 + p_1V_1) - (U_2 + p_2V_2)$$

- Ideal gas:

$$W = \frac{5}{2} N k_B (T_1 - T_2)$$

- Gas performs work at the turbine, going up to 180.000 rpm

Joule-Thomson-Expansion

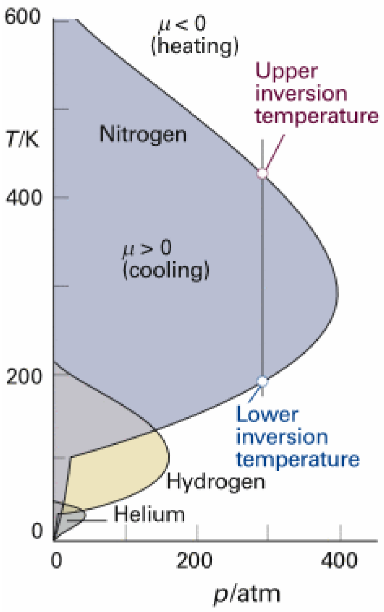


Pre-cooling

Heat-exchanger

Joule-Thomson-Valve

Inversion curve



- Expansion at nozzle, gas has to perform work against internal forces
- Change of internal energy during adiabatic expansion:

$$\Delta U = U_2 - U_1 = p_1 V_1 - p_2 V_2$$

- Joule-Thomson-Coefficient:

$$\mu_{\mathrm{JT}} = \left(\frac{\partial T}{\partial P}\right)_H = \frac{V}{C_{\mathrm{p}}}(\alpha T - 1)$$

- Cooling if $\mu_{\mathrm{JT}} > 0$
- Heating if $\mu_{\mathrm{JT}} < 0$

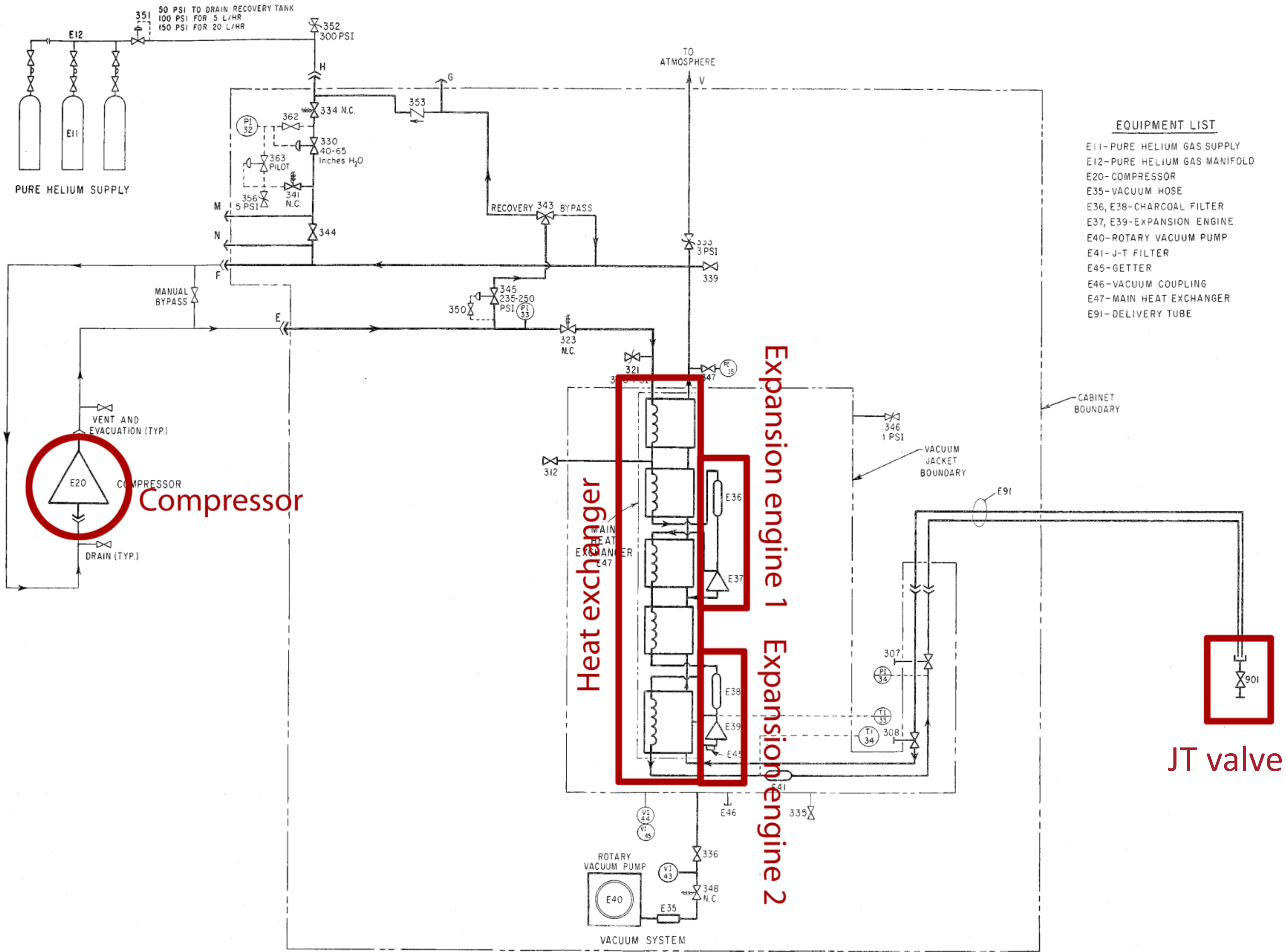
➢ $T_{\mathrm{inv,O_2}} = 764\ \mathrm{K}$

➢ $T_{\mathrm{inv,N_2}} = 621\ \mathrm{K}$

➢ $T_{\mathrm{inv,{}^4He}} = 40\ \mathrm{K}$

➢ $T_{\mathrm{inv,{}^3He}} = 23\ \mathrm{K}$

KIP He-Liquifier



EQUIPMENT LIST

E11-PURE HELIUM GAS SUPPLY
E12-PURE HELIUM GAS MANIFOLD
E20-COMPRESSOR
E35-VACUUM HOSE
E36, E38-CHARCOAL FILTER
E37, E39-EXPANSION ENGINE
E40-ROTARY VACUUM PUMP
E41-J-T FILTER
E45-GETTER
E46-VACUUM COUPLING
E47-MAIN HEAT EXCHANGER
E91-DELIVERY TUBE

Compressor

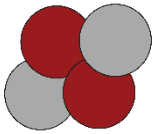
Heat exchanger

Expansion engine 1

Expansion engine 2

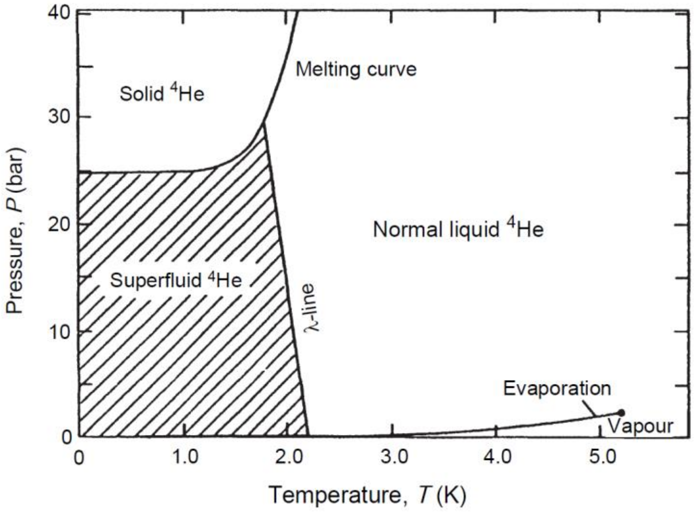
JT valve

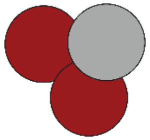
Cryoliquid Helium

^{4}He: 

- Natural occurrence as gas: helium-rich natural gas reservoirs enriched by alpha decay

- Nuclear spin *I* = 0 (boson)

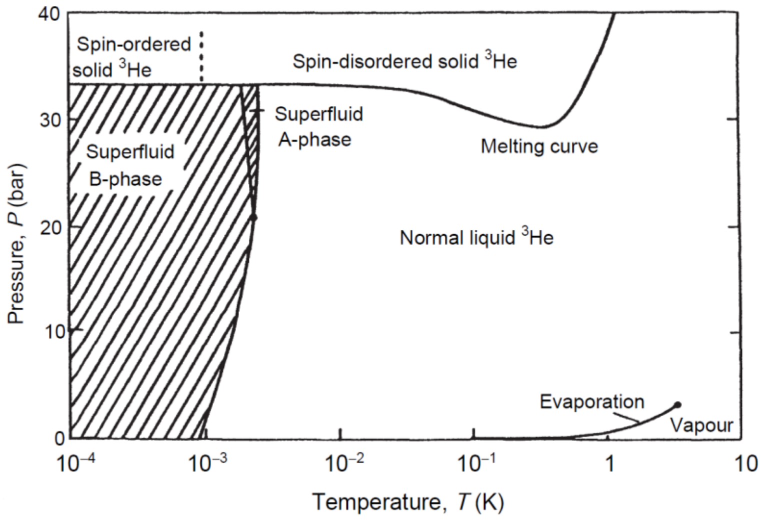


^{3}He: 

- Byproduct of tritium fabrication in a nuclear reactor:

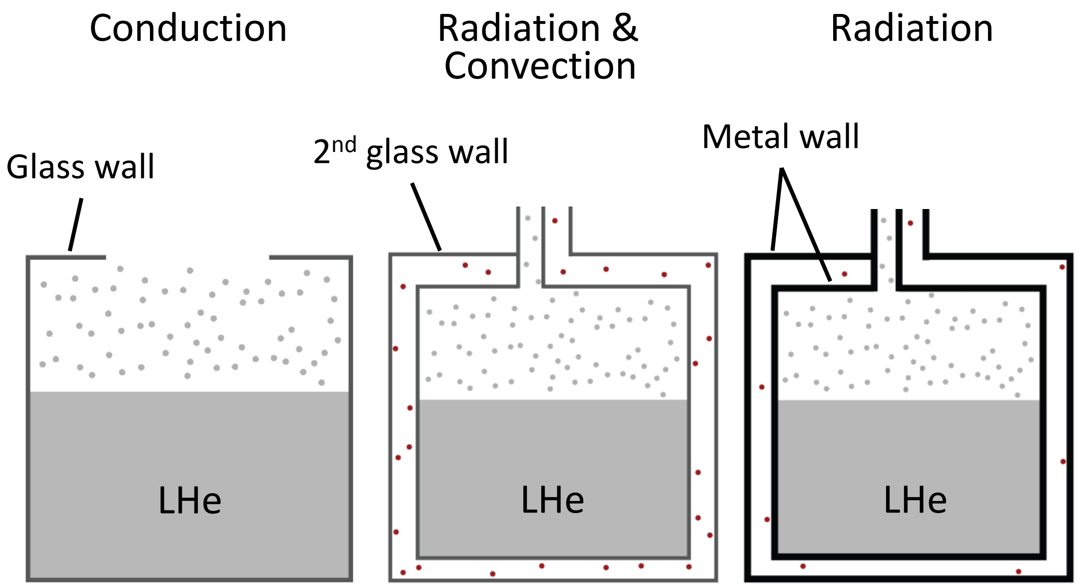
$$
\begin{aligned}
{}^6_3\mathrm{Li} + {}^1_0 n \quad &\Rightarrow \quad {}^3_1 T + {}^4_2\mathrm{He}\,, \\
{}^3_1 T \quad &\overset{12.3y}{\Rightarrow} \quad {}^3_2\mathrm{He} + {}^{\;\,0}_{-1}\mathrm{e} + \bar{\nu}
\end{aligned}
$$

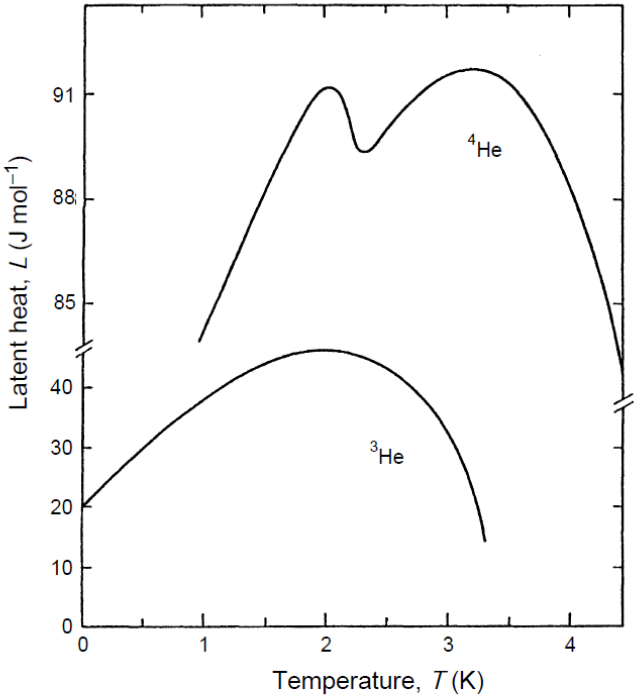
- Nuclear spin *I* = 1/2 (fermion)



Latent Heat of Evaporation

- Latent heat *L*: energy needed for the phase transition from liquid to gas
- *L* of helium very small compared to other liquids
- Very good shielding against external parasitic heat input needed
- Energy input from:





L at boiling point:

$$L_{^3\mathrm{He}} = 16\ \mathrm{J\ mol^{-1}}$$

$$L_{^4\mathrm{He}} = 84\ \mathrm{J\ mol^{-1}}$$

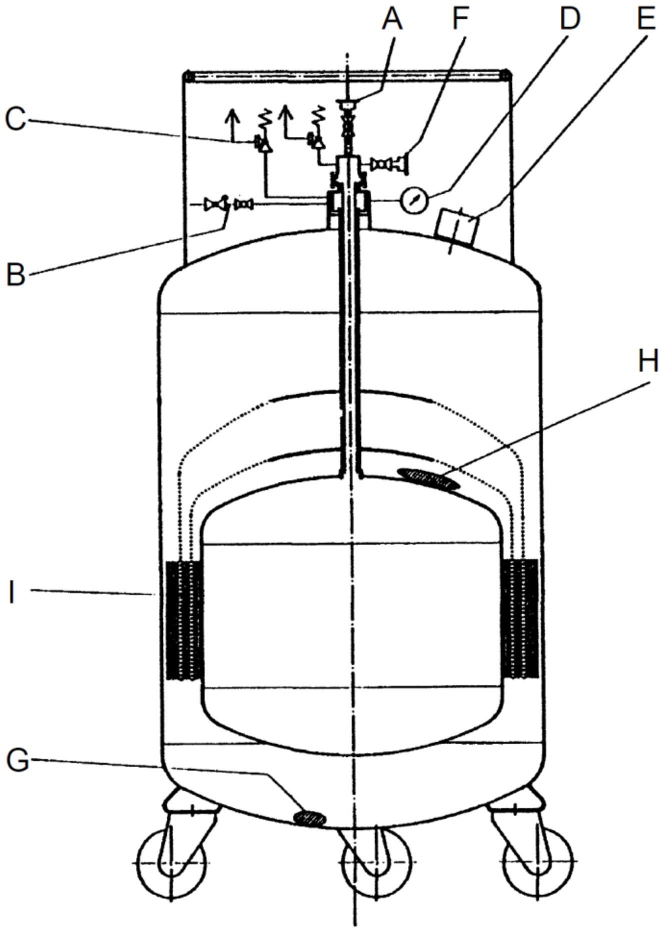
$$L_{\mathrm{H2}} = 449\ \mathrm{J\ mol^{-1}}$$

$$L_{\mathrm{N2}} = 2792\ \mathrm{J\ mol^{-1}}$$

$$L_{\mathrm{Ar}} = 6447\ \mathrm{J\ mol^{-1}}$$

Transport of liquid Helium

- A: Connection for transfer tube
- B: Overflow valve
- C: Safety valve
- D: Manometer
- E: Vakuum and safety valves
- F: Gas valve
- G: Getter Material
- H: Adsorbent Material
- I: Superinsulation



Rate of evaporation ~ 1 %/day

Transport of liquid Helium

- A: Connection for transfer tube
- B: Overflow valve
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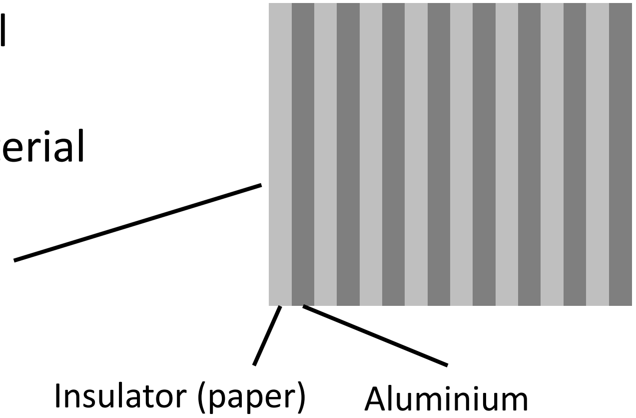
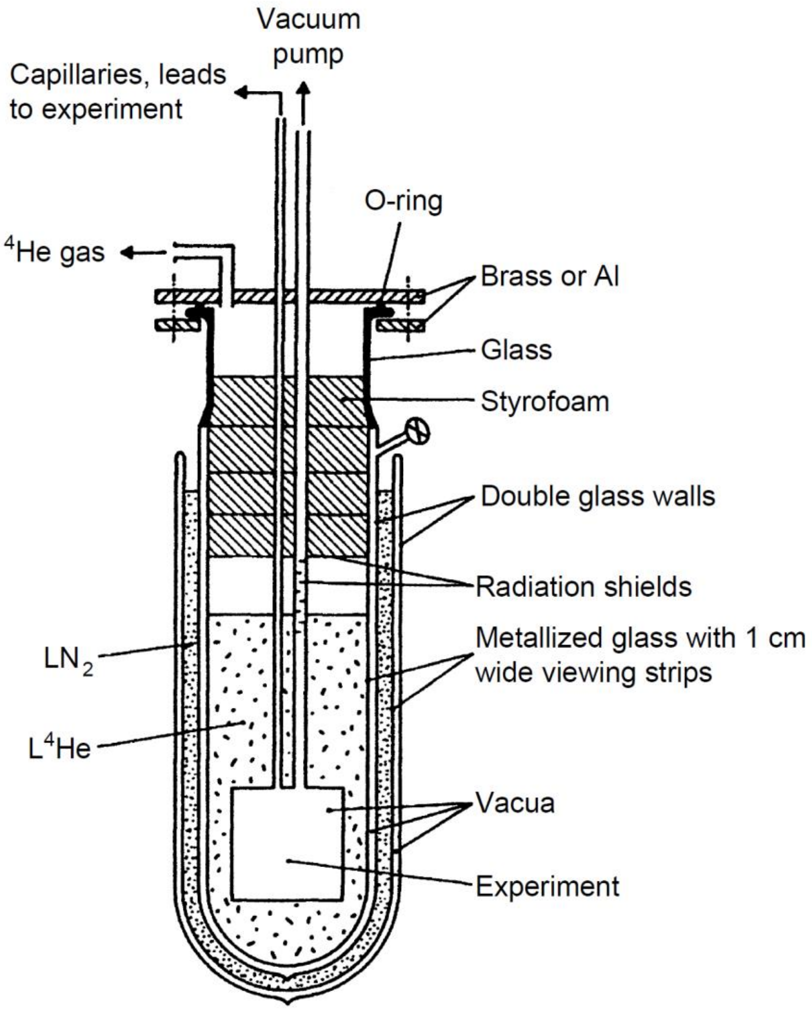


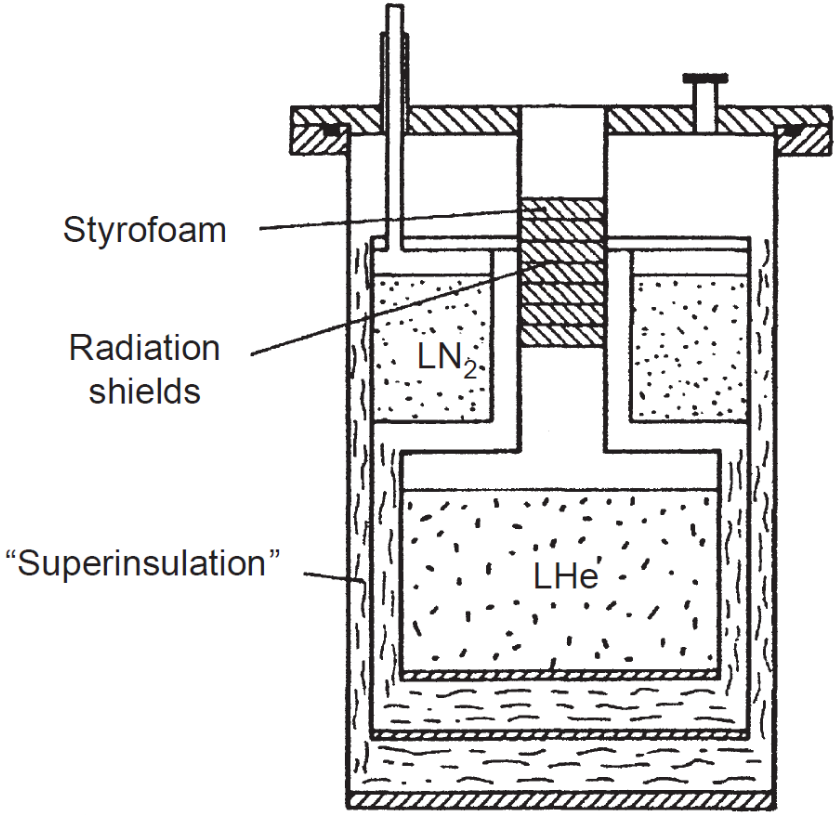
^{4}He Cryostat

^{4}He Bath Cryostat

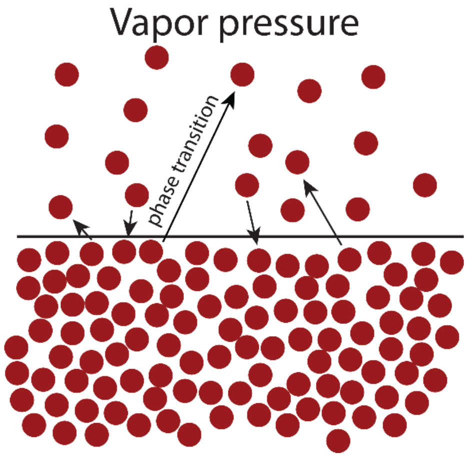
"Historic":

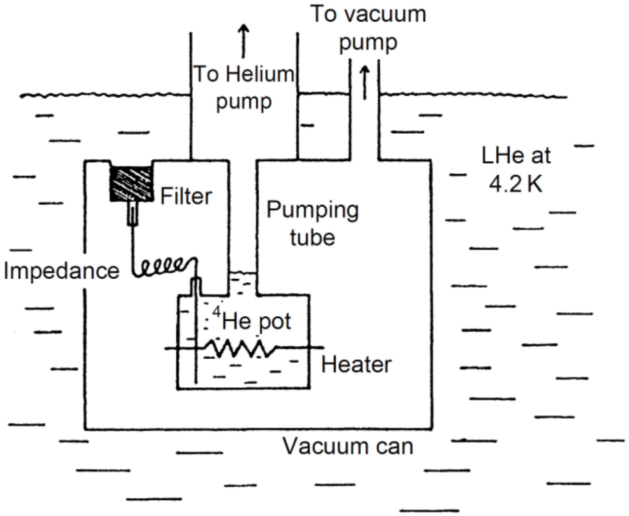


Modern:



Evaporation Cryostat



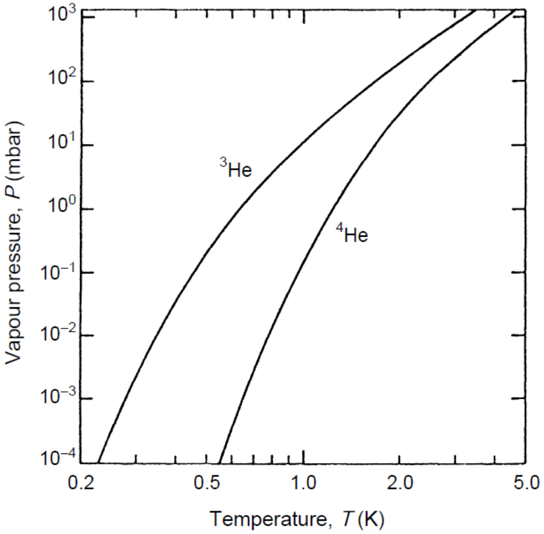


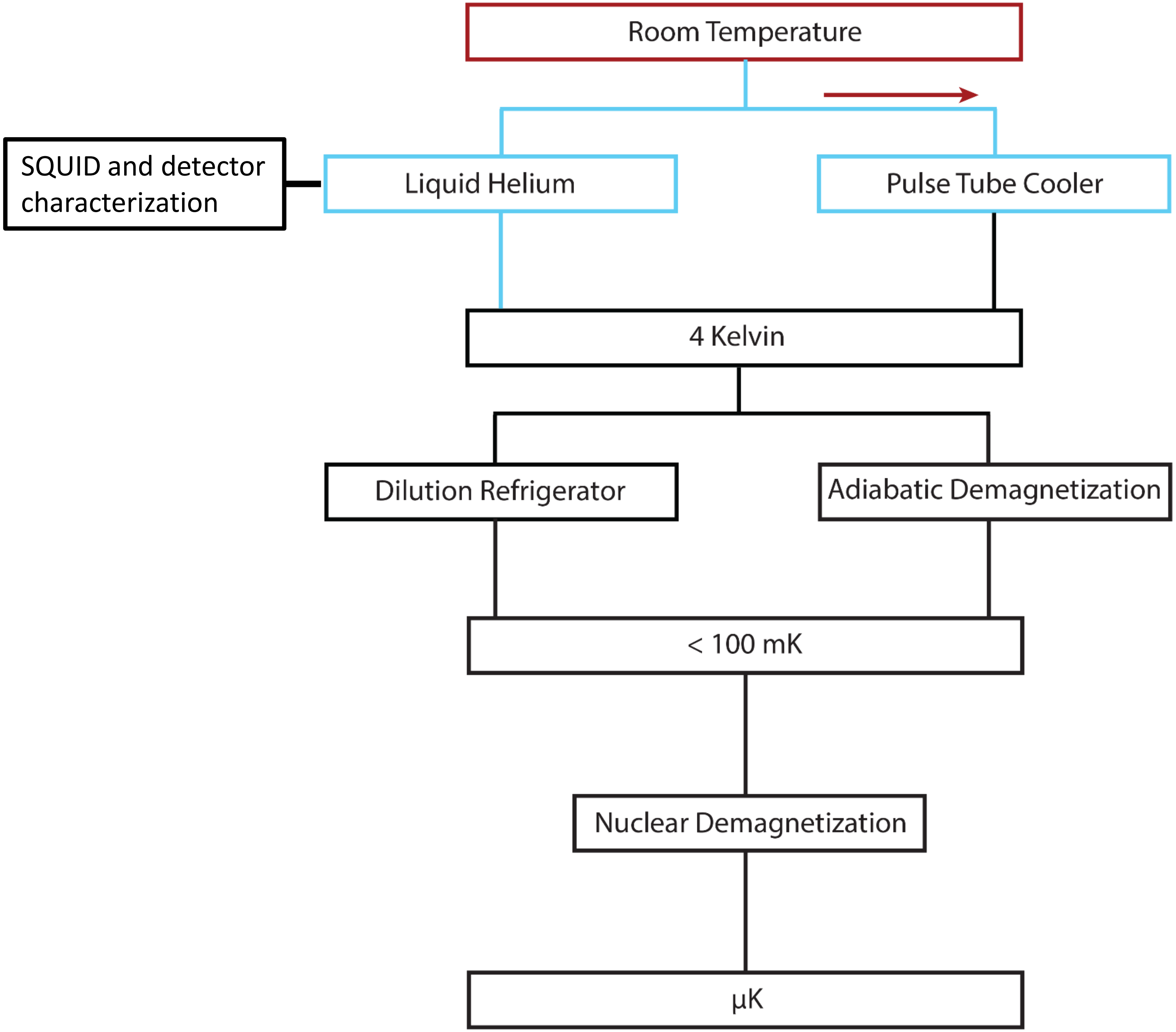
Vapor pressure: $P_{\text{vap}} \propto \mathrm{e}^{-L/RT}$

Cooling power: $\dot{Q} = \dot{n}_g L$

Typical achieved temperatures:

^{4}He: 1.3 K
^{3}He: 0.3 K





Room Temperature

SQUID and detector characterization

Liquid Helium

Pulse Tube Cooler

4 Kelvin

Dilution Refrigerator

Adiabatic Demagnetization

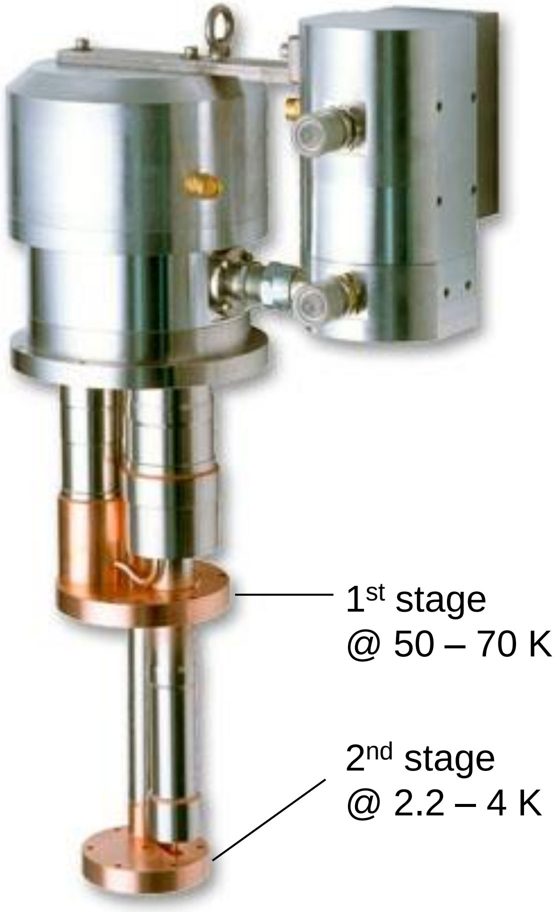
< 100 mK

Nuclear Demagnetization

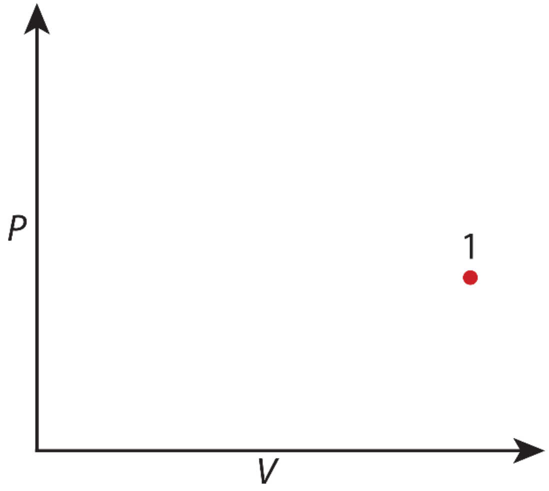
µK

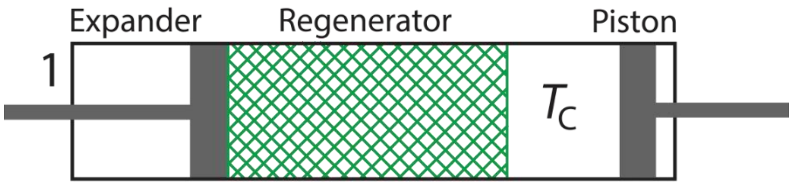
Pulse Tube Cooler

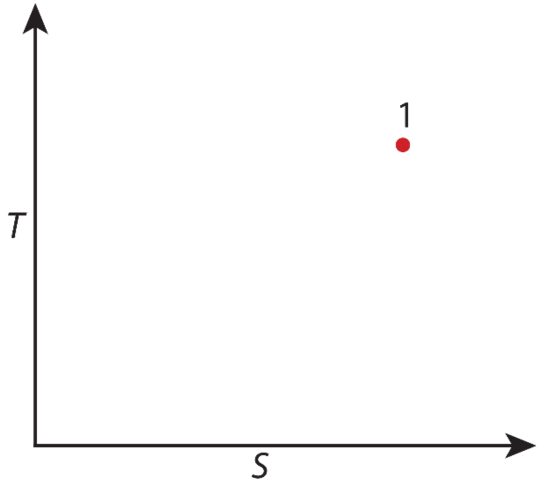
- Closed cycle refrigeration
- From 300 K to 2.2 K without cryoliquids
- Working principle: Compression, expansion and displacement of gas
- Helium as working gas under high pressure (18 -22 bar)
- Power consumption: 2 – 8 kW



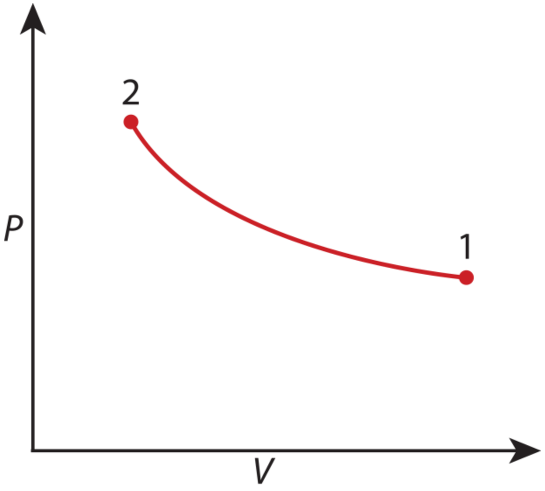
Ideal Stirling Cycle

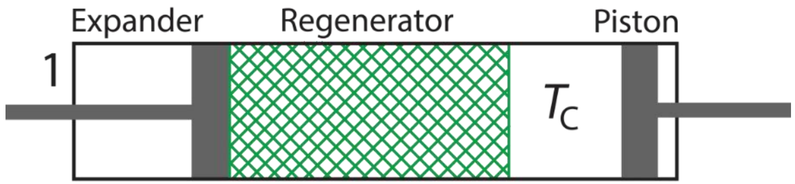




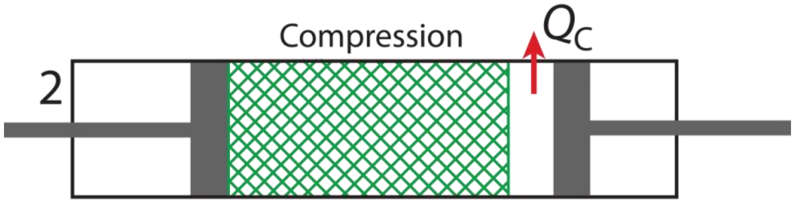


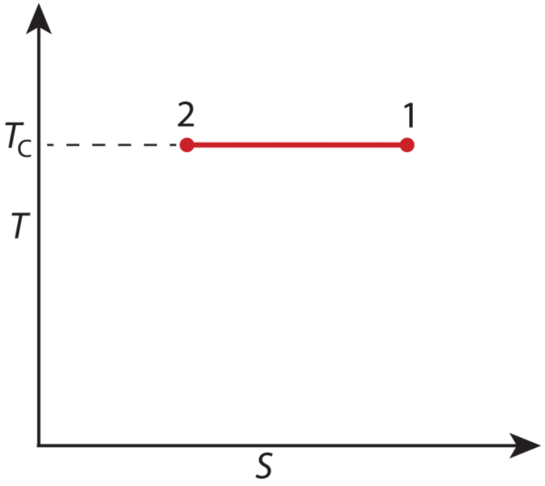
Ideal Stirling Cycle



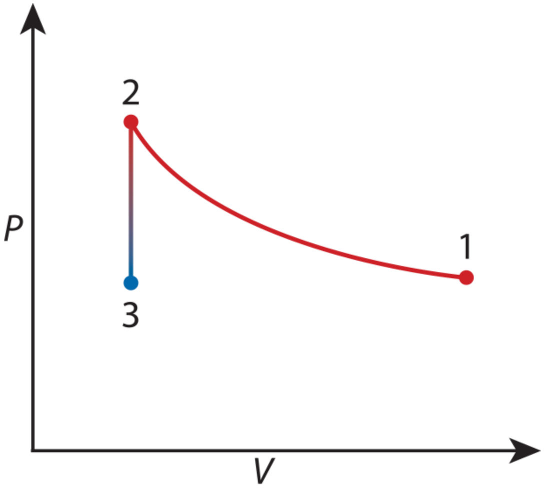


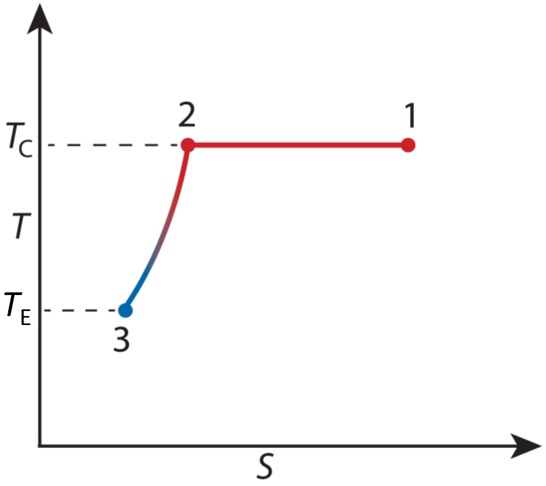
Isothermal compression

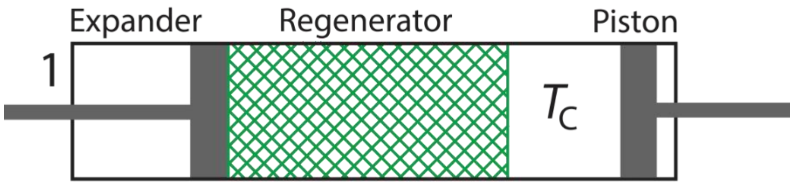




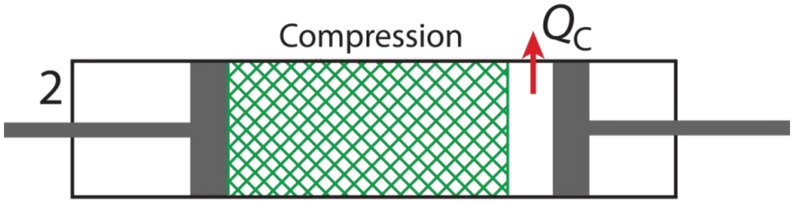
Ideal Stirling Cycle



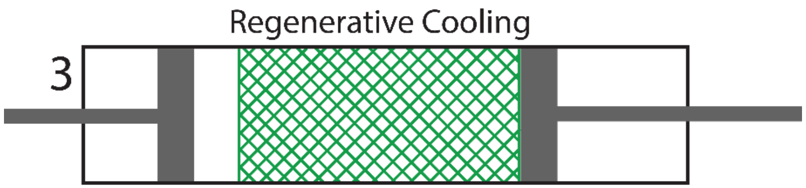




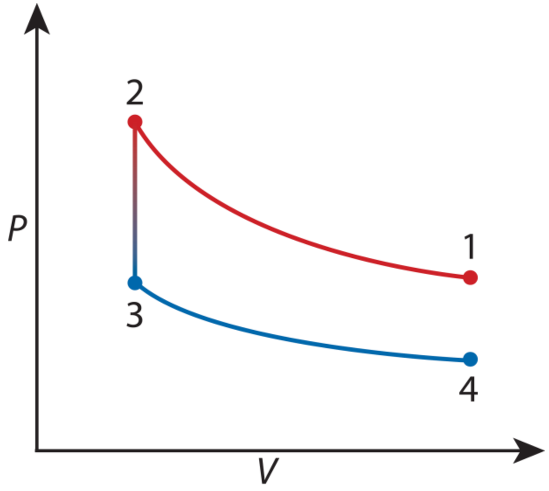
Isothermal compression

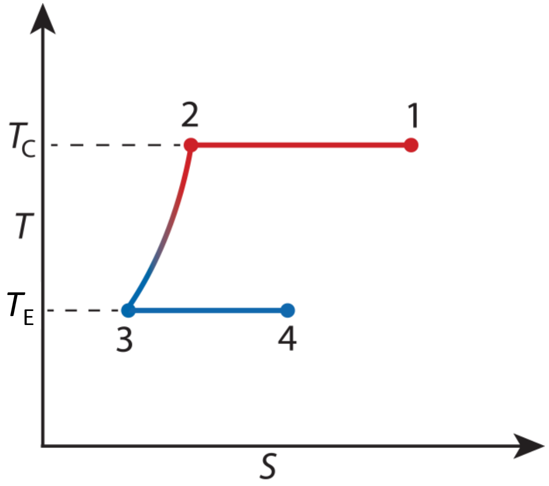


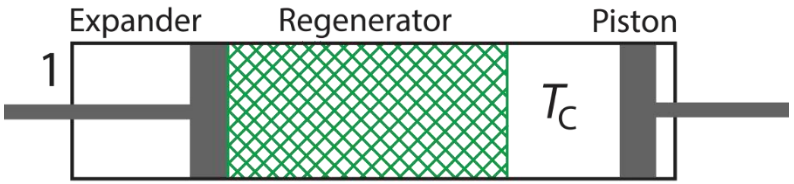
Isochoric precooling



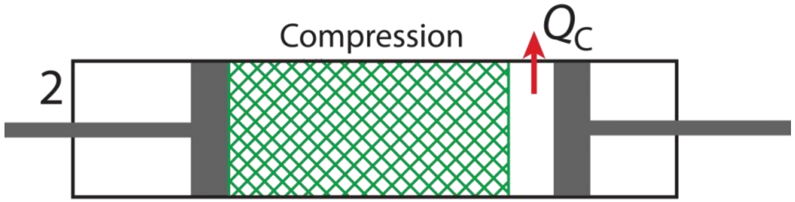
Ideal Stirling Cycle



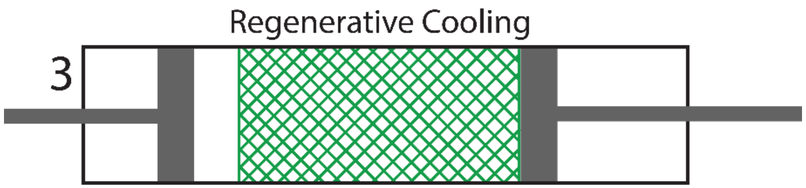




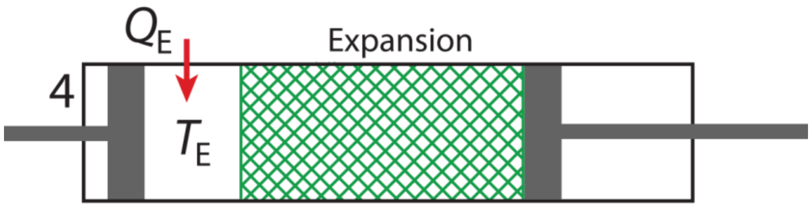
Isothermal compression



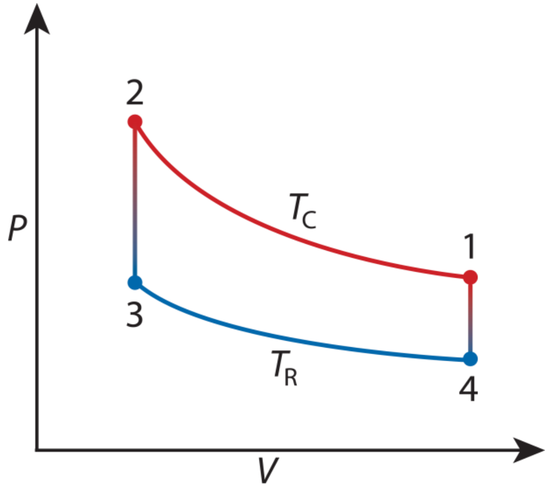
Isochoric precooling

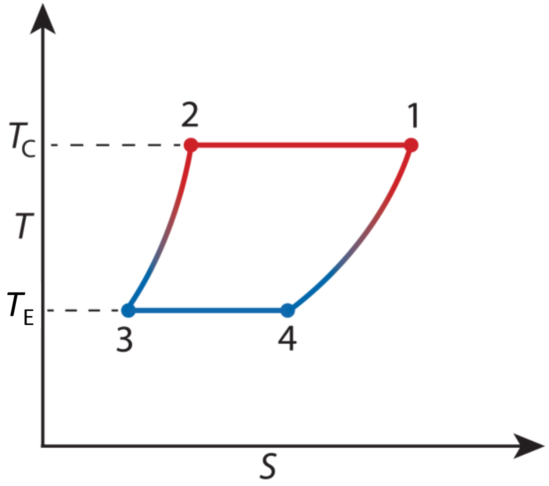


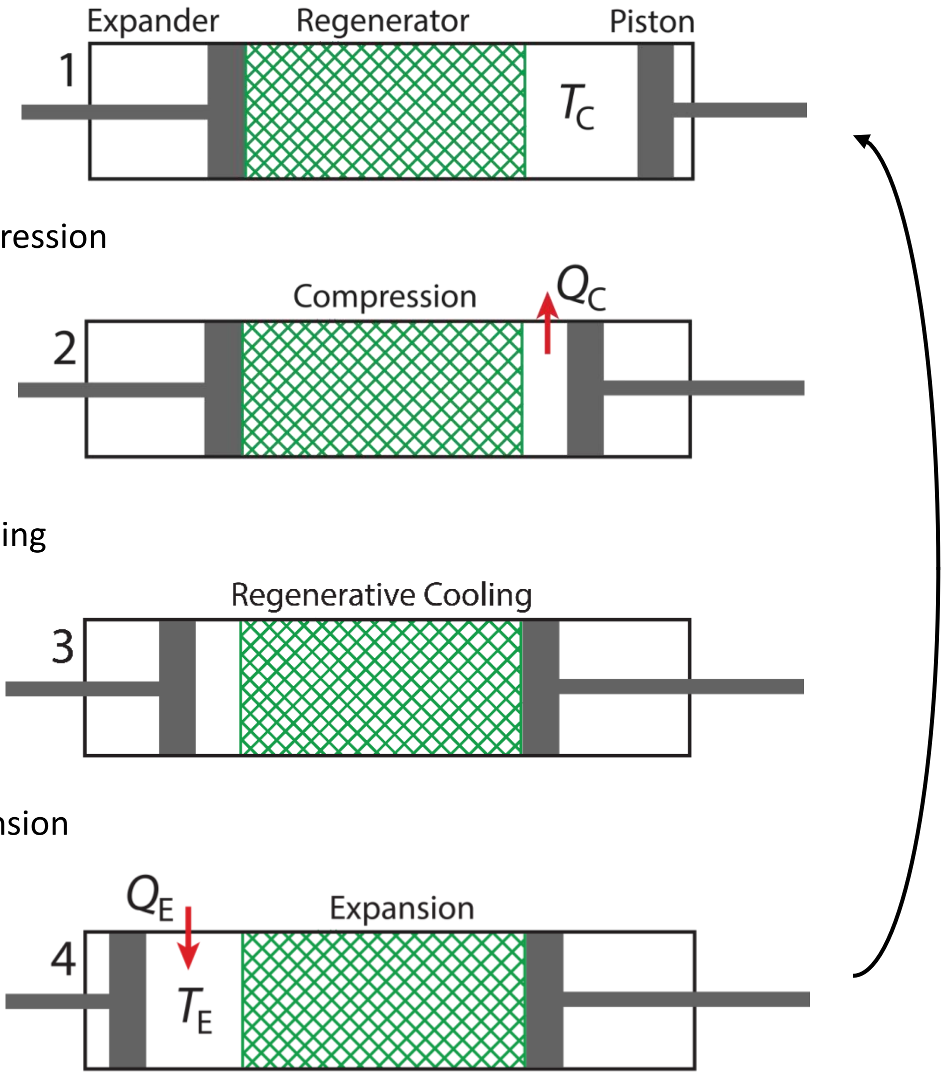
Isothermal expansion



Ideal Stirling Cycle







Isothermal compression

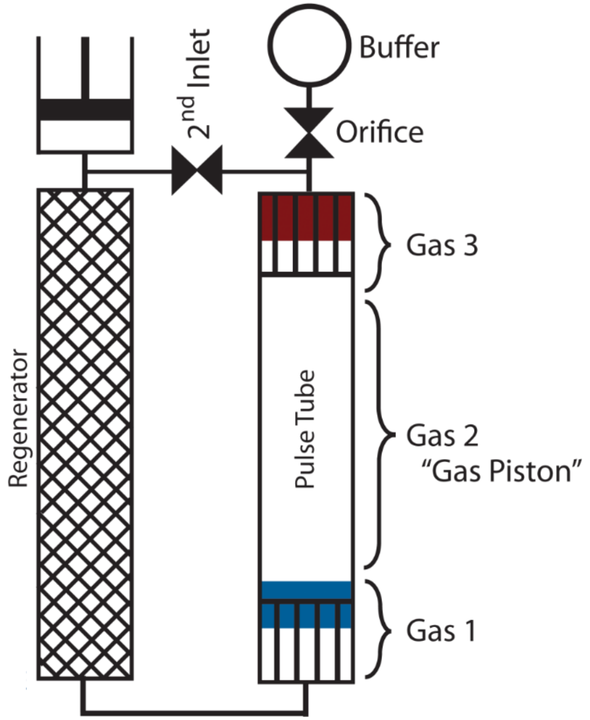
Isochoric precooling

Isothermal expansion

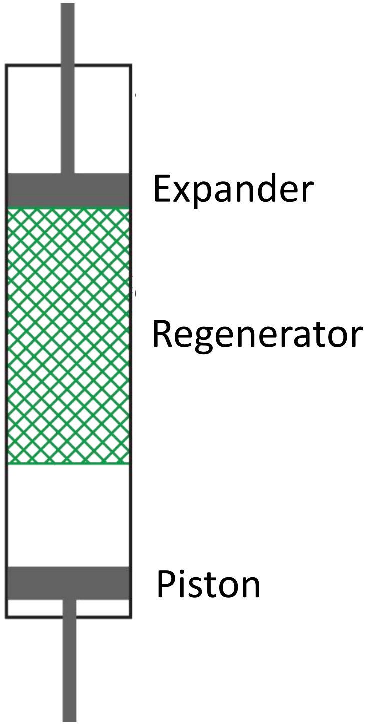
Isochoric reheating

Pulse Tube Cooler vs. Stirling

Stirling-type PTC



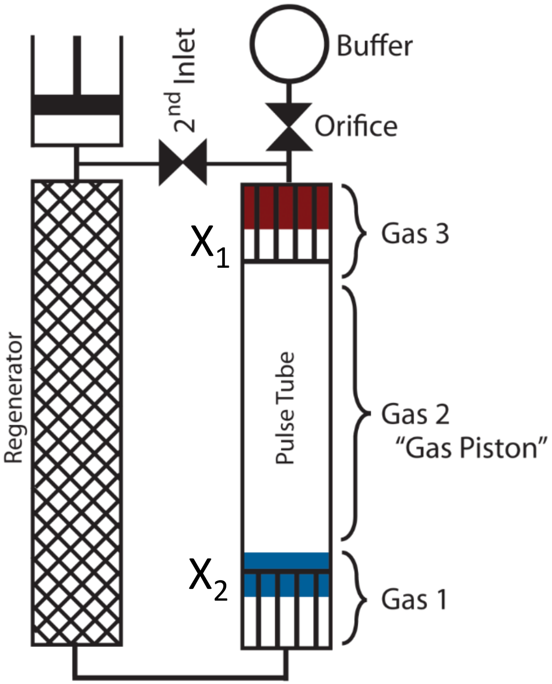
Stirling



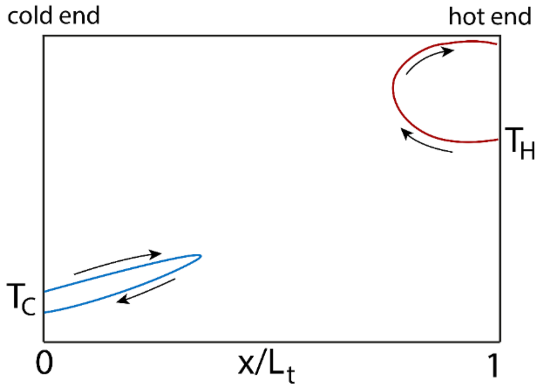
Advantages of PTC:

- No moving parts inside the cryostat (less vibrations)
- Compressor unit and tube system can be placed far away and optimized independently

Pulse Tube Cooler



Movement of gas inside pulse tube



Expansion:

- Gas 3 from buffer moves into pulse tube at X_1 with temperature T_H
- Gas 1 at cold end expands and moves back into the regenerator with temperature $T < T_C$

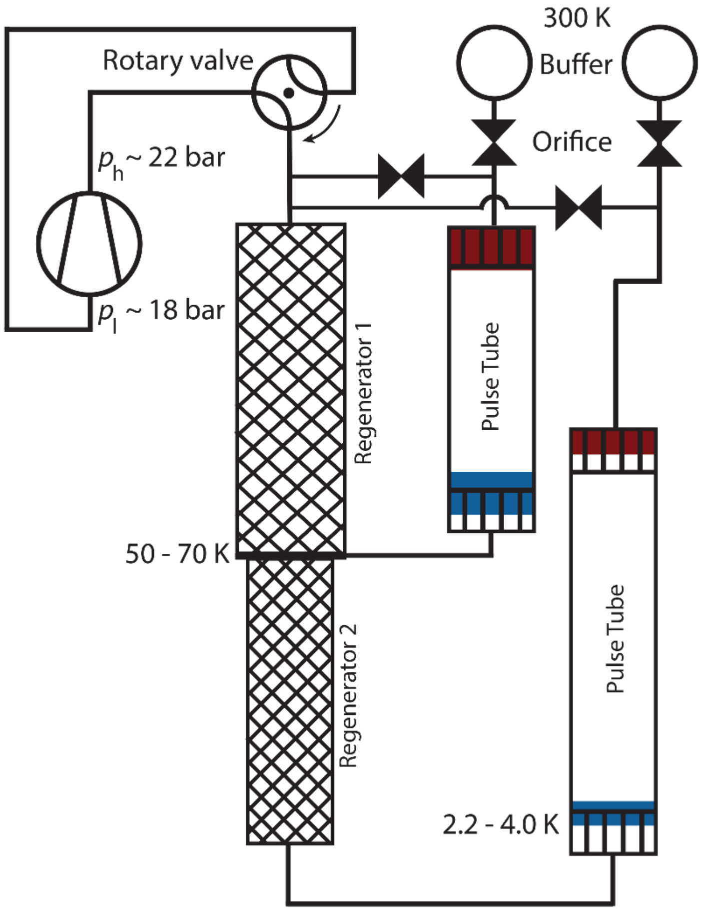
Compression:

- Gas 3 at X_1 is compressed and leaves the pulse tube with temperature $T > T_H$
- Gas 1 in the regenerator is compressed and moves into X_2 with T_C

Gas 2 functions as a displacer and insulator for Gas 1 and Gas 3 during expansion and compression

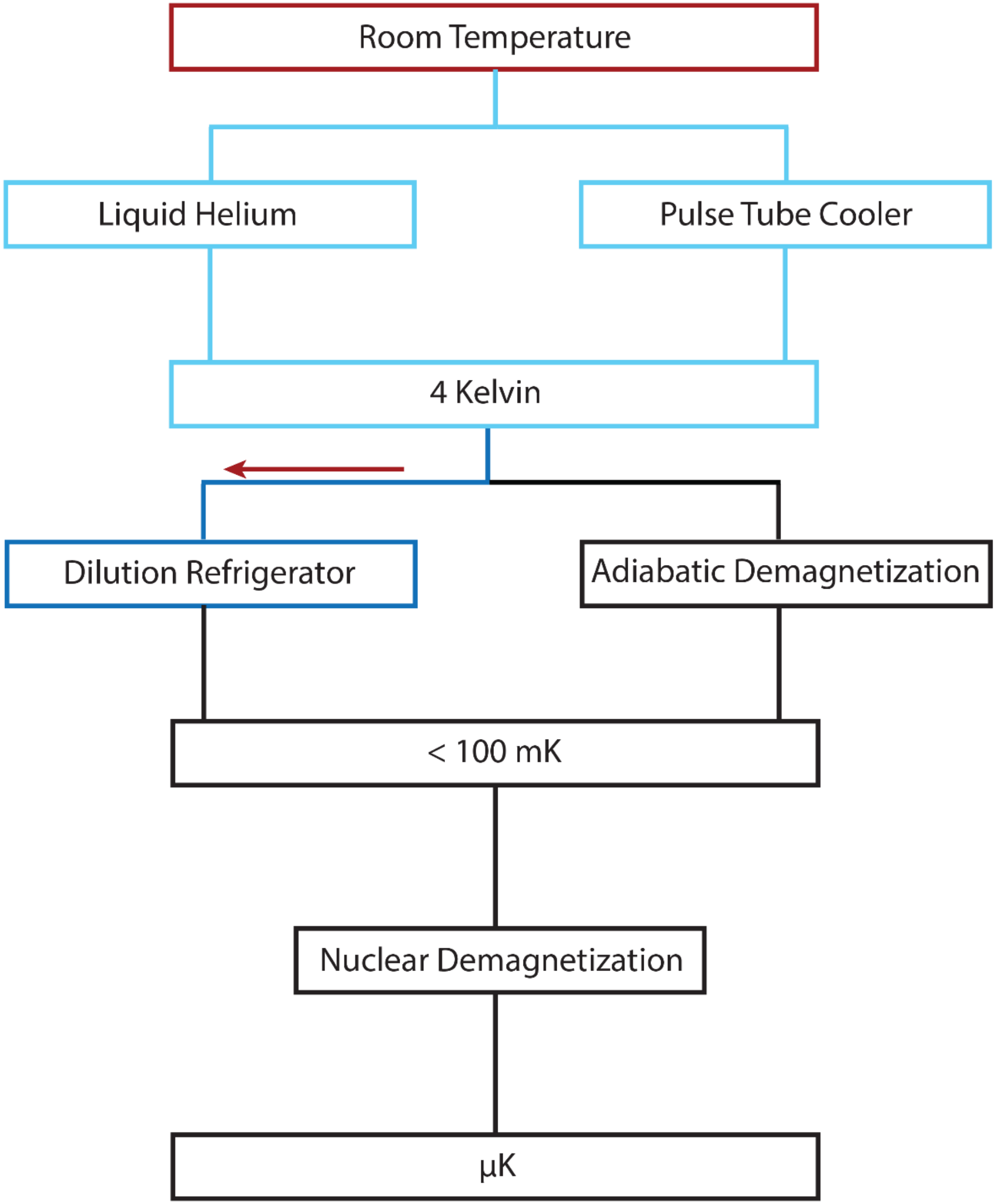
Two-Stage Pulse Tube Cooler

GM-type PTC



Pulse tube

Compressor



Room Temperature

Liquid Helium

Pulse Tube Cooler

4 Kelvin

Dilution Refrigerator

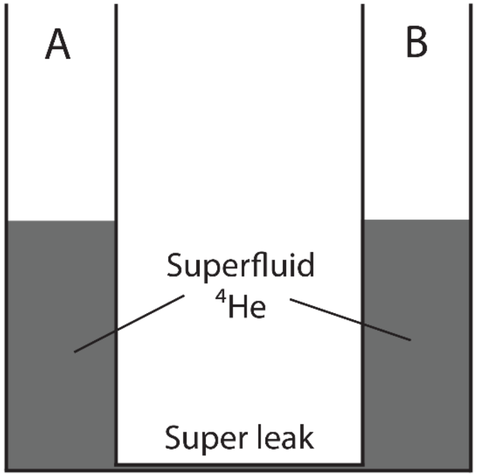
Adiabatic Demagnetization

< 100 mK

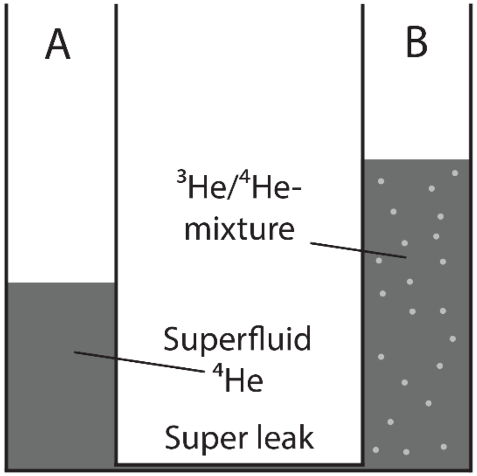
Nuclear Demagnetization

µK

Osmotic Pressure



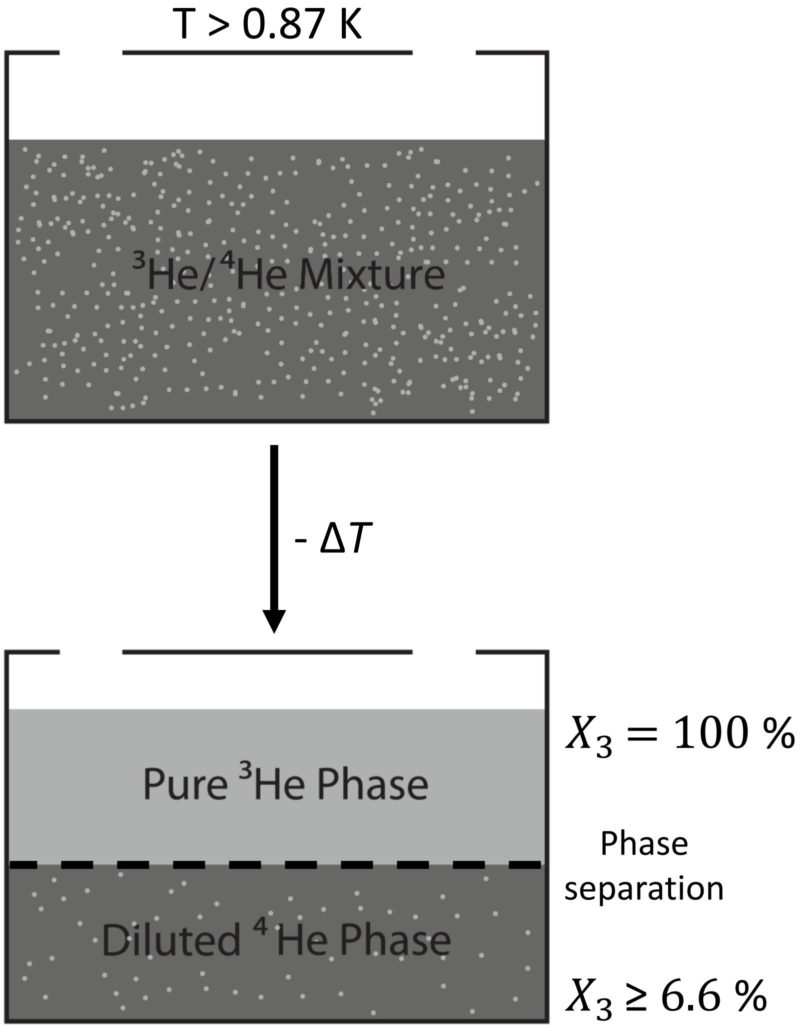
- Two columns filled with superfluid ^{4}He
- Super leak: only superfluid ^{4}He can pass through
- $T = 1 - 2$ K



- Adding ^{3}He to column B creates an osmotic pressure
- ^{3}He atoms can't pass the super leak due to their non-zero viscosity
- Superfluid ^{4}He passes from A to B trying to equalize the concentration:

$$\frac{X_{3,A}}{X_{4,A}} = \frac{X_{3,B}}{X_{4,B}}$$

Mixing ^{3}He and ^{4}He



T > 0.87 K

^{3}He/^{4}He Mixture

- ΔT

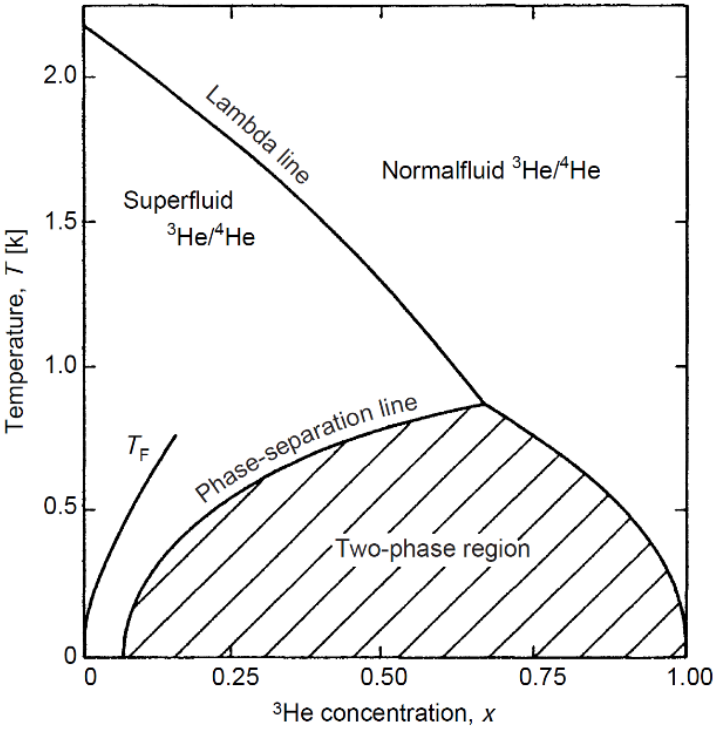
Pure ^{3}He Phase

$X_3 = 100\ \%$

Phase separation

Diluted ^{4}He Phase

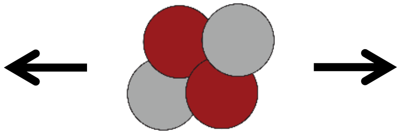
$X_3 \geq 6.6\ \%$



Dilution Refrigerator

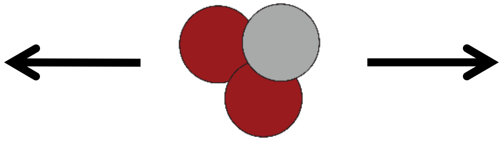
Diluted phase:

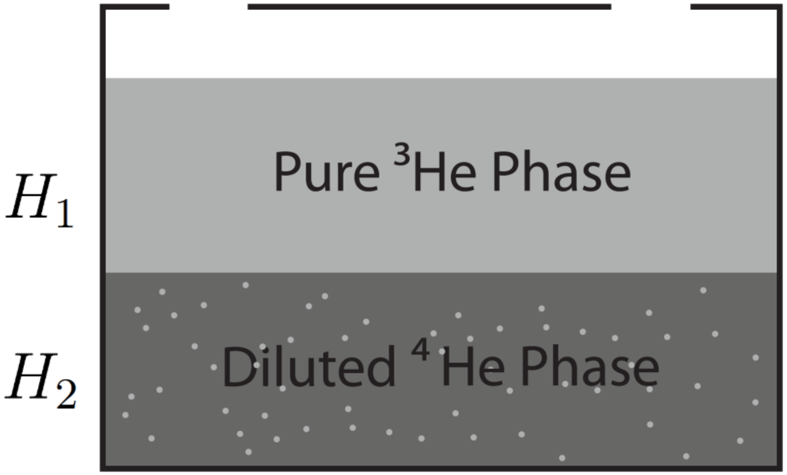
- ^{4}He behaves as a “superfluid background”
- ^{3}He can be treated as fermi gas with effective mass m*
- Zero-Point-Energy of ^{3}He higher than that of ^{4}He
- Adding ^{3}He increases kinetic energy, reducing the effective binding energy, hence limiting amount of ^{3}He solved in ^{4}He



$$m_{^4\mathrm{He}} > m_{^3\mathrm{He}}$$

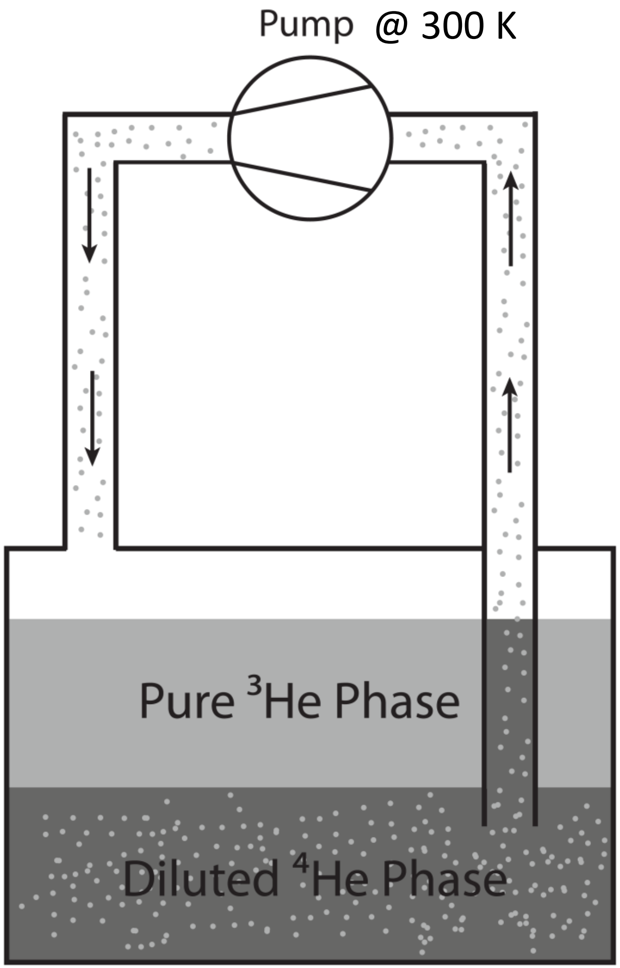
$$E_{Z,4\mathrm{He}} < E_{Z,3\mathrm{He}}$$

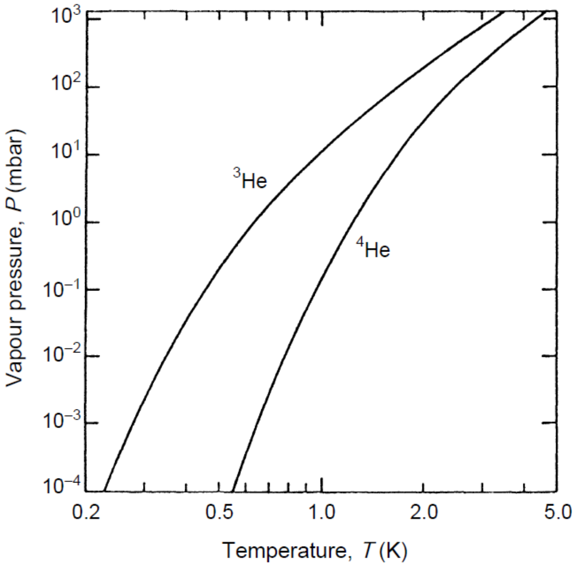




- Phases have different Enthalpy H_1 and H_2
- Removing ^{3}He from diluted phase results in ^{3}He reflow from pure phase
- ^{3}He transition into the diluted phase requires energy
- Cooling power: $\dot{Q} = \dot{n}\Delta H = \dot{n}L$

Dilution Refrigerator





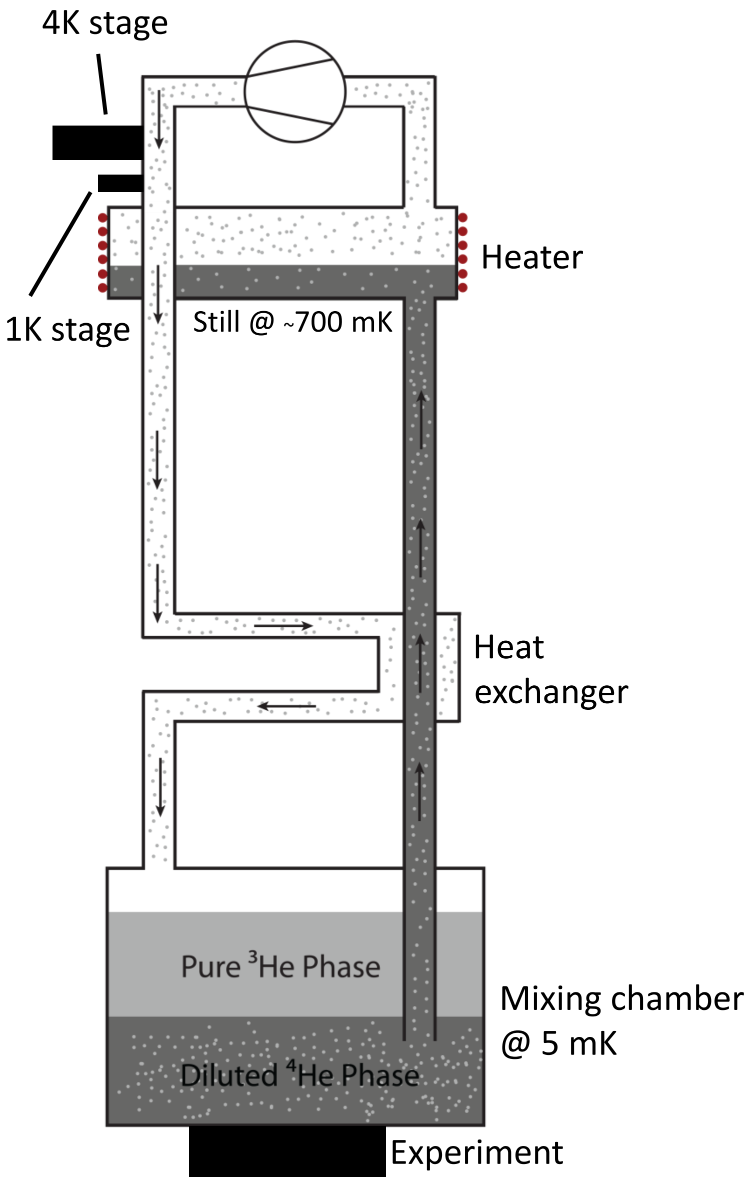
- Pumping on the diluted phase removes He particles

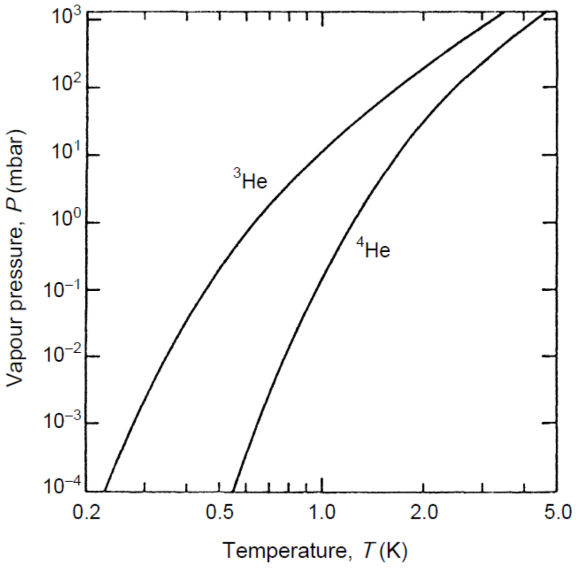
- More ^{3}He is pumped than ^{4}He due to different vapour pressures

$$p_{^3\mathrm{He}} > p_{^4\mathrm{He}}$$

- Given scenario: He flowing back into the pure phase is too warm, no relevant cooling effect is achieved

Dilution Refrigerator





- Pre-cooling pumped helium with available cooling stages
- **Still**: kept at ~ 700 mK to keep the ^{3}He vapor pressure high

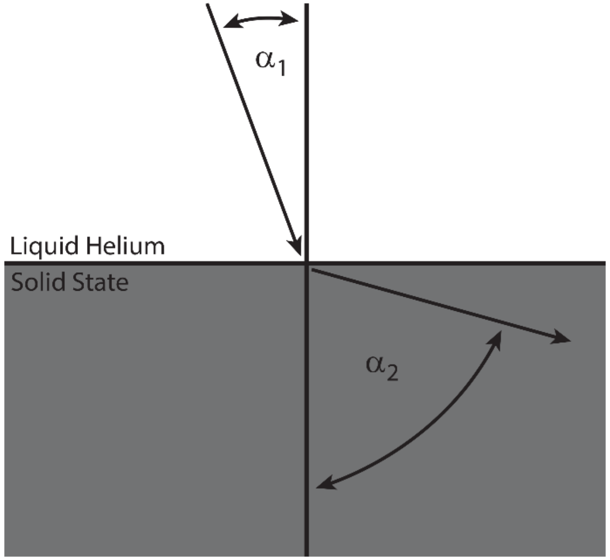
$$p_{^3\mathrm{He}} \gg p_{^4\mathrm{He}}$$

- **Heat exchanger**: crucial for the final temperature of the cryostat.
- **Cooling power**: $\dot{Q} = \dot{n}_{^3\mathrm{He}}(95\,T^2_{\mathrm{M}} - 11\,T^2_{\mathrm{H}})$

$$\frac{T_H}{T_M} = 2.8 \text{ for } \dot{Q} = 0$$

Heat Exchanger

Surface Boundary Resistance (**Kapitza-Resistance**):



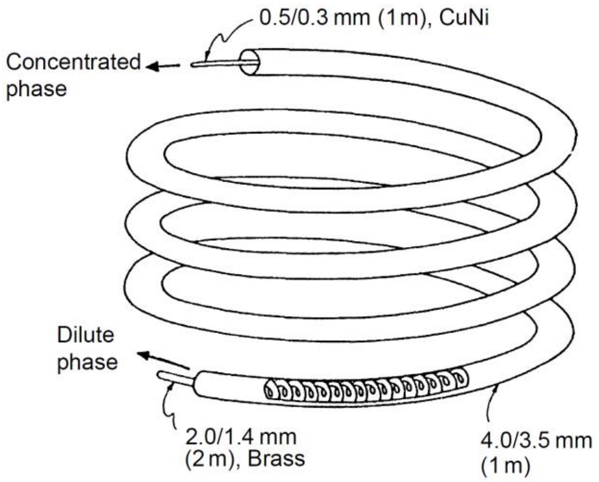
- Energy carriers (electrons or phonons) are scattered at the interface of two materials
- Total reflection for $\alpha_1 > 4°$
- Phonon transmission: $t < 10^{-3}$
- Kapitza-Resistance: $R_{\mathrm{K}} = \frac{a}{A} T^{-3}$

Heat exchanger and mixing chamber must have a huge surface area up to several 100 m^2 !

Heat Exchanger

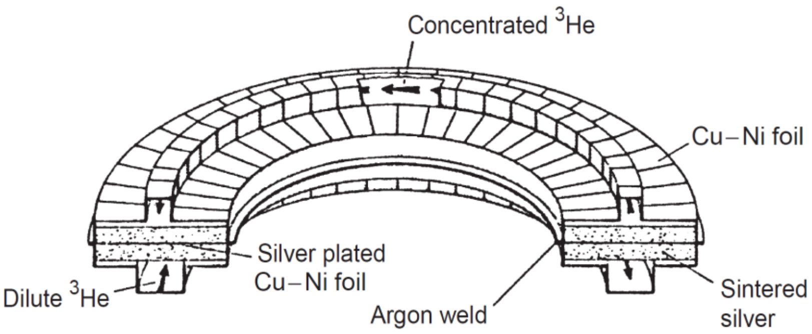
Continuous heat exchanger:

- Two concentric capillaries
- Diluted phase moves between the tubes, concentrated phase moved in inner capillary
- Does not provide enough surface area for very low temperatures
- 30 mK can be reached with a single continuous heat exchanger

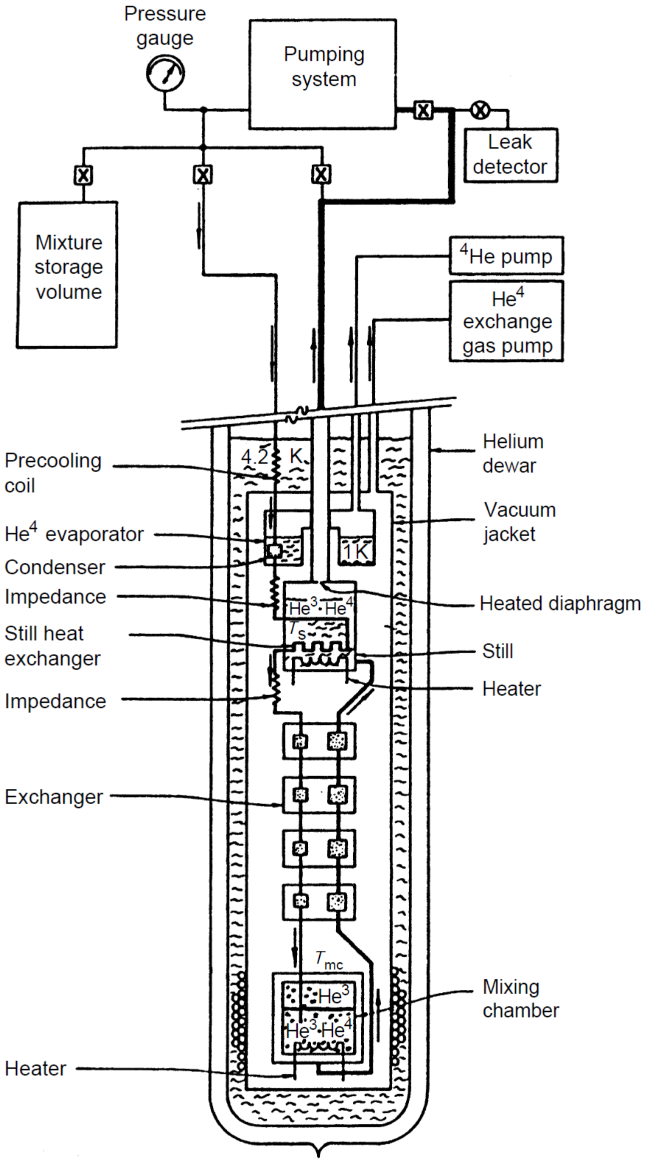


Step heat exchanger:

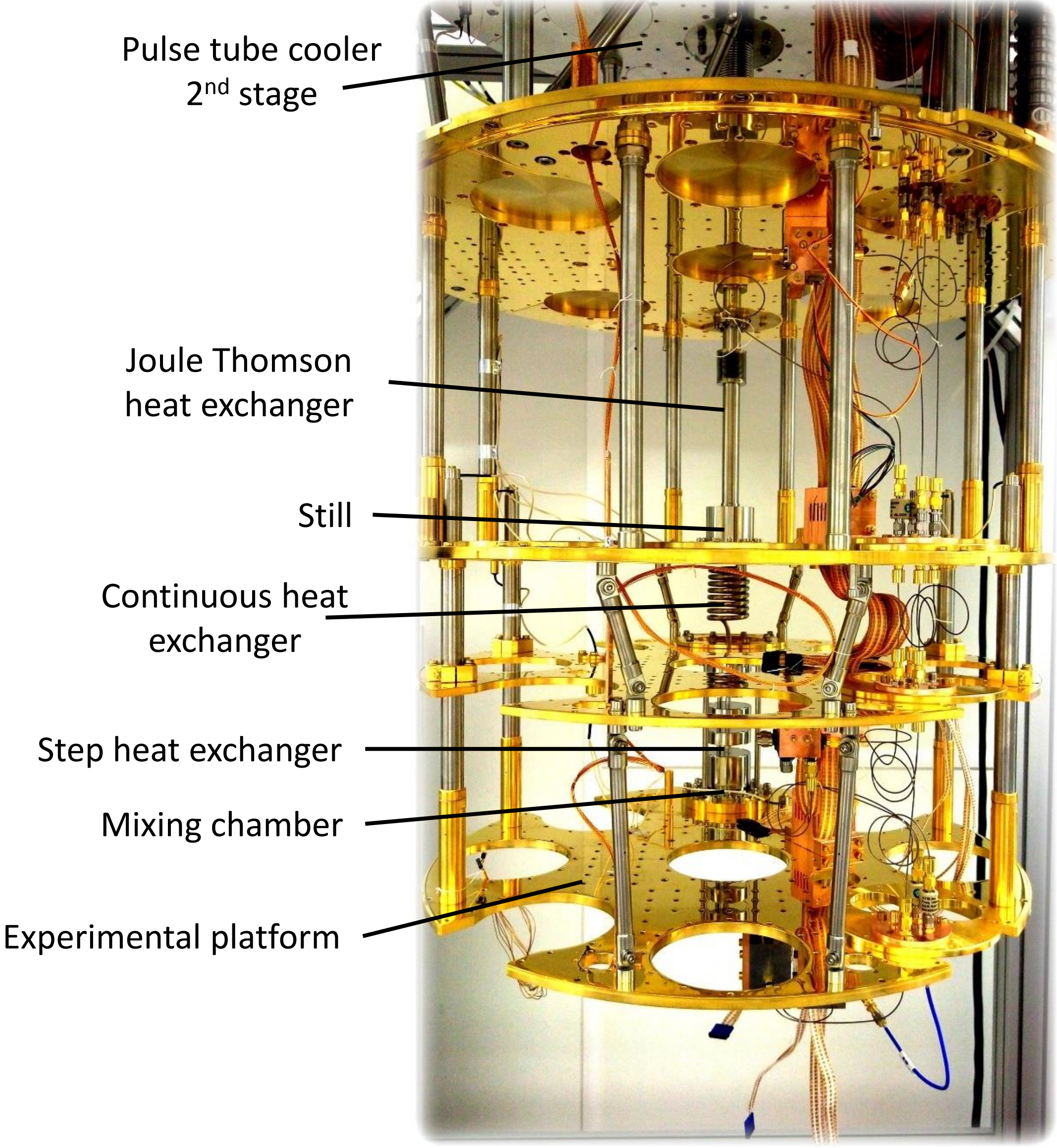
- Two metal tubes welded together
- Filled with sintered metal powder to create a huge surface area
- “step” because several are connected in series, each having different geometrical and thermodynamic properties optimized for a certain temperature
- 4 mK are usually reached using several step heat exchangers in series

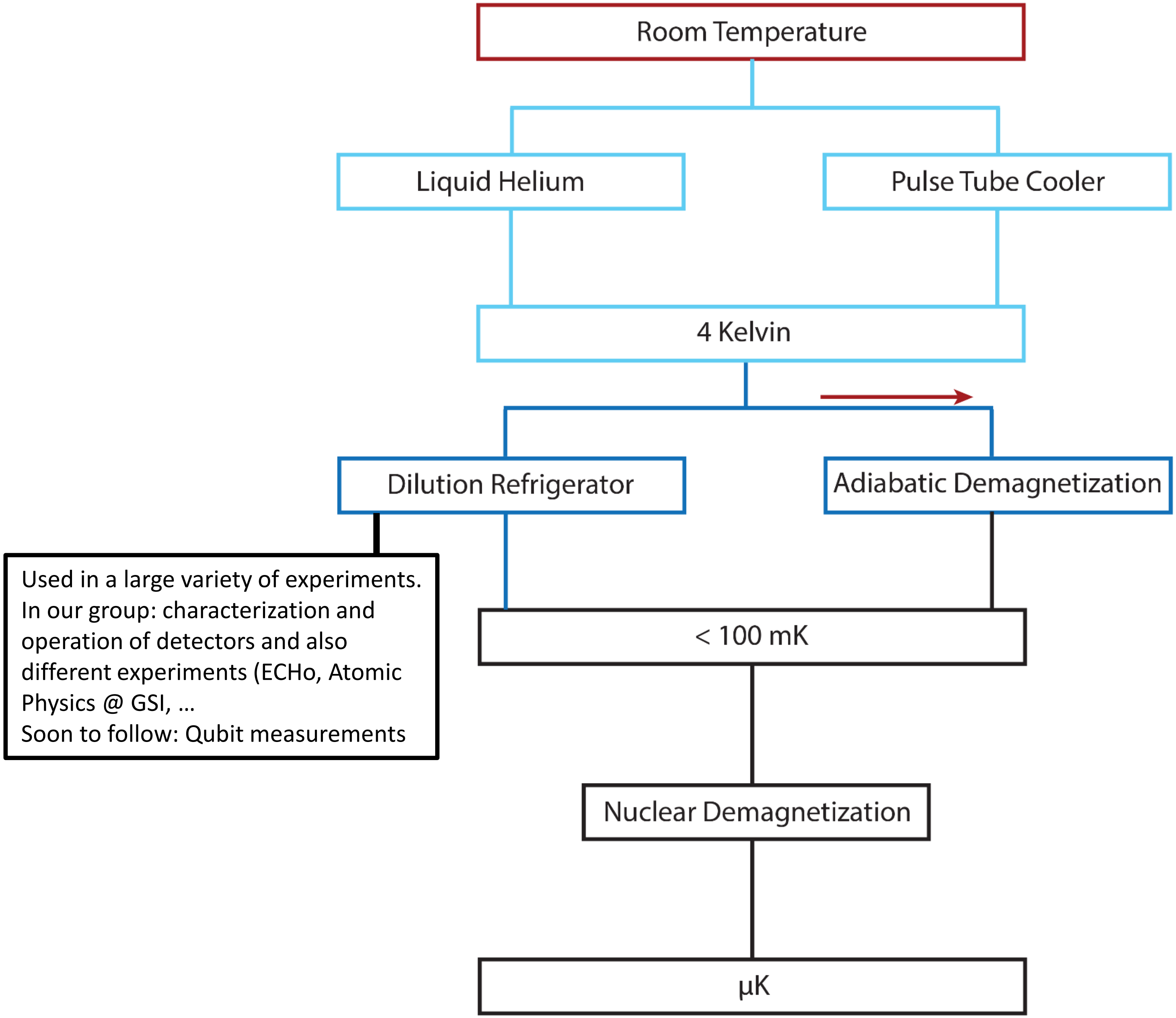


Dilution Refrigerator



ECHo Cryostat





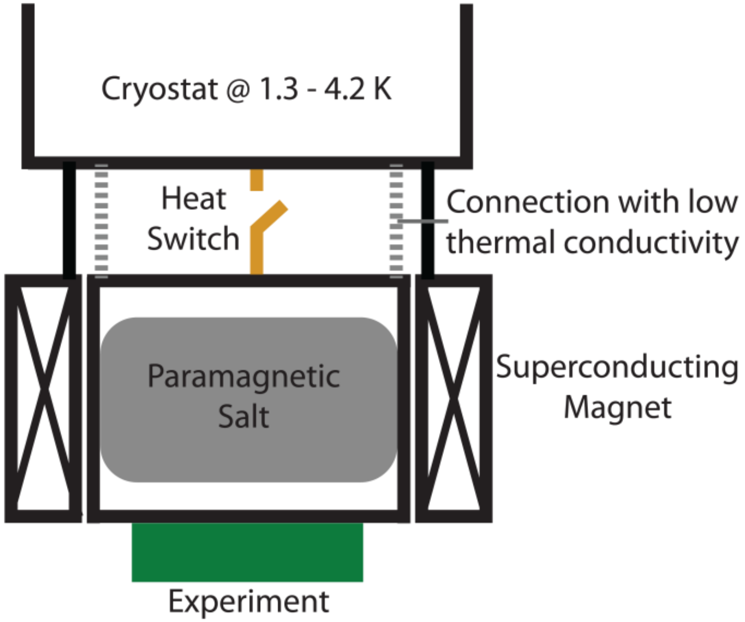
Used in a large variety of experiments.
In our group: characterization and operation of detectors and also different experiments (ECHo, Atomic Physics @ GSI, …
Soon to follow: Qubit measurements

Adiabatic Demagnetization Refrigerator

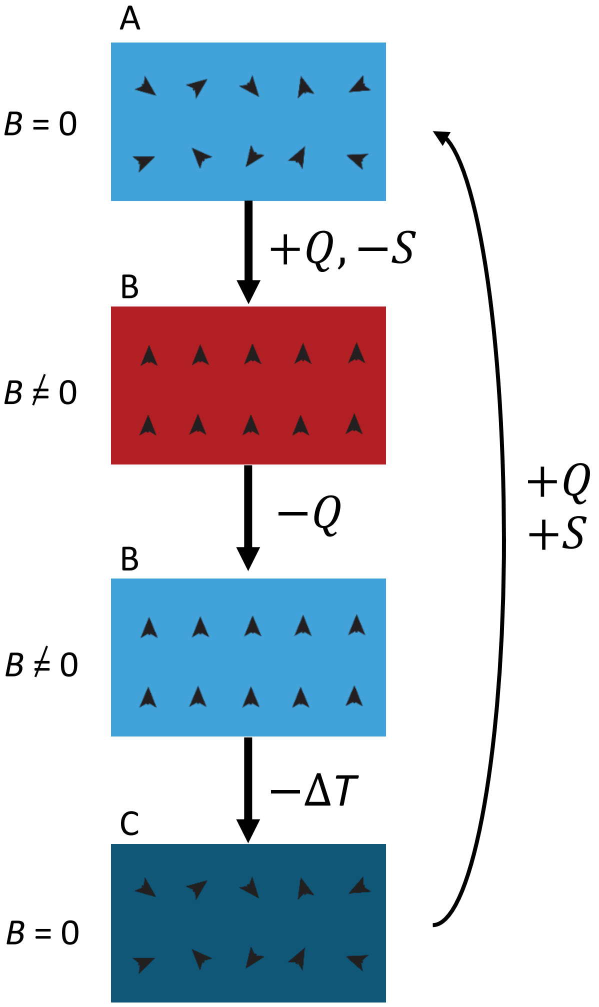
- System pre-cooled by LHe or PTC
- Paramagnetic material with unpaired electrons in shell
- Each spin carries magnetic moment:

$$\boldsymbol{\mu} = -g\mu_B\boldsymbol{S}$$

- Material has to be an insulator because conduction electrons influence the magnetic moments
- Paramagnetic salt in good or bad thermal contact with heat bath (depending on HS open or closed)
- B-Field created by a superconducting magnet (B > 5 T)



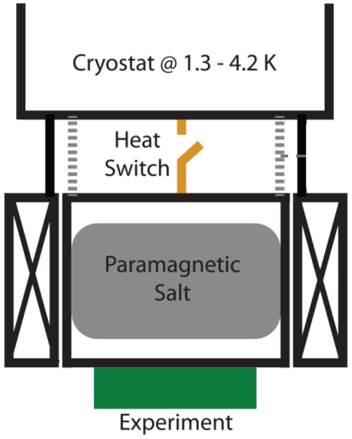
Cooling Principle

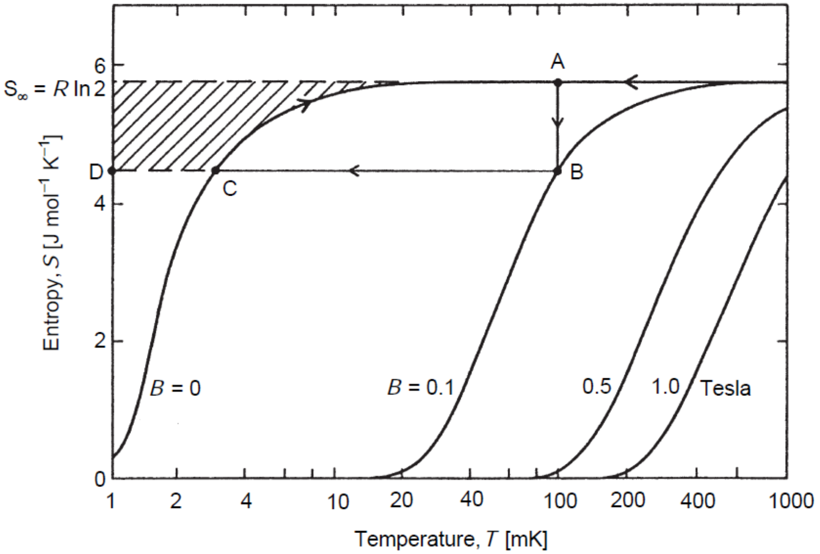


- Heat of magnetization is absorbed by ^{4}He or PTC:

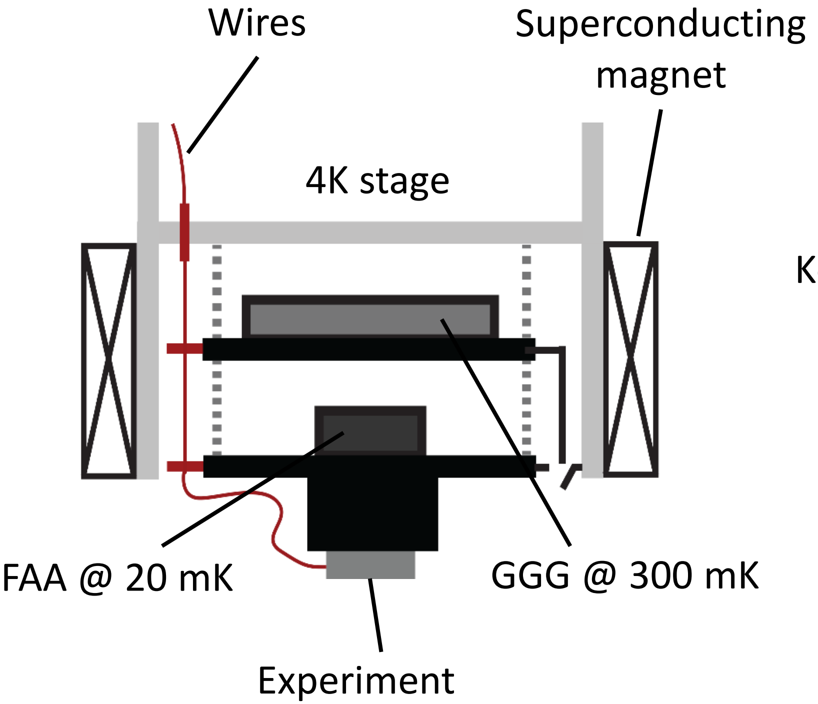
$$Q(T_\mathrm{i}) = nT_\mathrm{i}[S(0, T_\mathrm{i}) - S(B_\mathrm{i}, T_\mathrm{i})]$$

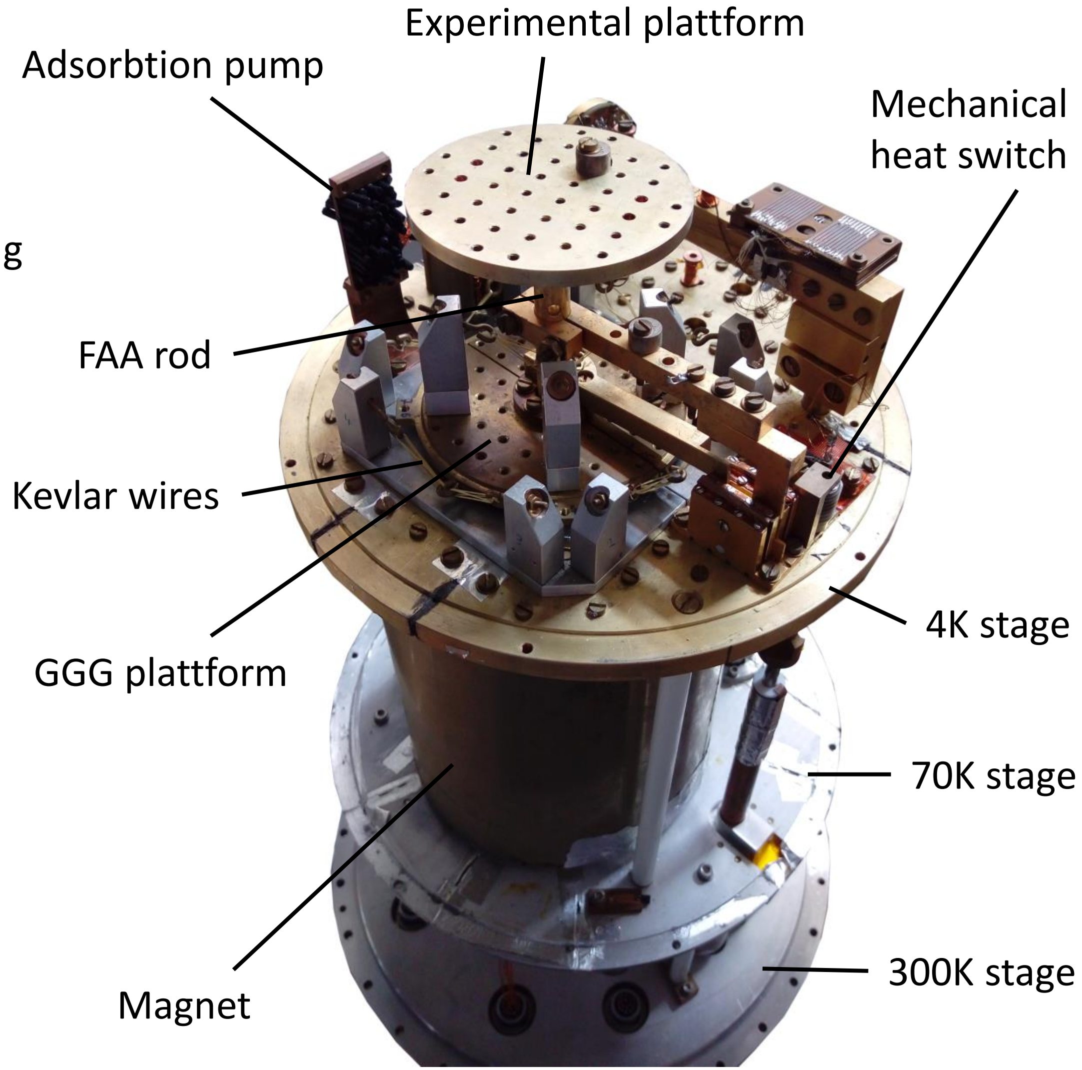
- Ordering temperature limits the lowest temperature to be reached

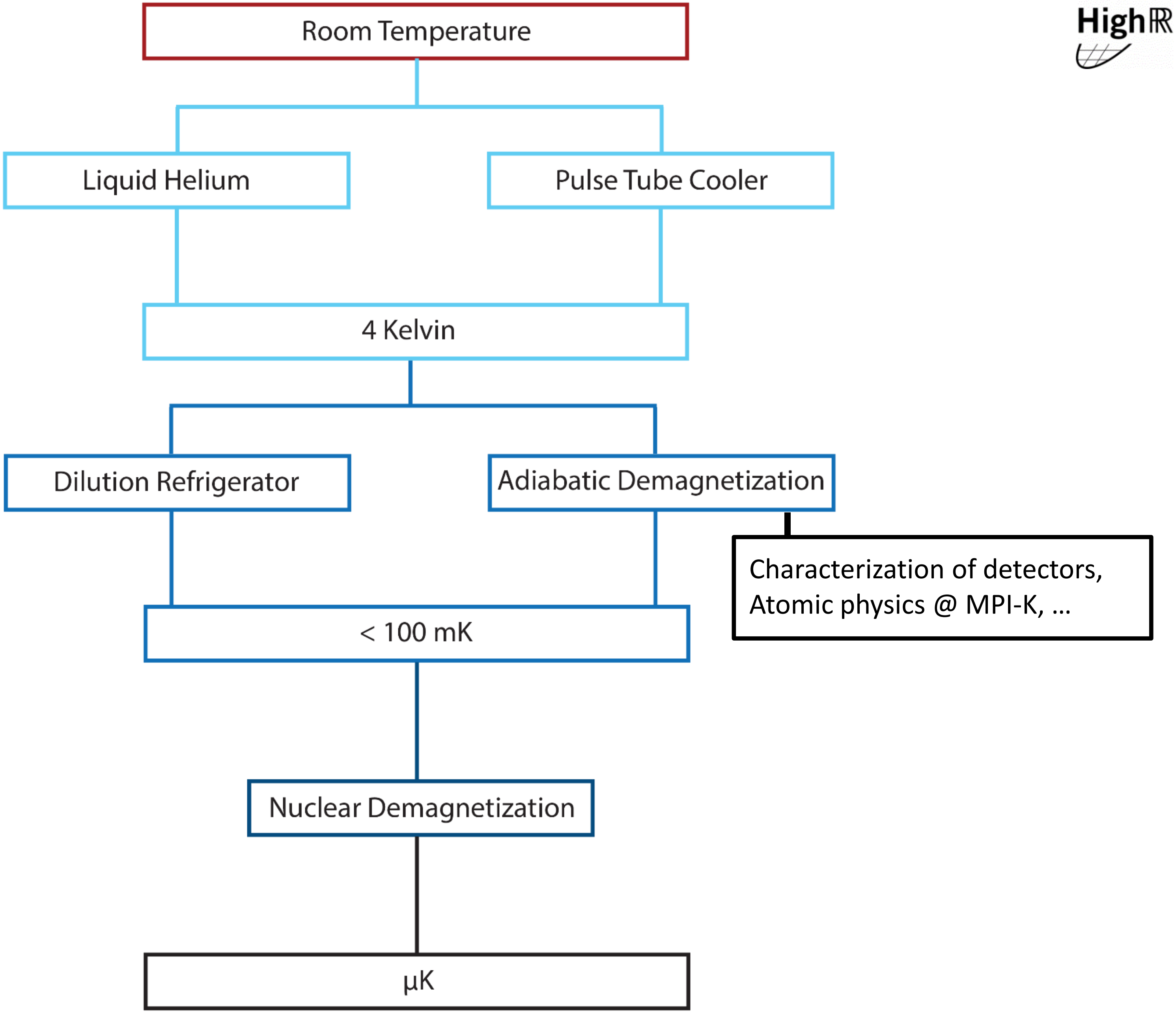




Two-stage ADR







Room Temperature

Liquid Helium

Pulse Tube Cooler

4 Kelvin

Dilution Refrigerator

Adiabatic Demagnetization

Characterization of detectors,
Atomic physics @ MPI-K, …

< 100 mK

Nuclear Demagnetization

µK

Nuclear Demagnetization

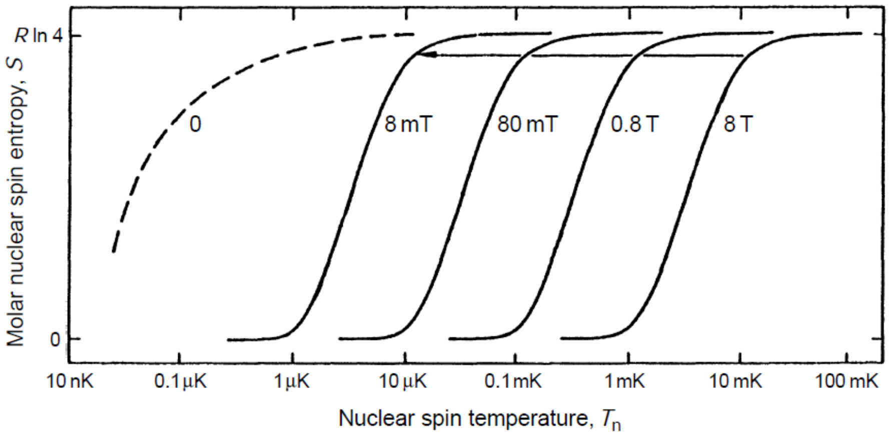
- Working principle similar to the electronic ADR
- Take material with ***S*** = 0 and ***I*** ≠ 0
- Magnetic moment, electron vs. nucleus:

$$\mu_\mathrm{B} = 9.27 \times 10^{-24}\,\mathrm{J\,T^{-1}}$$
$$\mu_\mathrm{n} = 5.05 \times 10^{-27}\,\mathrm{J\,T^{-1}}$$

- Small magnetic moment provides **advantages**:
  - Lower ordering temperature of spins, $T \sim 0.1\ \mu\mathrm{K}$
  - Material can (must) be a metal providing high thermal conductivity
  - Magnetic moment density larger in pure metals than in diluted paramagnetic salts

- **Drawback**: smaller magnetic moment also means a smaller “reaction” to external magnetic fields

$$\Delta S_\mathrm{n} \sim \frac{\Delta S_\mathrm{e}}{1000}$$

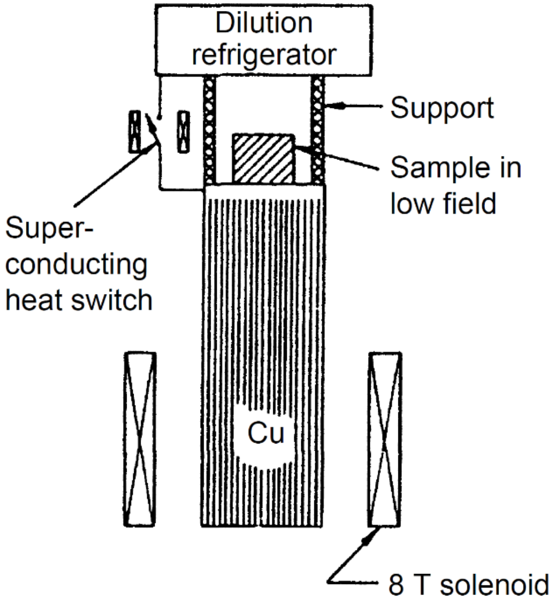


Nuclear Demagnetization

Finding the right material with requirements:

- Normal conducting metal
- High thermal conductivity
- Low spin ordering temperature
- Has no ordered electron spins
- Easy to fabricate with high purity

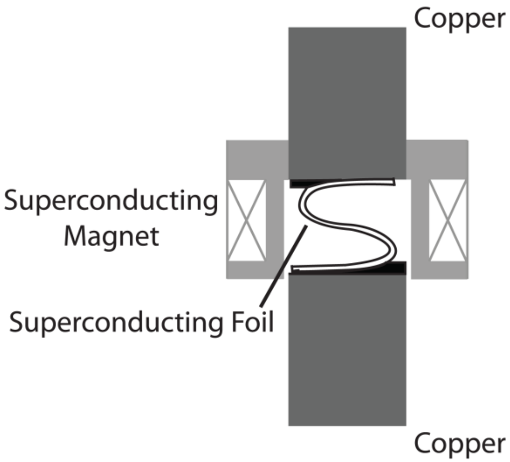
→ Copper or Platinum



Superconducting heat switch

- Very low thermal conductivity in SC state (conduction dominated by phonons)
- Thin aluminium foil shaped like an S
  - Small mean free path of phonons
  - No normal conducting path after $B \rightarrow 0$

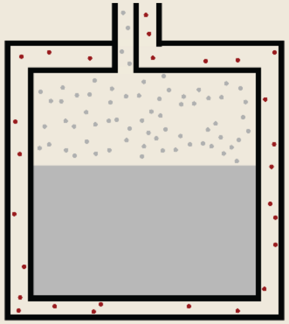
Heat switch



Heat Leaks in NDR

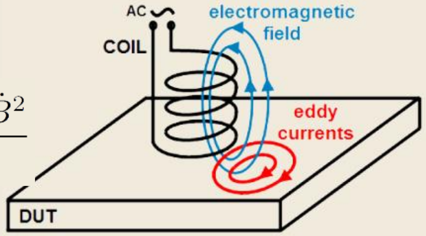
External heat leaks:

- Conduction (mountings, wires, heat switch, …)
- Convection (residual gas)
- Radiation (also by radioactive sources)



Eddy currents:

- B-field influences electrons
- Vibrations inside B-field
- Changing B-field
- Heat input: $\dot{Q}_e = \dfrac{G V \dot{B}^2}{\rho}$



Time dependent leaks:

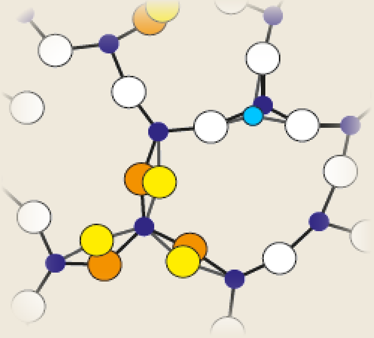
- Hydrogen bubbles found in certain metals
- Ortho-Para conversion of hydrogen releases energy

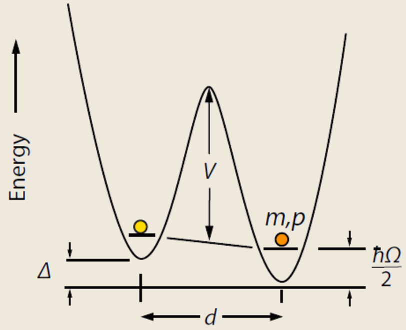
Ortho hydrogen

Para hydrogen

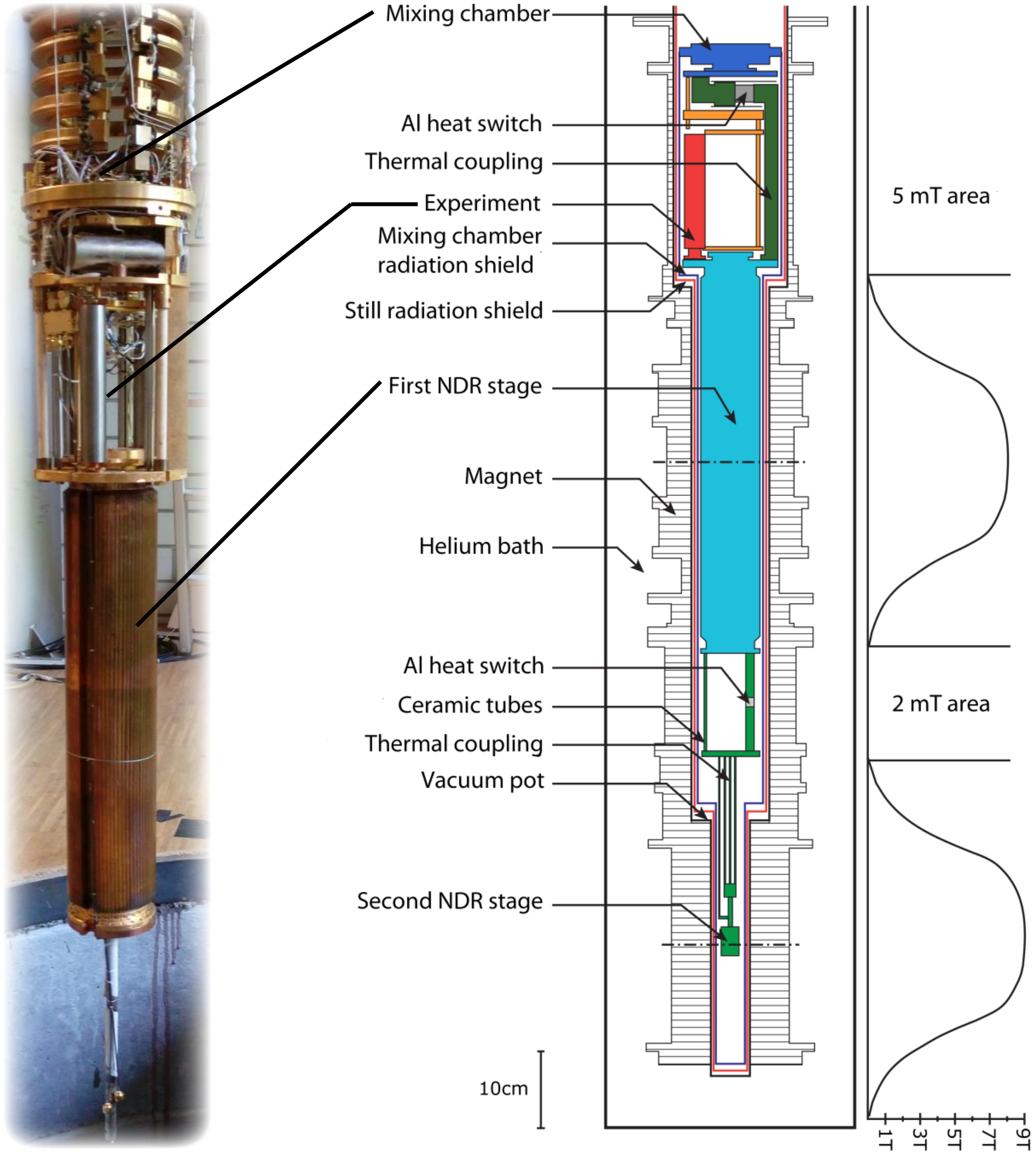
Internal leaks:

Tunneling systems





Nuclear Demagnetization

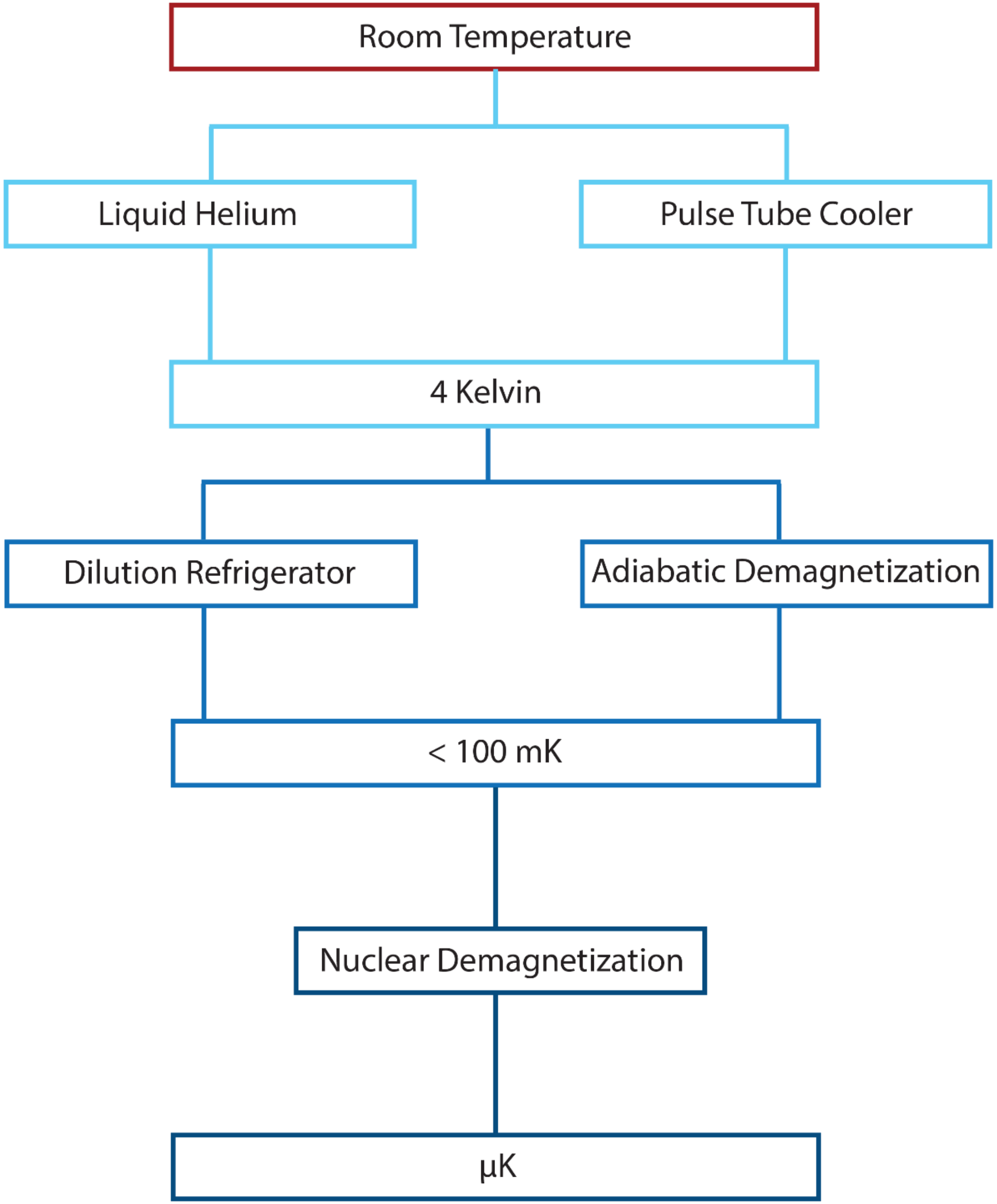


Experiments:

- Thermometry
- Search for superconductors
- Investigation of materials
  - ^{3}He
  - Glasses
  - Quantum magnets

Specific examples:

- Noise Thermometer
- Novel fractional quantum Hall States in two-dimensional electron systems
- Gravity waves on a surface of topological superfluid ^{3}He-B
- Superconductivity in Polycrystalline Boron-Doped Diamond



Room Temperature

Liquid Helium

Pulse Tube Cooler

4 Kelvin

Dilution Refrigerator

Adiabatic Demagnetization

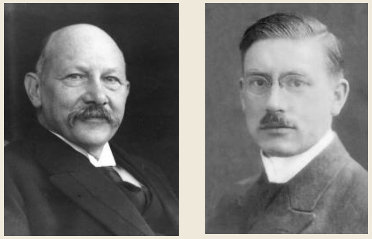
< 100 mK

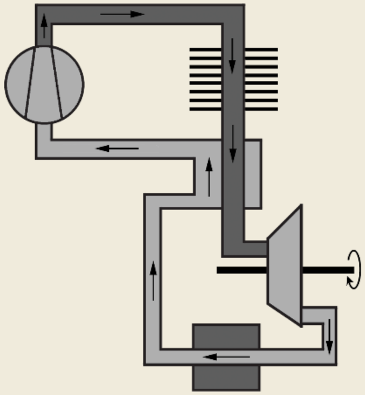
Nuclear Demagnetization

µK

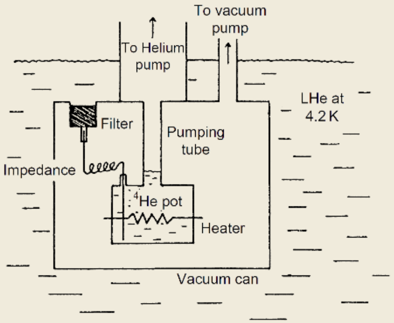
Summary

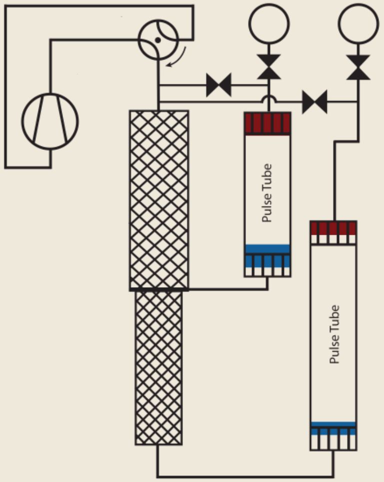
Historic Introduction & Cryoliquids



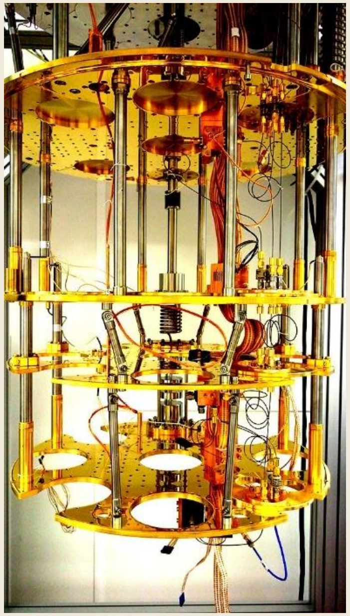


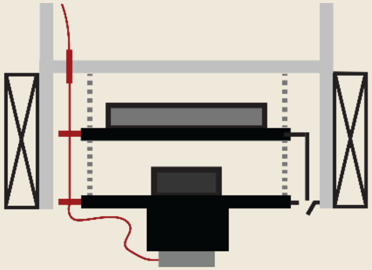
^{4}He Cryostat & Pulse Tube Cooler



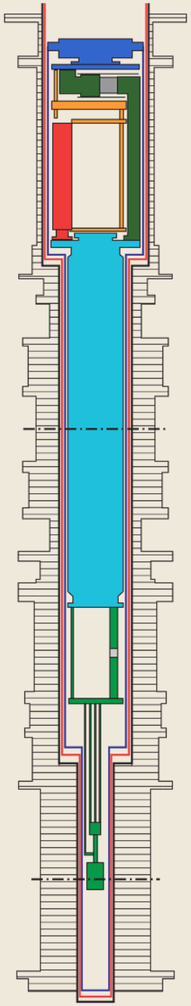


Dilution Refrigerator & ADR



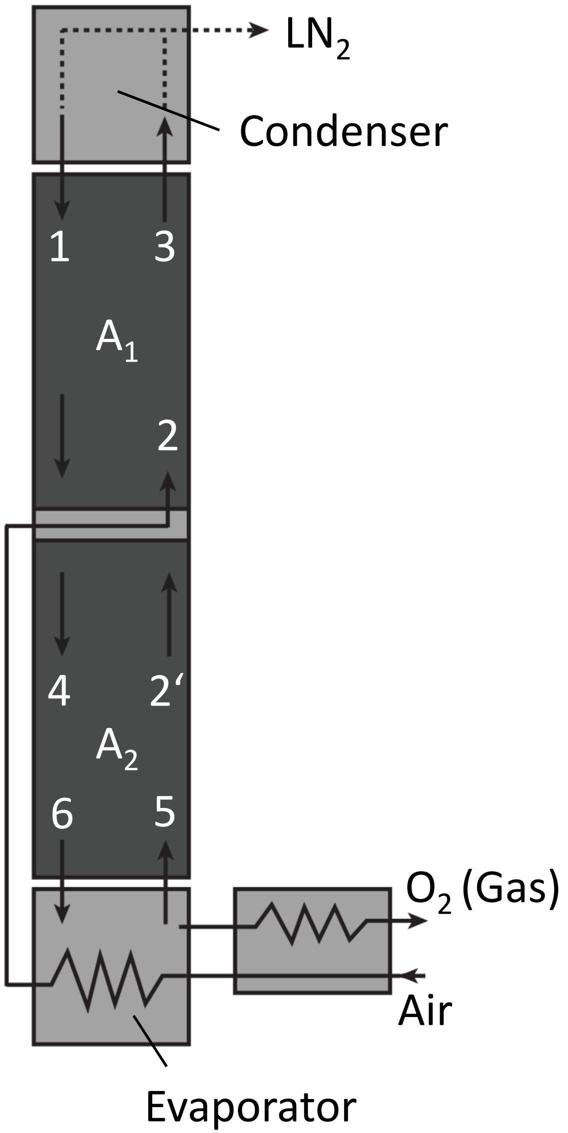


Nuclear Demagnetization



Thank you for your attention!

Cryoliquid LN_2 @ $T = 77$ K



Separation of air into nitrogen and oxygen

1. LN_2 (~77 K) trickles from condensor into volume A_1
2. Pre-cooled air (~ 85 K) enters A_1 from below and mixes with LN_2
3. Nearly pure nitrogen (~ 99.5 %) reaches the condenser, part of it gets removed while the rest goes to 1
4. Droplets reaching A_2 consist of 50 % nitrogren and 50 % oxygen
5. Oxygen coming from the evaporator meets droplets from 4 which in turn release their nitrogren
6. Oxygen dropletts move back to the evaporator where they get evaporated again, part of the oxygen is removed

Cooldown with Cryoliquids

Cooldown with liquid helium:

Heat of evaporation @ 4.2 K:

$$L_{4\mathrm{He}} = 2.6\ \mathrm{kJ}\ /\mathrm{l}$$

Enthalpie from 4.2 K to 300 (77) K :

$$\Delta H_{4\mathrm{He}} = 200\ (64)\mathrm{kJ}\ /\mathrm{l}$$

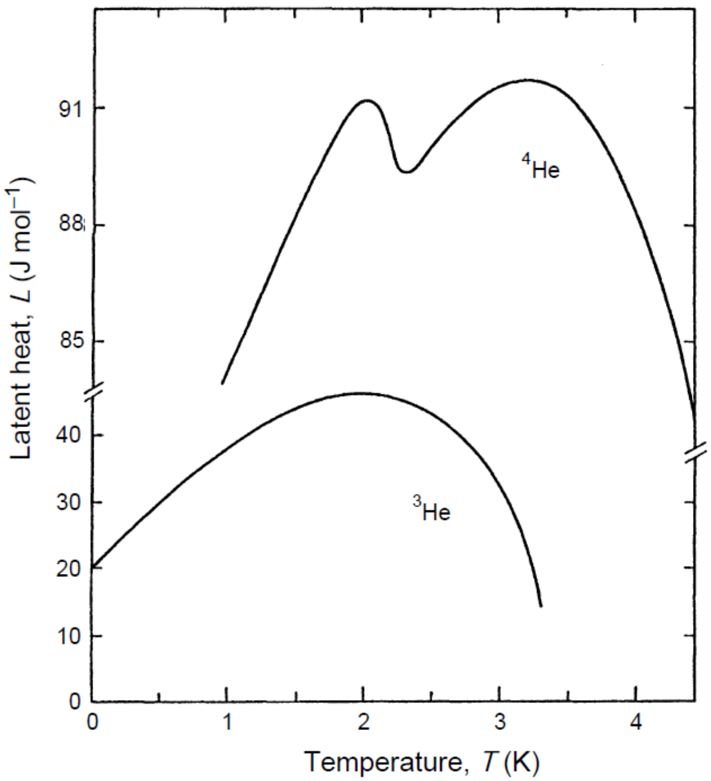
Amount of cryoliquid [*l*] necessary to refrigerate 1 kg of aluminum, stainless steel (SS) or copper.

cryoliquid	temperature change [K]	Al	SS	Cu
N_2	300 → 77	1.0 (0.63)	0.53 (0.33)	0.46 (0.28)
^{4}He	77 → 4.2	3.2 (0.20)	1.4 (0.10)	2.2 (0.16)
^{4}He	300 → 4.2	66 (1.6)	34 (0.8)	32 (0.8)

→

- Pre-cooling of equipment with LN_2 to 77 K
- Use liquid ^{4}He to go from 77 K to 4.2 K
- Use the enthalpy of the helium gas, hence it should leave the cryostat as warm as possible

Latent Heat of Evaporation



$$L_{N2} = 5570\ \mathrm{J\ mol^{-1}}$$

- Latent heat: energy needed for the phase transition from liquid to gas
- *L* of helium very small compared to other liquids
- Very good shielding against external parasitic heat input needed
- Heat load from:

Conduction: Phonons $\dot{Q} = \frac{Ab}{4L}(T_2^4 - T_1^4)$

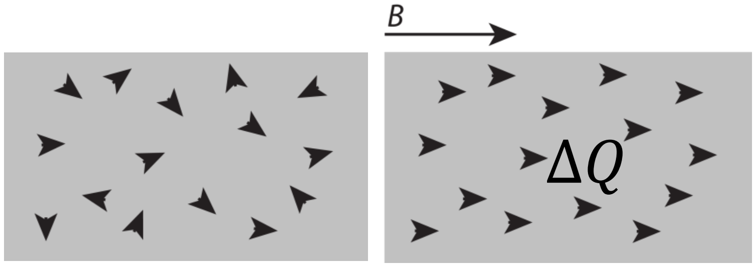
Electrons $\dot{Q} = \frac{A\kappa_0}{2L}(T_2^2 - T_1^2)$

Radiation: $\dot{Q}[\mathrm{W}] = 5.67 \times 10^{-12} A[\mathrm{cm}^2](T_1^4 - T_2^4)$

Gas particles: $\dot{Q}[\mathrm{W}] \approx 0.02 a A[\mathrm{cm}^2] P[\mathrm{mbar}](T_2 - T_1)[\mathrm{K}]$

Adiabatic Demagnetization

Paramagnetism:

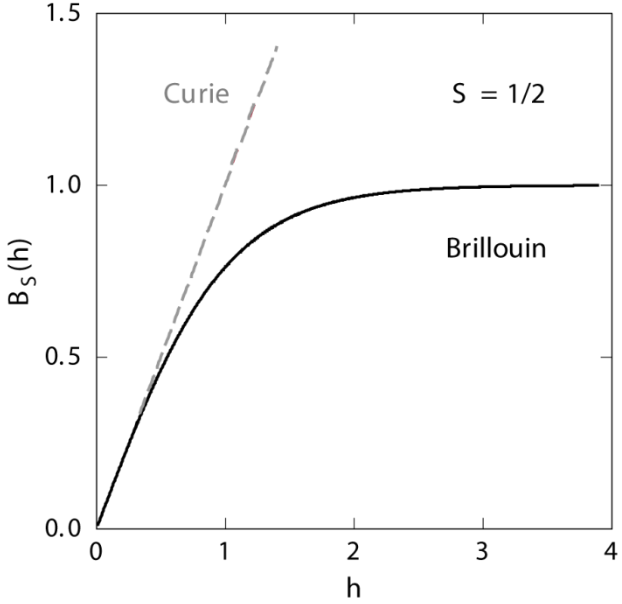


- Ions with unpaired electrons in shell carry magnetic moment:

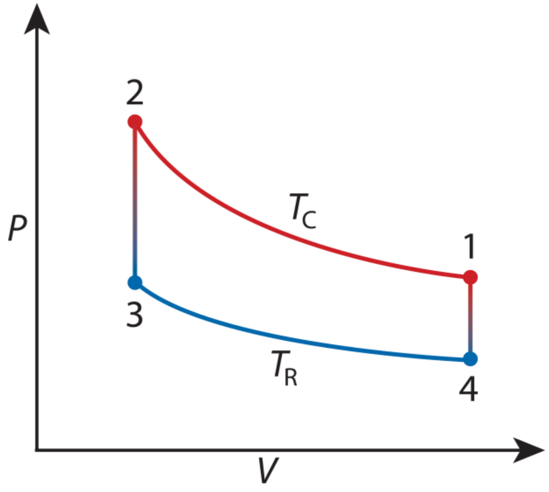
$$\boldsymbol{\mu} = -g\mu_B \boldsymbol{J}$$

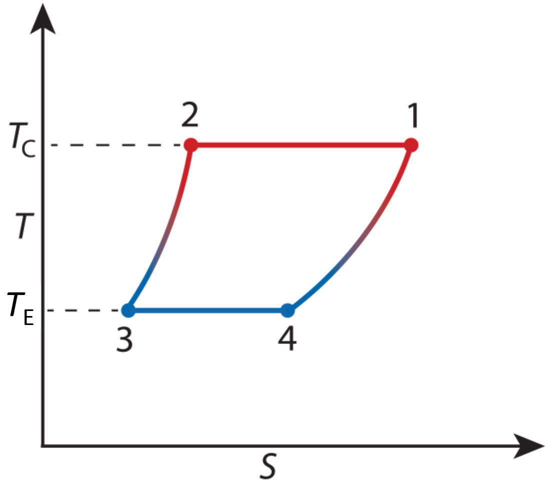
- Spins align along magnetic field lines of external origin
- Suszeptibility: $\chi = \frac{\boldsymbol{M}}{\boldsymbol{H}} > 1$
- Entropy: $S = \mathrm{R}\ln(2J+1)$
- Magnetization: $M = ng\mu_B J\mathcal{B}(h)$
- For $h < 1$, Curie Law: $\chi = \frac{M}{H} = \frac{C}{T}$

Brillouin function



Non-ideal Stirling Cycle





How is a real machine different from the ideal?

- Harmonic motion of pistons (instead of abrupt)
- Void volume in regenerator
- Regenerator ineffectiveness
- Pressure drop through regenerator
- Non-isothermal compression and expansion

Nuclear Spin and Magnetic Moment

- Nuclear spin relaxation very slow (especially in insulators)

$$\frac{\mathrm{d}M}{\mathrm{d}t} = -\frac{(M - M_0)}{\tau_1}$$

- Nuclear spin relaxation slower than interaction between nuclear and electron spins

$$\tau_{\mathrm{eff}} = \tau_1 \frac{C_{\mathrm{e}}}{C_{\mathrm{n}} + C_{\mathrm{e}}}, \quad C_{\mathrm{n}} \gg C_{\mathrm{e}}$$

- Small magnetic moment (less interaction between nuclear moments means lower ordering temperature

$$\mu_{\mathrm{B}} = 9.27 \times 10^{-24}\,\mathrm{J\,T^{-1}}$$
$$\mu_{\mathrm{n}} = 5.05 \times 10^{-27}\,\mathrm{J\,T^{-1}}$$