Refrigeration at Ultralow Temperatures

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U.S. DEPARTMENT OF ENERGY | Office of Science

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(And how it's used to make targets at JLab)

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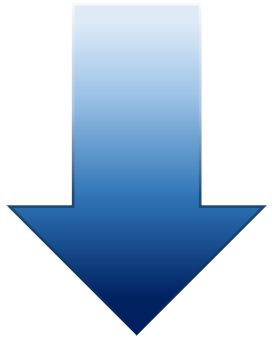
Outline

There is no “official” definition for what “Utralow” means, but I’ll say

any temperature less than 4.2 K (the normal boiling point of liquid helium).

Warmest



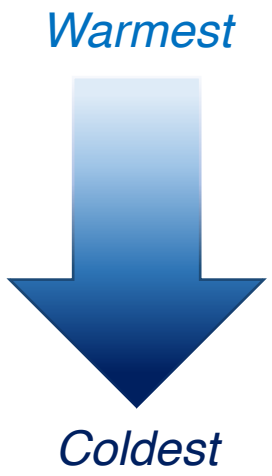
Coldest

I’ll describe four “common” techniques to achieve ultralow temperatures:

1. Mechanical Cryocoolers
2. Evaporation Refrigerators
3. Adiabatic Demagnetization Refrigerators
4. Dilution Refrigerators

Targets at JLab

A wide variety of targets (and temperatures) are used in electron-scattering experiments at JLab.



- Solid targets at room temperature: carbon, copper, lead, etc.
- Solid targets at cryogenic temperatures: carbon, copper, lead, etc.
- Liquid targets at cryogenic temperatures: hydrogen, helium
- Liquid hydrogen & deuterium: ~20 K
- Liquid or high-density gas ^{4}He & ^{3}He: <10 K
- Polarized solid targets: 1 K
- "Frozen-spin" polarized targets: < 0.05 K

Cryocoolers

Ideal “turn-key” solution for cooling small-load electronics, detectors, targets, magnets etc.

Can serve as a small-scale liquid helium plant (about 20 liters per day).

Also act as initial cooling stage for lower-temperature refrigeration systems like ADRs and Dilution Refrigerators (more on this later).

Gifford McMahon

- ✓ Lower cost
- ✓ Higher cooling above ~10 K
- ✗ Higher vibration
- ✗ Higher maintenance

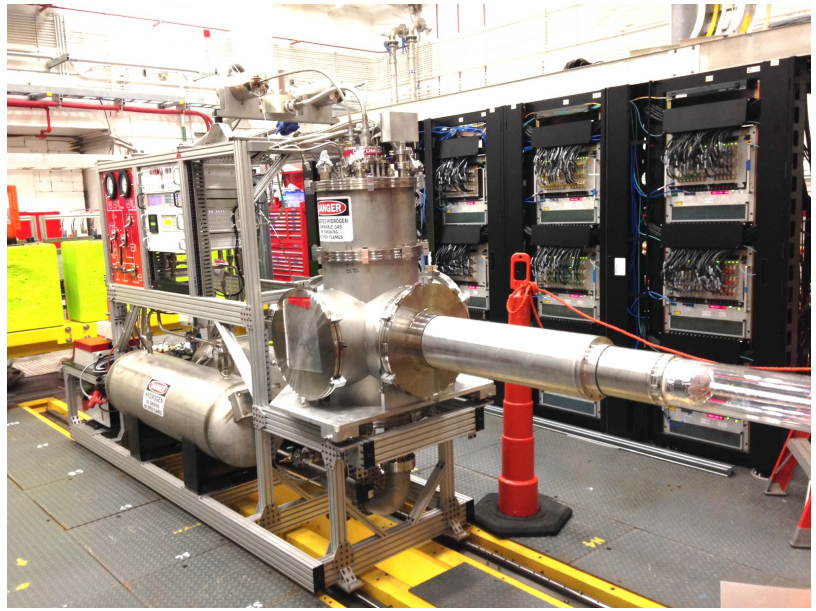
Cryomech Inc.

Pulse Tube

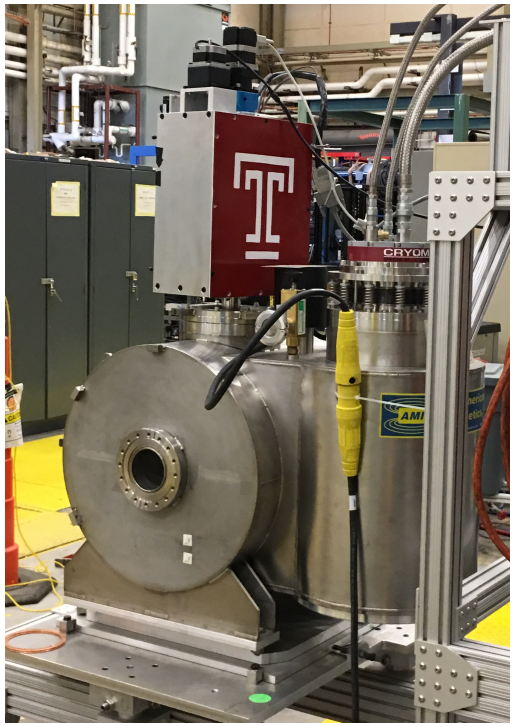
- ✗ Higher cost
- ✓ Lower base temperature: 2.3 K
- ✓ Lower vibration
- ✓ Lower maintenance

Cryocoolers

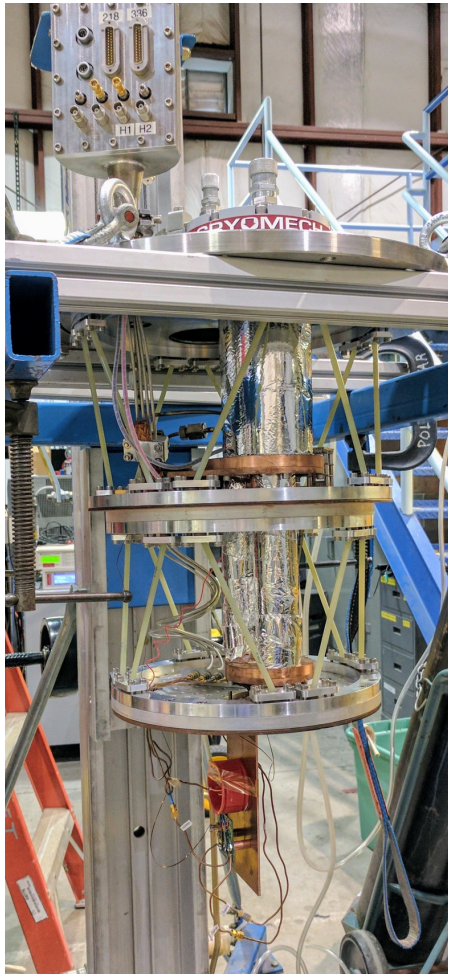
Some examples of cryocoolers in use around JLab



Hall D: Liquid hydrogen/helium target



Hall A: Superconducting magnet for Moller polarimeter



Target Group: 4K test apparatus

Evaporation Refrigerators

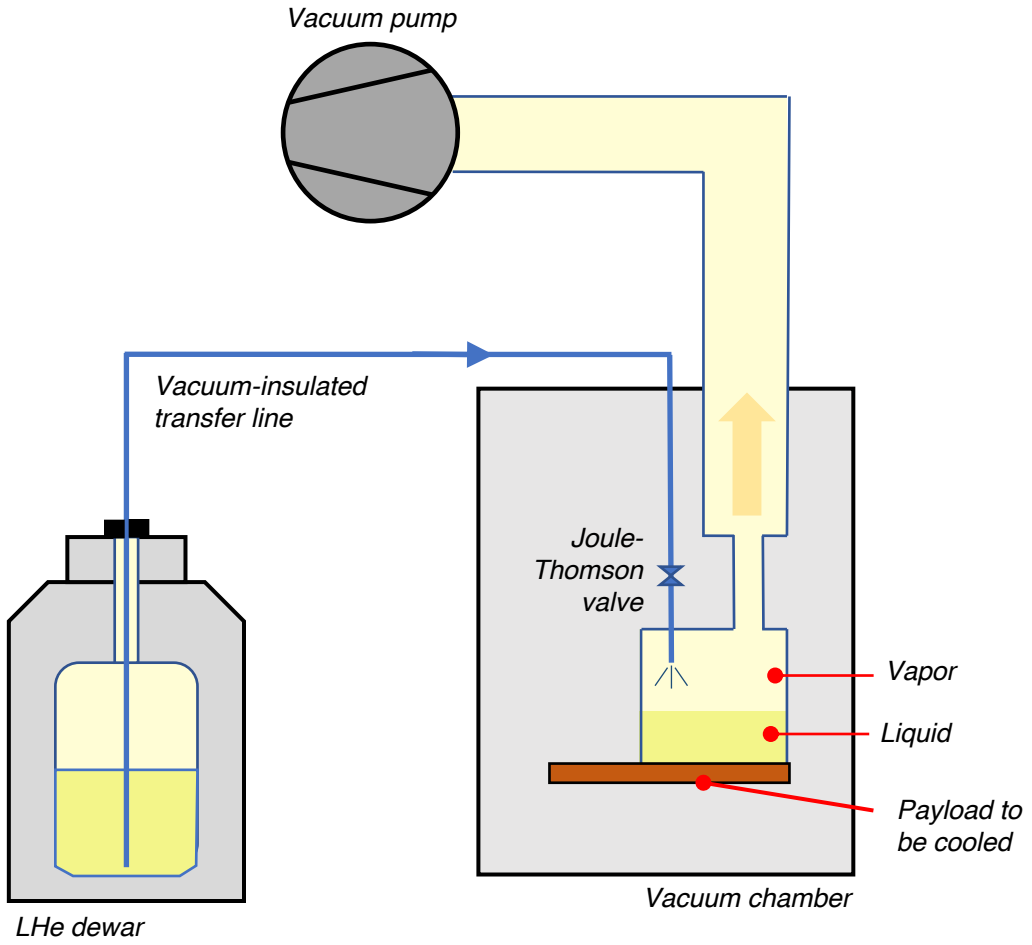
The simplest way to cool something is to submerge it in liquid helium

Liquid ^{4}He has a normal boiling point of 4.2 K and a latent heat of vaporization, $\mathrm{L} = 80\ \mathrm{J/mol}$

The cooling power is simply $\dot{Q} = \dot{n}L$,

where $\dot{n}$ is the rate of evaporation (mol/s)

The temperature can be lowered by reducing the vapor pressure (pumping)



Evaporation Refrigerators

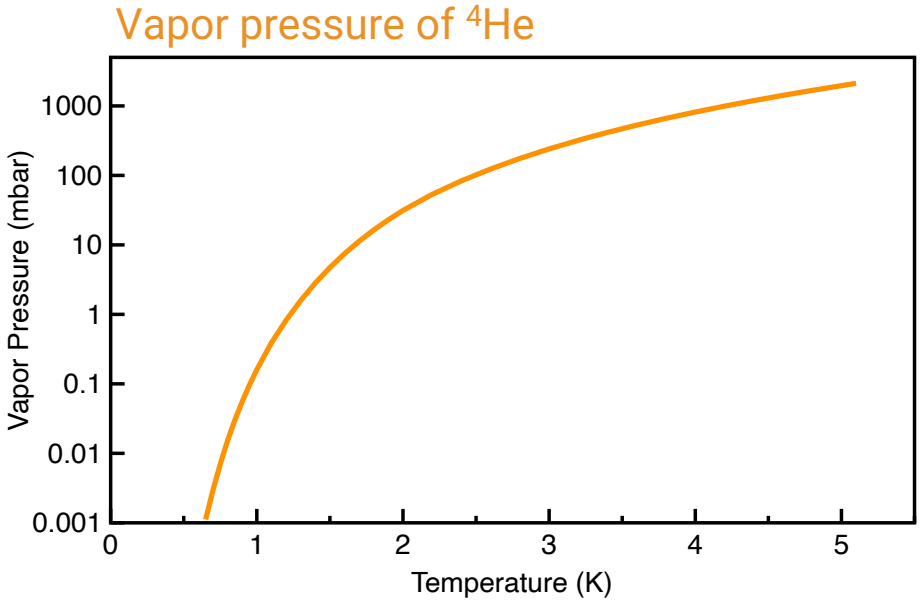
The simplest way to cool something is to submerge it in liquid helium

Unfortunately, the vapor pressure decreases rapidly with temperature: $P(T) \approx P_o e^{-L/RT}$

This means that the cooling power also drops rapidly with temperature $\dot{Q} \propto e^{-1/T}$

Example: pumping on LHe with a 5 l/s vac. pump

T(K)	P(mbar)	Q(W)
4	816	15
3	240	4
2	21	0.5
1	0.16	2.5E-3



Need more cooling power?

Use bigger pumps!!!

Need lower temperature?

Use a different helium!!!

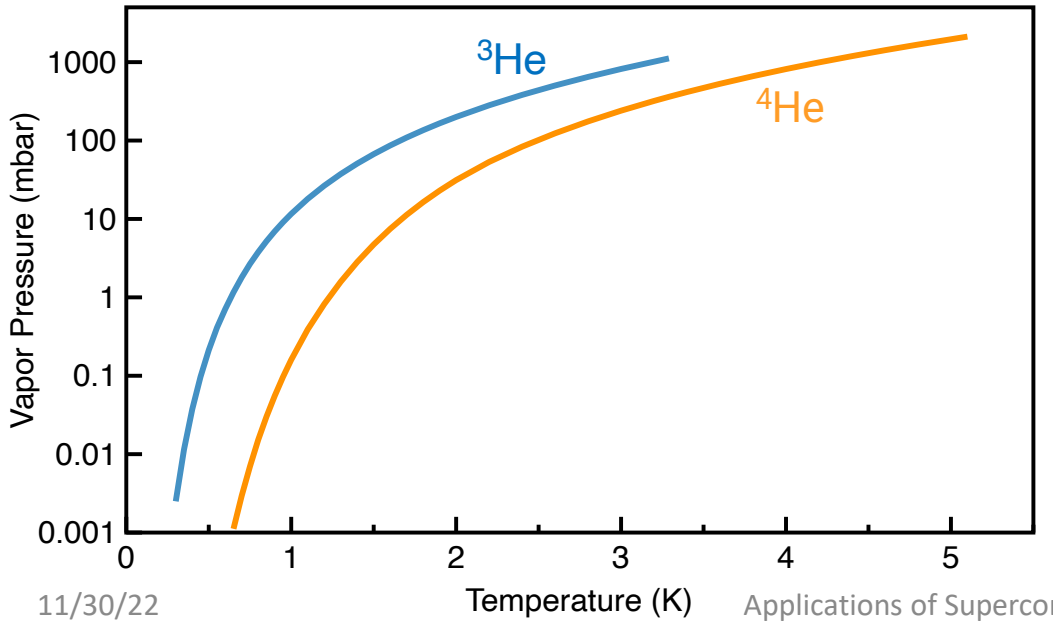
Evaporation Refrigerators

The simplest way to cool something is to submerge it in liquid helium

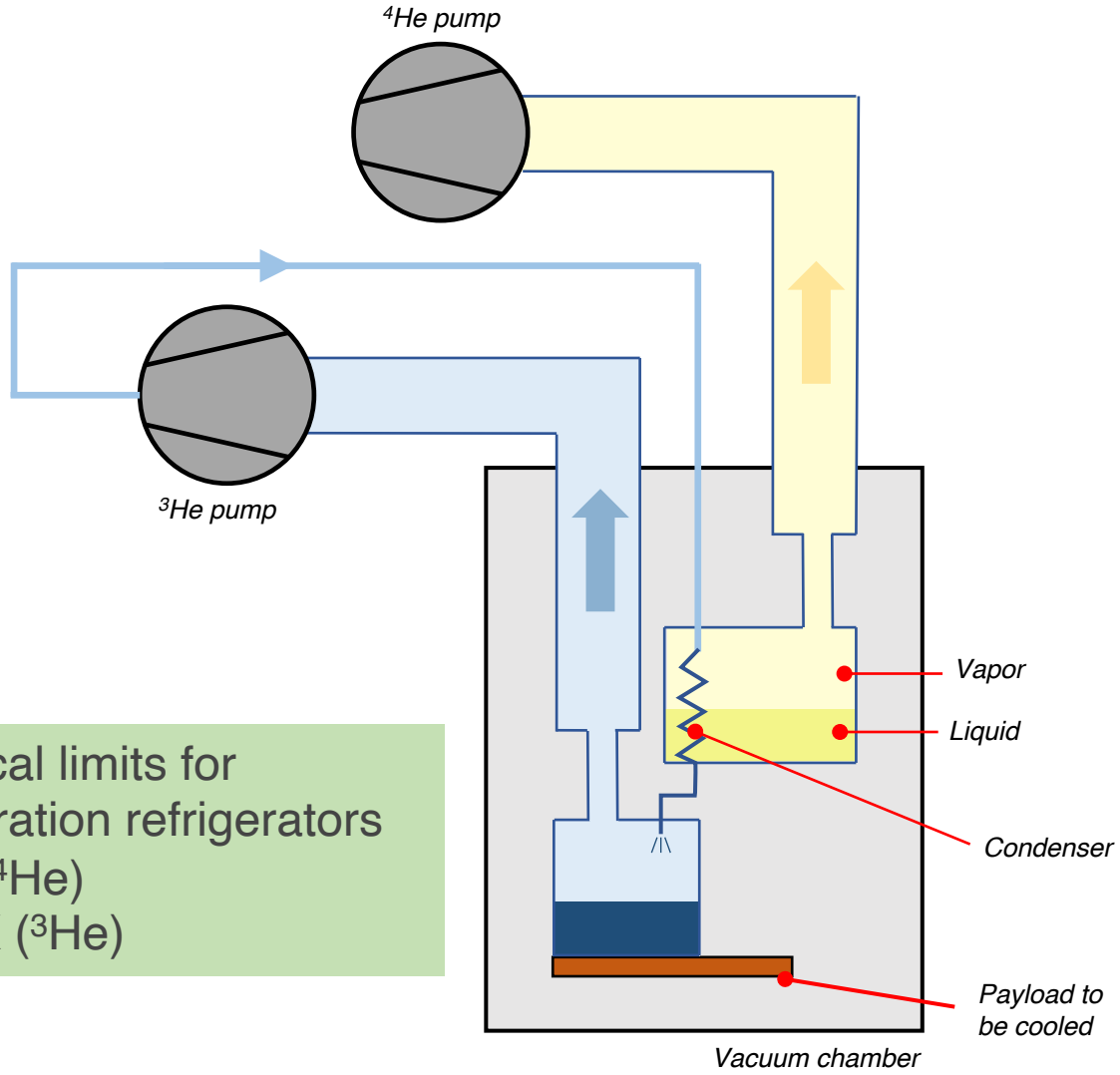
The lighter isotope of helium, ^{3}He, can be used to cool to a lower temperature, albeit at the cost of greater complexity (and more dollars!)

1 liter of liquid ^{4}He: ~ $10
1 liter of liquid ^{3}He: ~ $400,000*

*At the DOE price of ~$1000 per NTP gas liter

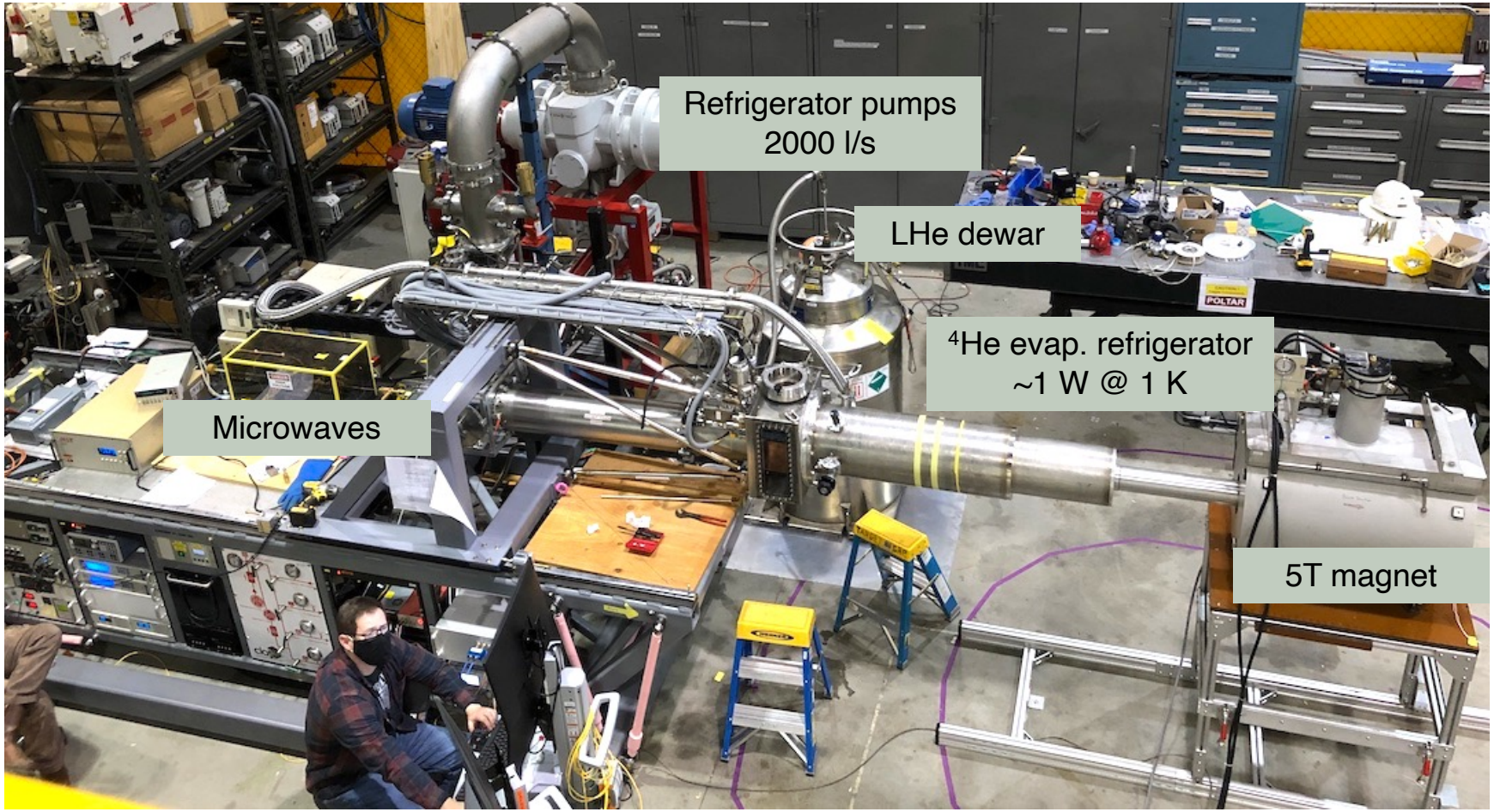


Practical limits for evaporation refrigerators
~1 K (^{4}He)
~0.3 K (^{3}He)



Evaporation Refrigerators

A 1K/ 5T dynamically polarized target being tested at JLab, April 2021



With one exception, all dynamically polarized targets at JLab have used high-power ^{4}He evaporation refrigerators

Evaporation Refrigerators

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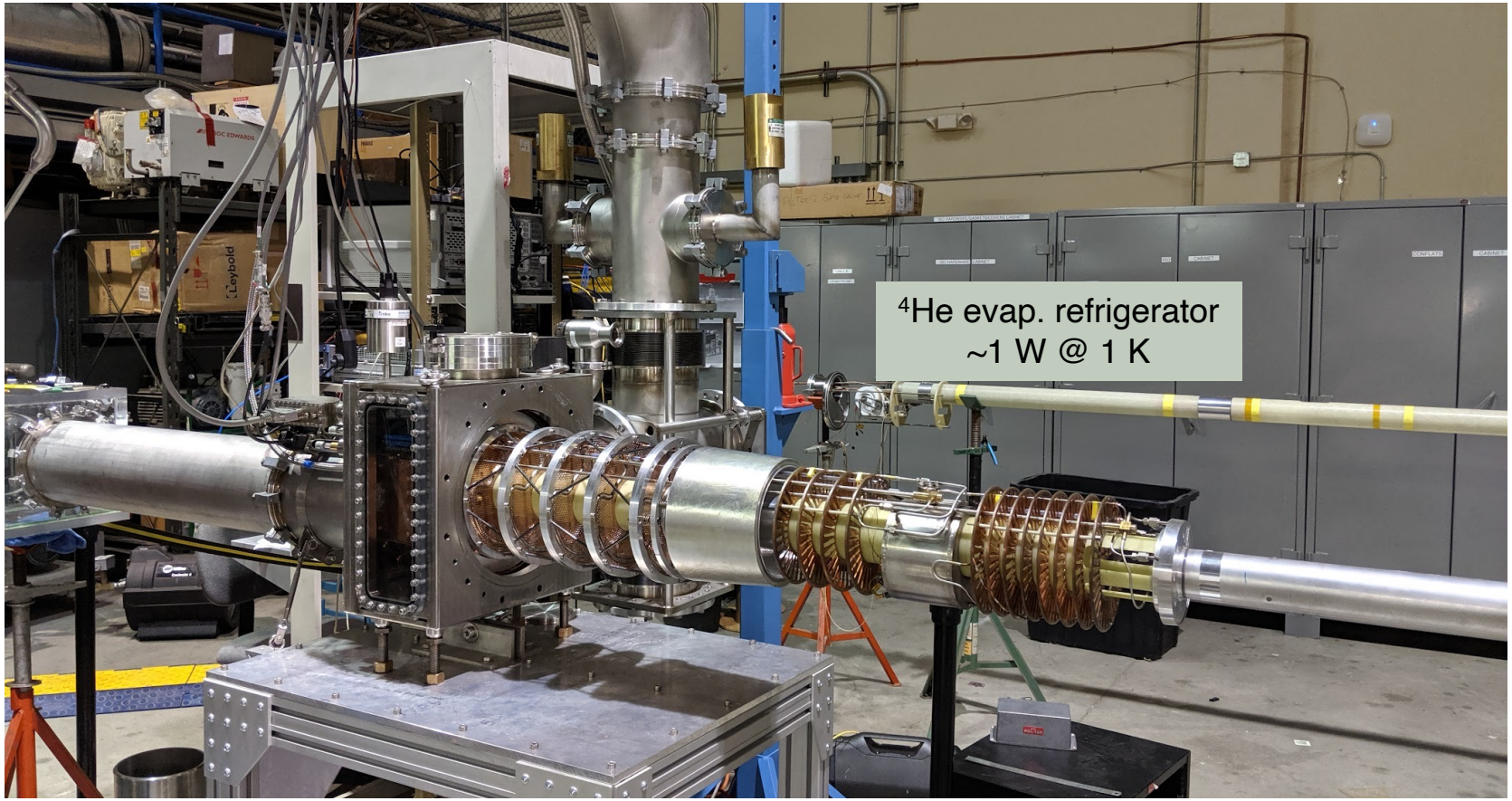
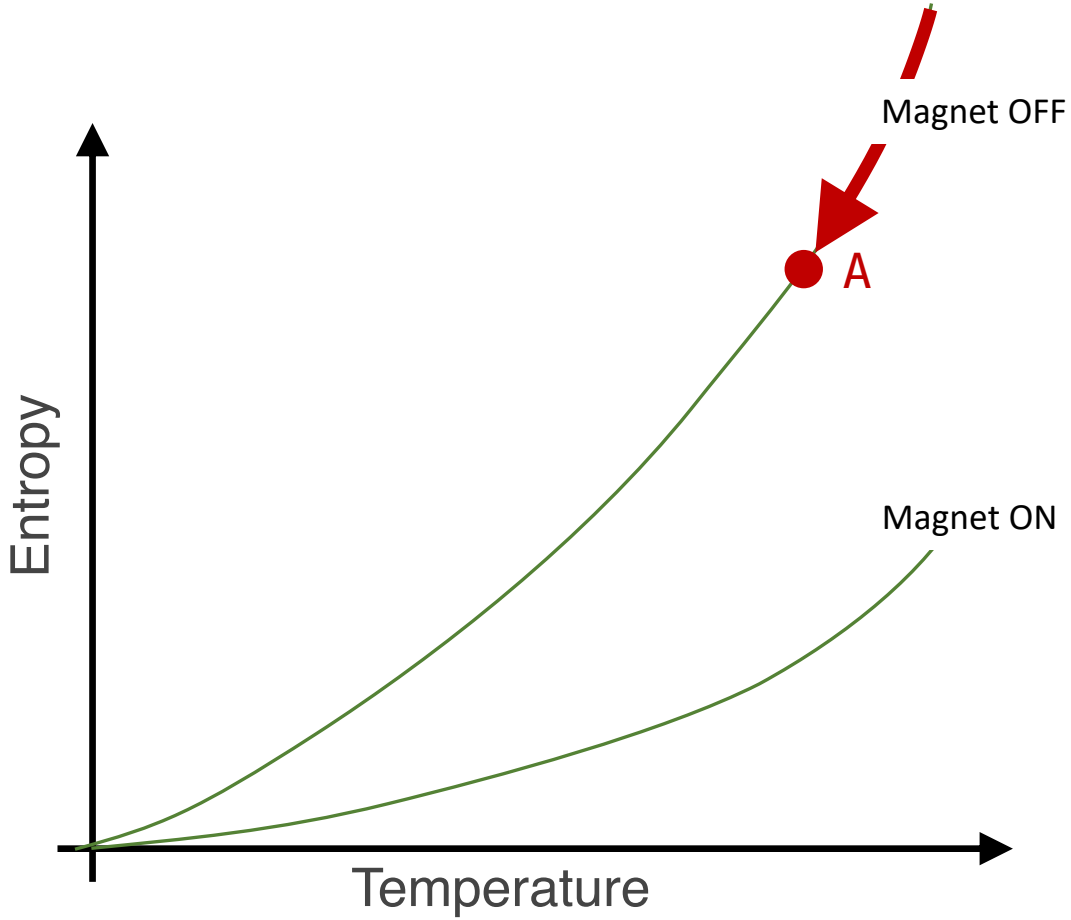


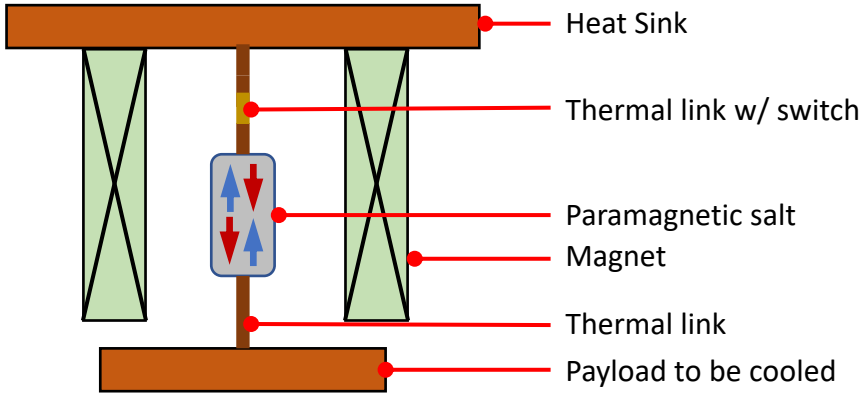
With one exception, all dynamically polarized targets at JLab have used high-power ^{4}He evaporation refrigerators

Adiabatic Demagnetization

Adiabatic Demagnetization Refrigerators (ADR) utilize the magnetocaloric effect to cool items in thermal contact with a paramagnetic substance



<u>Step 1:</u> Thermal switch closed. Cool heat sink (and payload) to point A.



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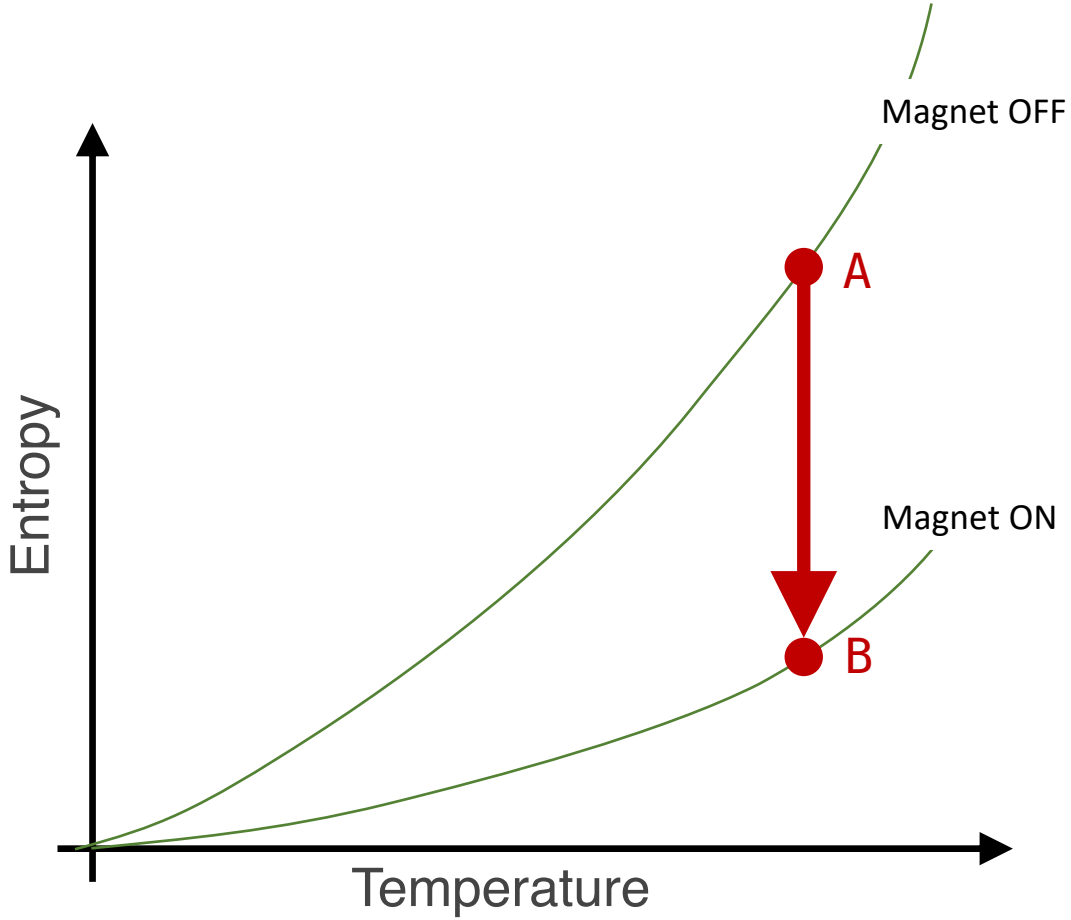
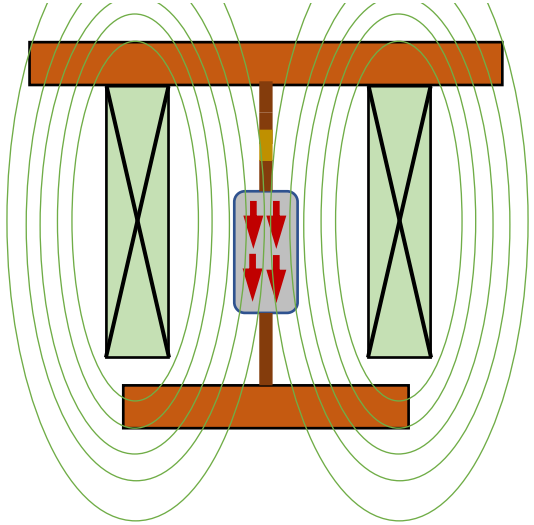


<u>Step 2:</u> Energize magnet and magnetize paramagnetic salt pill.
Reduce to entropy to point B.



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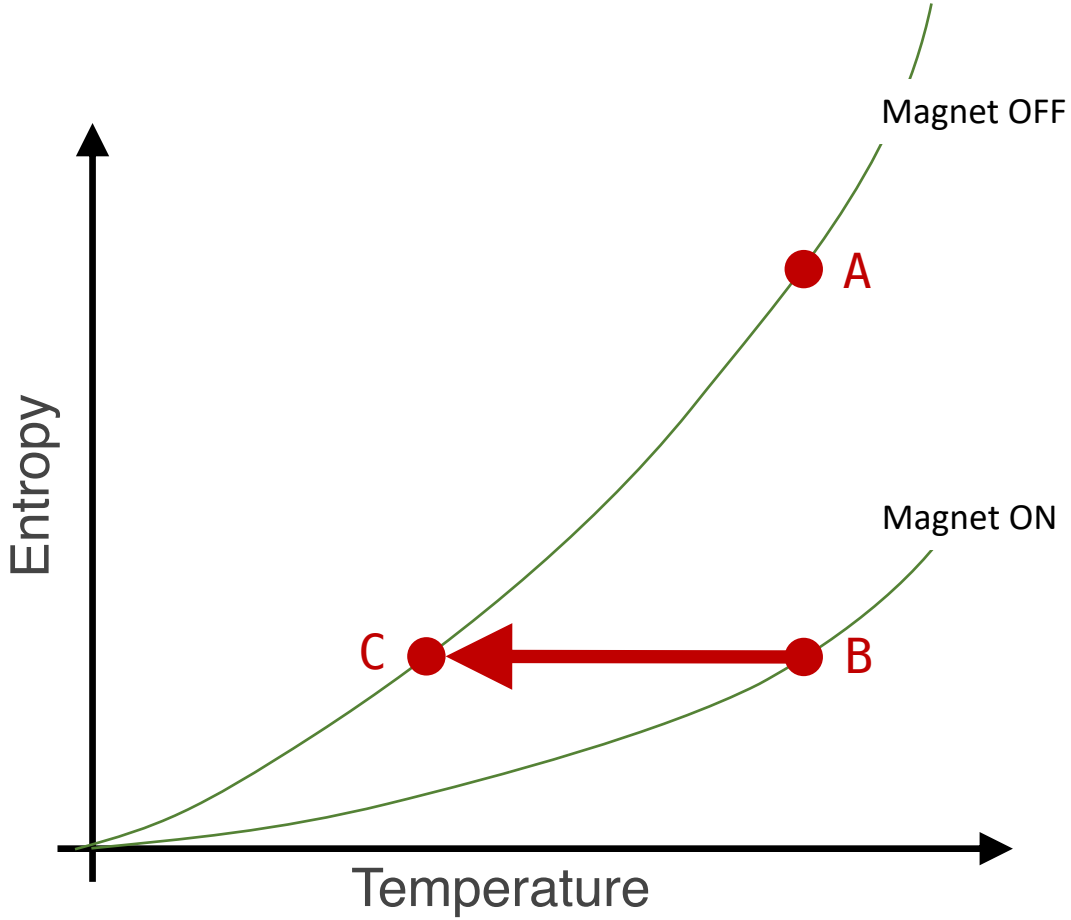
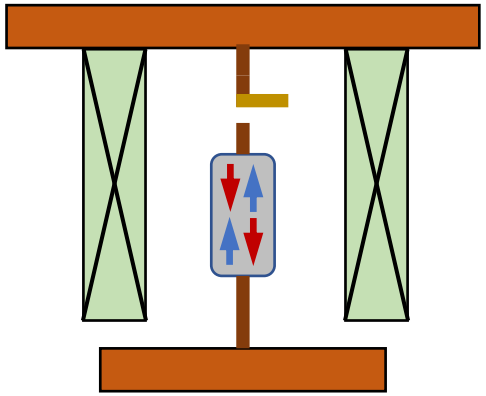


<u>Step 3:</u> Open heat switch & de-energize magnet. Paramagnets absorb heat and adiabatically cool payload to point C.



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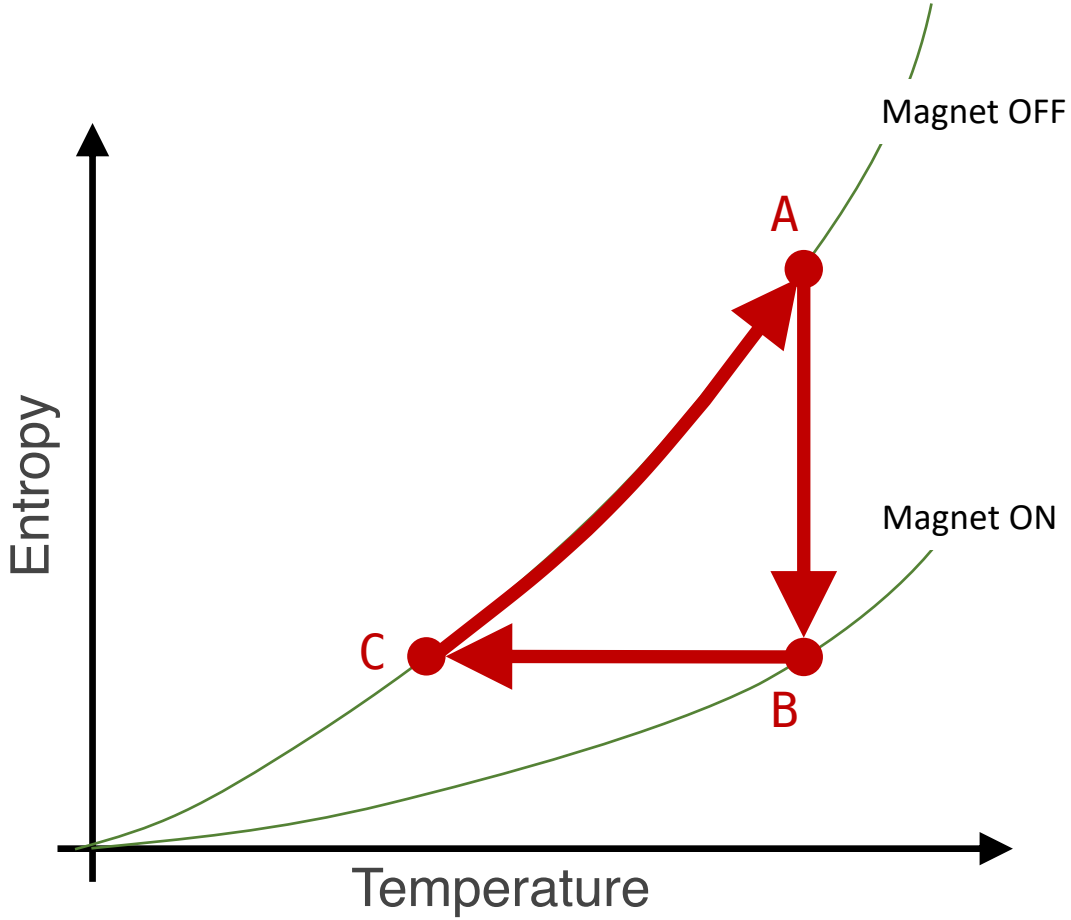
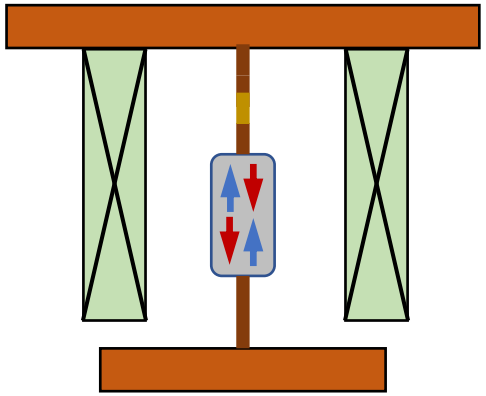


Step 4: Repeat as necessary as payload warms due to heat leaks.



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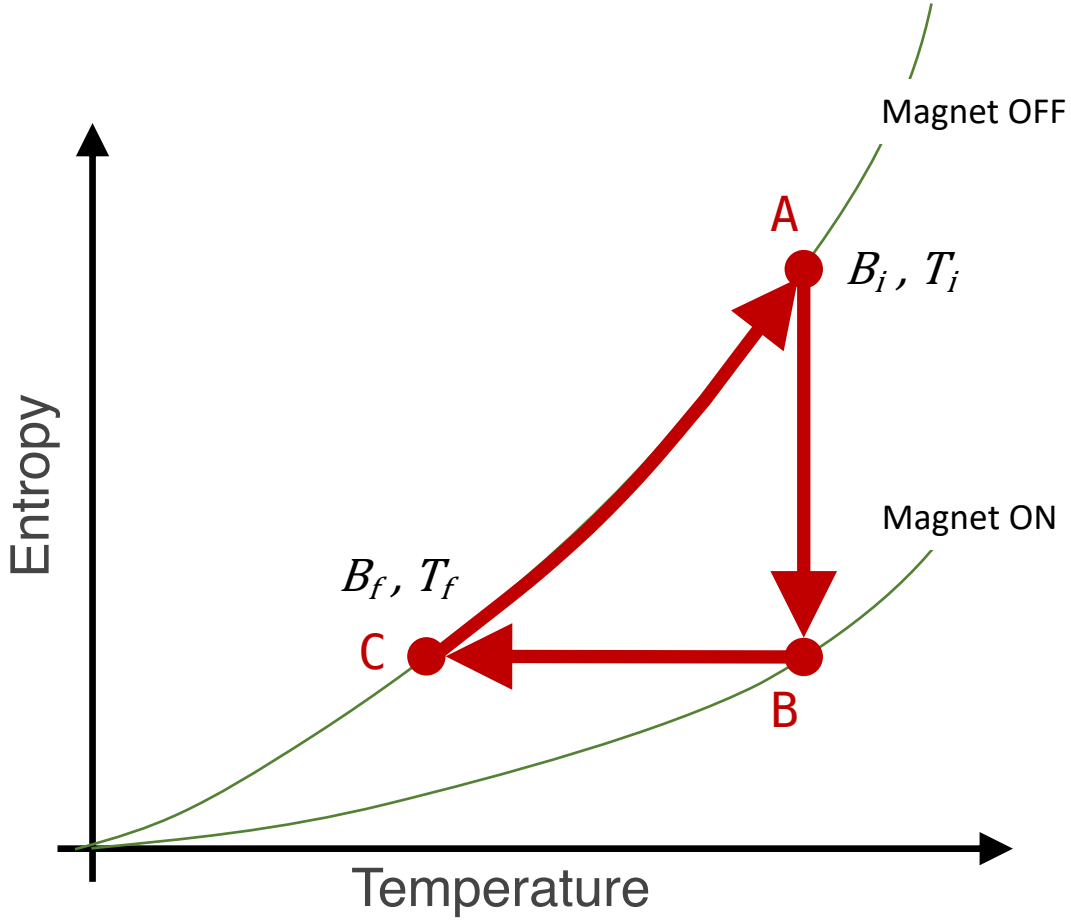


Ideally, the final temperature is given as

$$T_f = \frac{B_f}{B_i} T_i$$

But this violates the Third Law of Thermodynamics

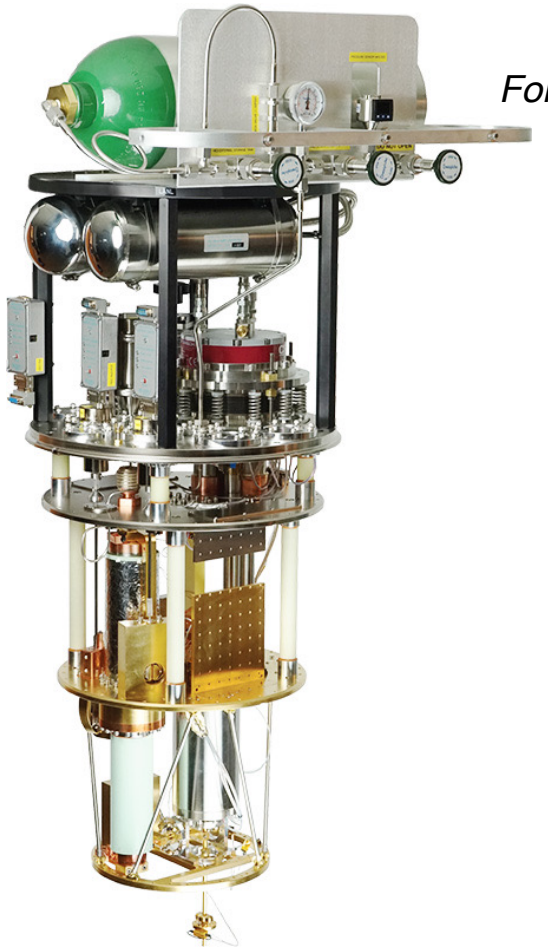
A more correct result has to include *b*, the magnetic field produced by the paramagnets themselves:

$$T_f = \frac{\left[b^2 + B_f^2\right]^{1/2}}{\left[b^2 + B_i^2\right]^{1/2}} T_i$$

Alternatively, the field can be decreased at a controlled rate to maintain a constant temperature, $T_f < T < T_i$ for a prolonged period of time.

Adiabatic Demagnetization

Modern day ADRs are available commercially



Form Factor Model 107 ADR

- Precooled by a pulse tube cryocooler & ^{3}He evap. stage
- Base temperature ~25 mK
- 250 hours at 100 mK with 0 W applied heat
- 6 J of *cooling energy* at 300 mK
  - 1700 hours @ 1 μW
  - 1.7 hours @ 1 mW

✓ No moving parts, very low vibration
✓ Simple operation
✗ Magnetic field
✗ Non-continuous cooling

Adiabatic Demagnetization

The very first polarized target!! *(that I know of)*

Although the earliest (brute-force) polarized targets utilized ADRs to cool the target sample, the non-continuous nature of these refrigerators make them poorly suited for modern-day polarized targets.

PHYSICAL REVIEW VOLUME 94, NUMBER 5 JUNE 1, 1954

Interaction of Polarized Neutrons with Polarized Mn^{55} Nuclei

S. BERNSTEIN, L. D. ROBERTS, C. P. STANFORD, J. W. T. DABBS, AND T. E. STEPHENSON
Oak Ridge National Laboratory, Oak Ridge, Tennessee

(Received November 16, 1953; revised manuscript received February 15, 1954)

The dependence of the capture cross section of polarized Mn^{55} nuclei for polarized neutrons upon relative orientations of incident and bombarded particles has been directly observed. The target material was the paramagnetic substance, manganous ammonium sulfate, which is known to have a large hyperfine structure coupling. It was placed in a magnetic field of 2350 oersteds at a temperature of 0.20°K. Under these conditions the polarization of the paramagnetic electrons is about 85 percent. Because of the large effective magnetic field created by the paramagnetic electrons at the Mn nucleus, the nuclei should achieve a polarization of 16 percent. The 2.6-hour activity of the residual nucleus, Mn^{56}, was measured after the sample had been bombarded with a beam of slow neutrons polarized to the extent of 32 percent by passage through magnetized iron. The activity for neutron polarizing field and sample polarizing field parallel was found to be 3.4 percent less than for the fields antiparallel. The difference in the two activities was found to depend upon the sample temperature in accordance with theory. The difference was found to be unaccompanied by a corresponding change in sample transmission. These results are interpreted to mean that the change in sample activity was due to the dependence of the capture cross section of the polarized Mn nuclei upon the relative orientation of the interacting particles. The observations are discussed in terms of available information about the energy level system of the compound nucleus, Mn^{56}.

INTRODUCTION

THE spin dependence of nuclear forces was first demonstrated experimentally by the scattering of neutrons from ortho- and para-hydrogen,[1–4] in which experiment the effects observed are caused by the relative spin directions taken by the hydrogen atoms of each isolated hydrogen molecule. The spin dependence of scattering is exhibited also in the spin incoherent scattering of neutron diffraction experiments,[5] in which the random orientations of the nuclear spins of the sample cause an isotropic diffuse background in diffraction patterns. In both of these methods it is the spin dependence of the scattering cross section which is observed, and the bulk nuclear magnetization of the sample is zero. We have observed directly the spin dependence of the *capture* cross section by bombarding a sample possessing a bulk nuclear magnetization with a beam of polarized neutrons, and observing the dependence of the target activity upon relative spin orientations of the incident and bombarded particles. (The angular distribution of gamma rays from such polarized samples containing radioactive nuclei has been observed.[6] An experiment in which neutron effects of nuclear polarization in gadolinium sulfate were observed, has been mentioned briefly,[7] but no details of the experiment have been reported.)

For polarizing the nuclei we relied upon the method of Rose[8] and Gorter,[9] i.e., the use of the strong magnetic field produced at the nucleus by hyperfine structure coupling in an appropriate paramagnetic substance. An external magnetic field is applied to the sample merely for the purpose of polarizing the electronic moments. The sample used in this work was the deuterated paramagnetic salt, $Mn^{55}SO_4 \cdot (ND_4)_2SO_4 \cdot 6D_2O$. Its Mn^{55} nuclei were polarized by partial adiabatic demagnetization of the salt. This substance was selected because its hyperfine structure is known to be large,[10] its low-temperature properties are suitable, the 13-barn neutron capture cross section of Mn^{55} is acceptably large, and the 2.60-hour half-life of the radioactive compound nucleus, Mn^{56}, is convenient.

The electronic system of the Mn^{++} ion possesses spin angular momentum only. The nuclear polarization is given by

$$f_N = \frac{S(I+1)hc}{3}\frac{A}{kT}\frac{M_e}{M_\infty} \qquad (1)$$

in which S is the electron spin quantum number, I is the nuclear spin quantum number, k is the Boltzmann constant, T is the temperature, M_e is the magnetic moment of the electron spin system, M_∞ is the electronic magnetic moment at saturation, h is Planck's constant, c is the velocity of light, and A is the hyperfine structure coupling constant. The spatial average value of A for this substance has been calculated from measurements[10] to be 0.00932 cm^{-1}. Substituting the appropriate values $S=5/2$, $I=5/2$, expression (1) becomes

$$f_N = \frac{0.0391}{T}\frac{M_e}{M_\infty}. \qquad (2)$$

[1] Halpern, Estermann, Simpson, and Stern, Phys. Rev. **52**, 142 (1937).
[2] Brickwedde, Dunning, Hoge, and Manley, Phys. Rev. **54**, 266 (1938).
[3] L. W. Alvarez and K. S. Pitzer, Phys. Rev. **58**, 1003 (1940).
[4] Sutton, Hall, Anderson, Bridge, DeWire, Lavatelli, Long, Snyder, and Williams, Phys. Rev. **72**, 1147 (1947).
[5] Shull, Wollan, Morton, and Davidson, Phys. Rev. **73**, 262 (1948).
[6] Ambler, Grace, Halban, Kurti, Durand, Johnson, and Lemmer, Phil. Mag. **349**, 216 (1953).
[7] J. A. Beun, Ned. Tijdschr. Natuurk. **18**, 245 (1952).
[8] M. E. Rose, Phys. Rev. **75**, 213 (1949).
[9] C. J. Gorter, Physica **14**, 504 (1948).
[10] B. Bleaney and D. J. E. Ingram, Proc. Roy. Soc. (London) **A205**, 336 (1951).

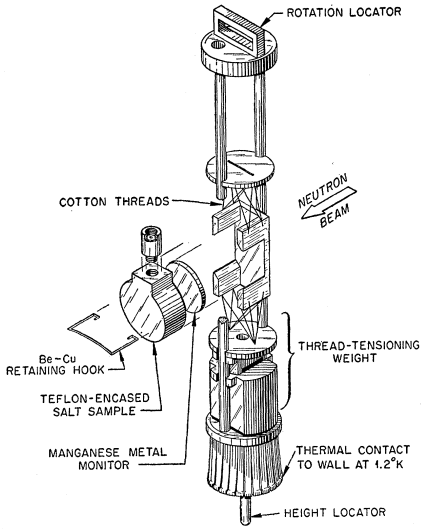
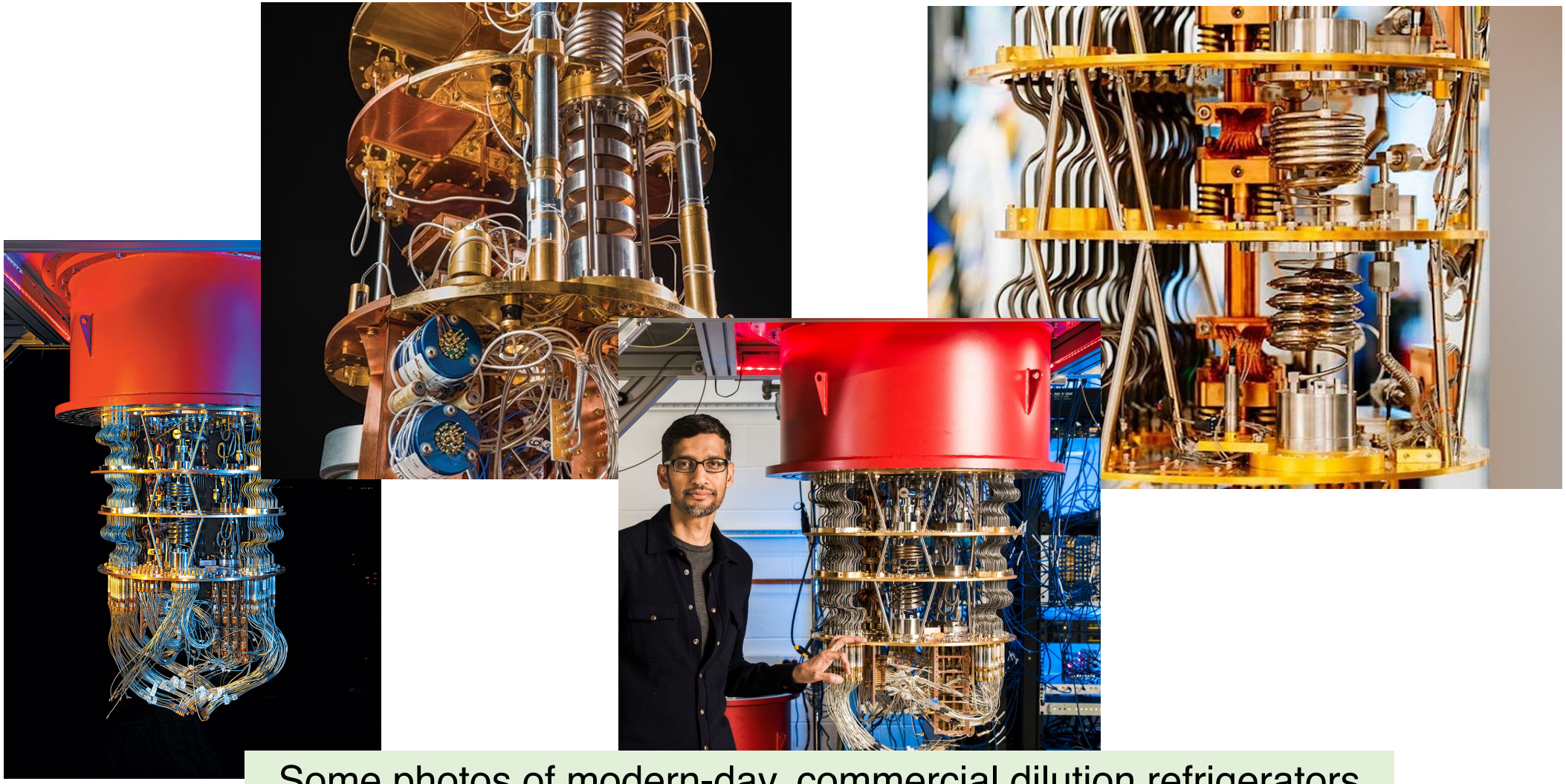


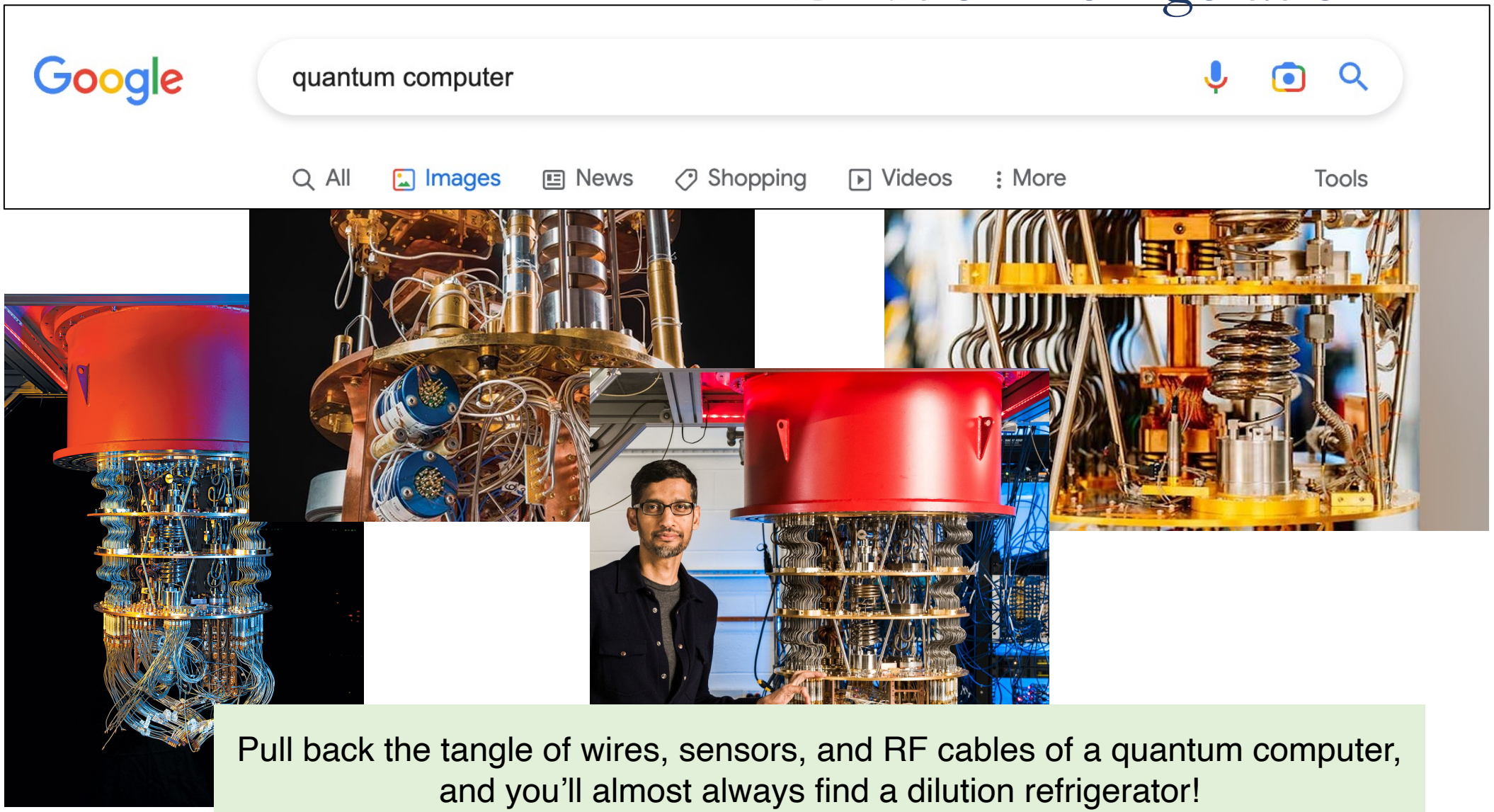
FIG. 2. Sample and monitor assembly.

Dilution Refrigeration



Some photos of modern-day, commercial dilution refrigerators

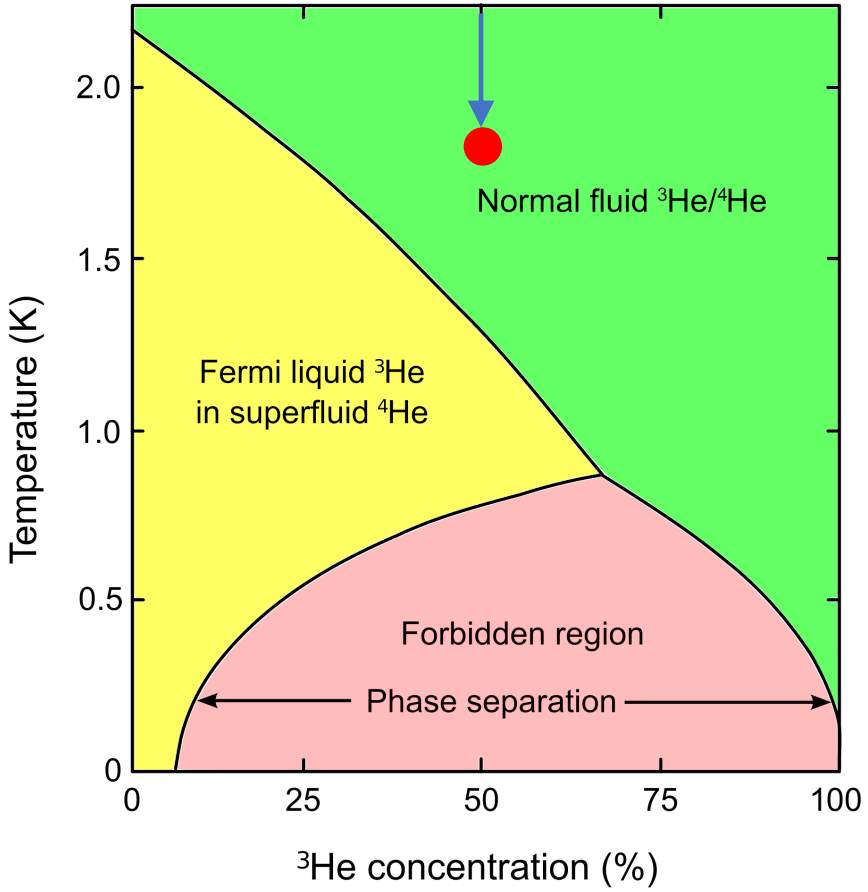
Dilution Refrigeration



Pull back the tangle of wires, sensors, and RF cables of a quantum computer, and you'll almost always find a dilution refrigerator!

Dilution Refrigeration

Dilution refrigeration is based on the behavior of ^{3}He-^{4}He *mixtures* at low temperatures



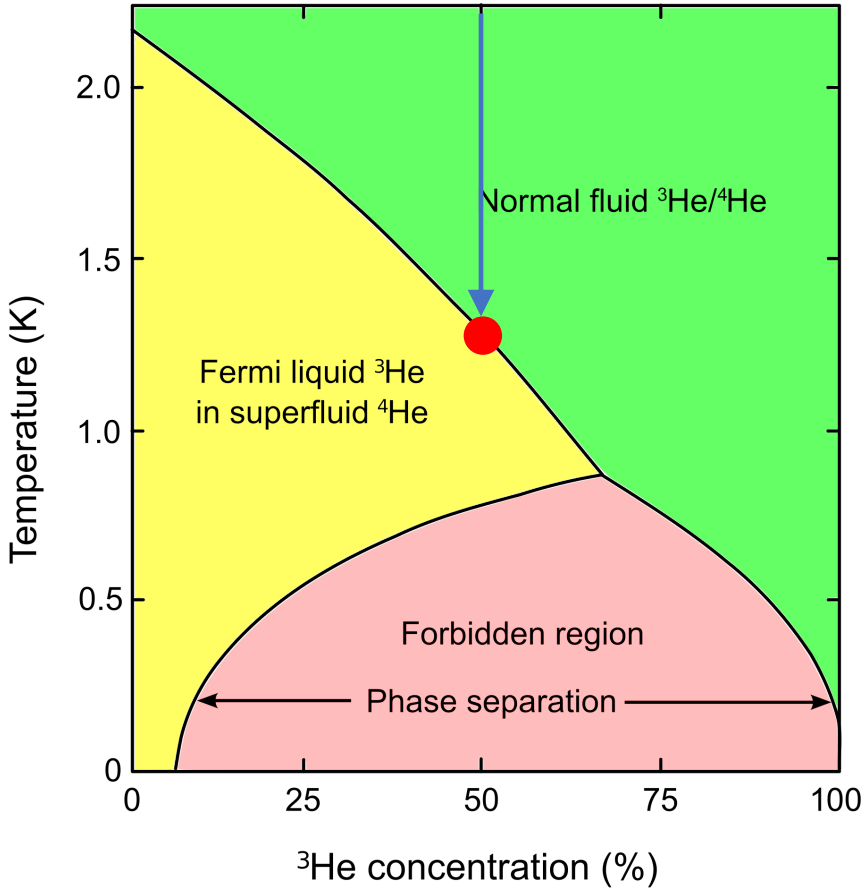
Example: a container filled with a 50:50 mix of ^{3}He and ^{4}He

Following condensation, the mixture is a uniform, viscous liquid

Temperature

Dilution Refrigeration

Dilution refrigeration is based on the properties of 3He-4He mixtures at low temperatures



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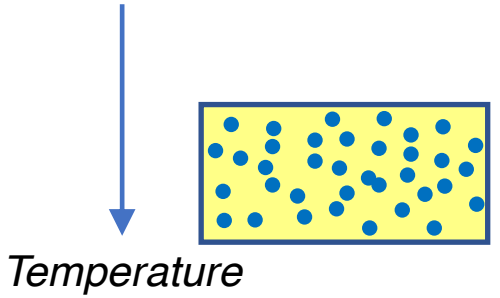

At 1.3 K, the ^{4}He fraction becomes a superfluid with zero viscosity and zero entropy. The ^{3}He fraction acts like a Fermi-Dirac "gas" of atoms floating in the superfluid "vacuum".

Dilution Refrigeration

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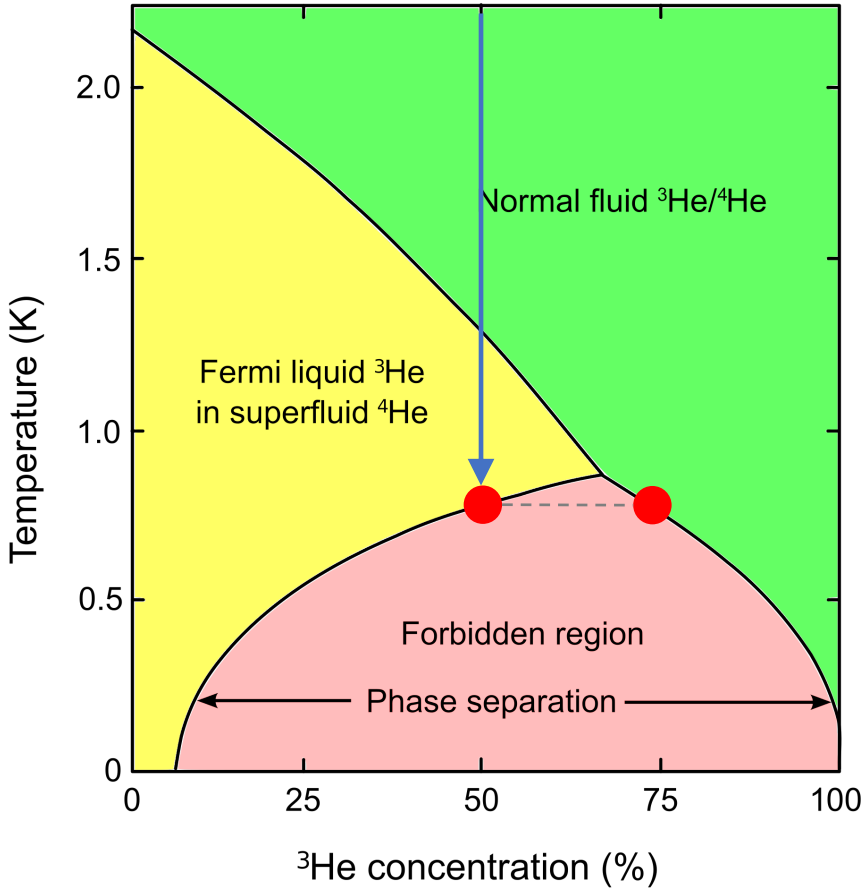

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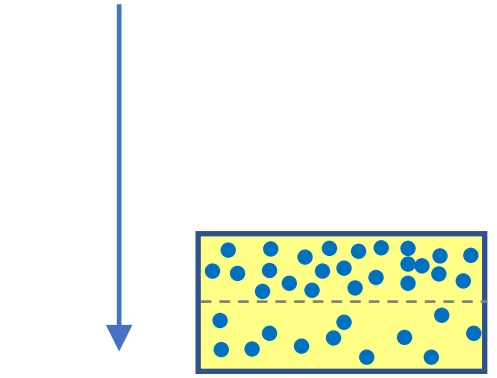
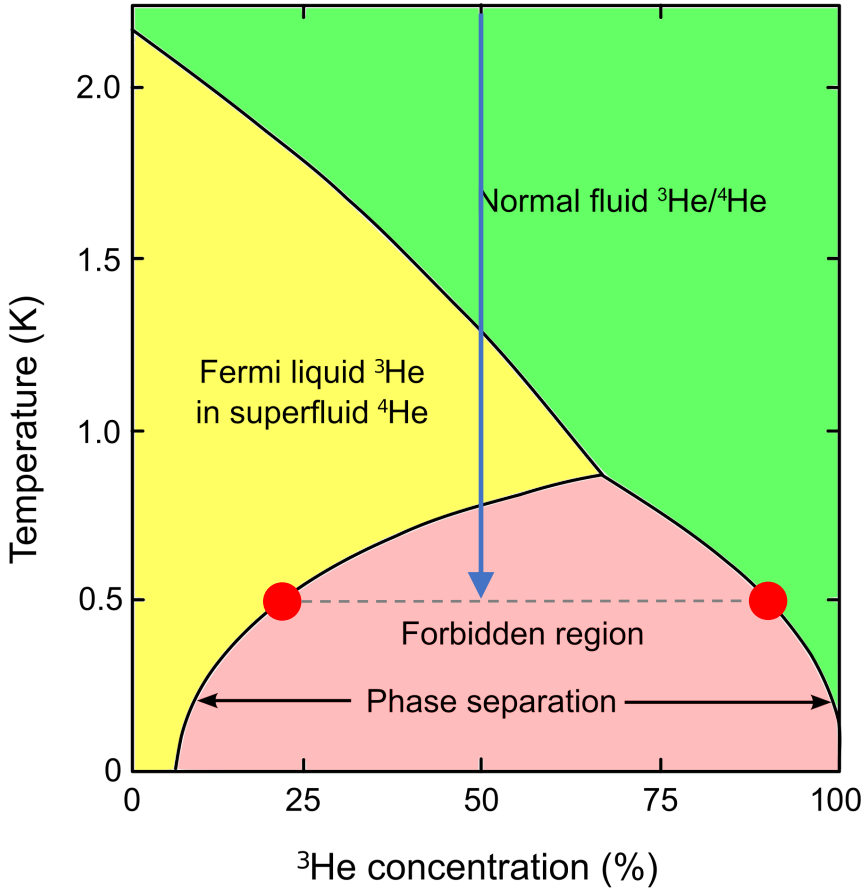


At 0.75 K, phase separation occurs in the container. The ^{3}He fraction in the lower phase is 50% and in the upper phase 75%.

Dilution Refrigeration

Dilution refrigeration is based on the properties of 3He-4He mixtures at low temperatures



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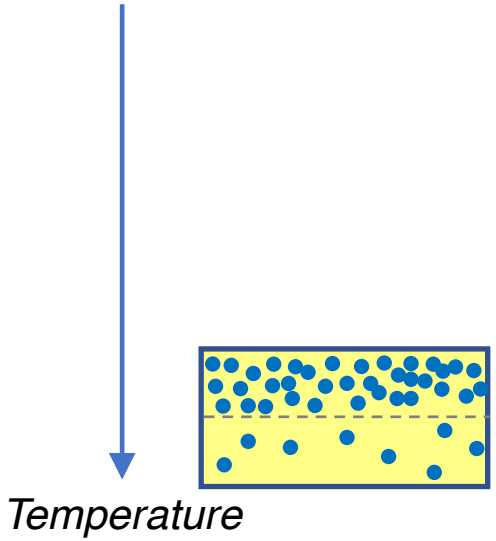
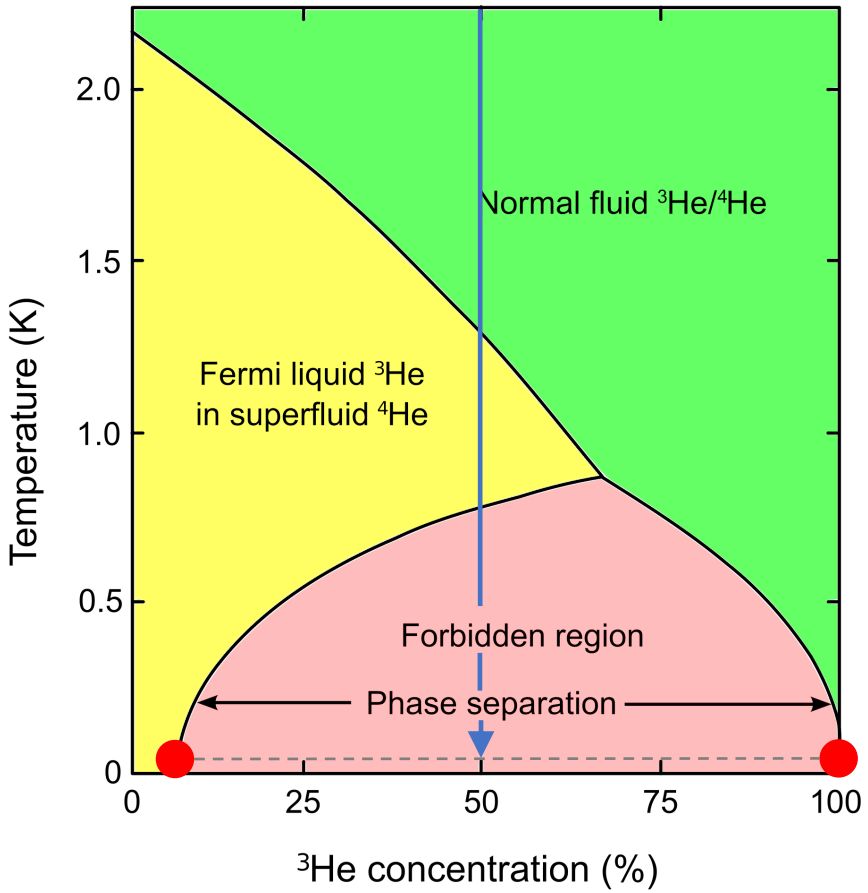


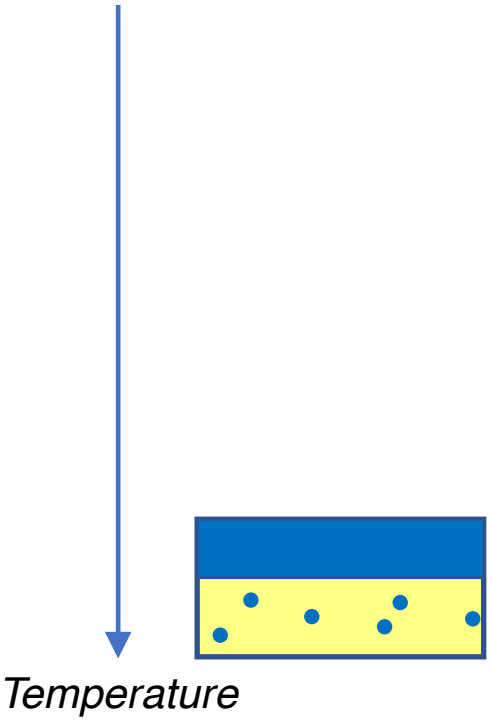
As the container continues to cool, more ^{3}He floats to the top and upper concentration increases, while the lower one decreases.

Dilution Refrigeration

Dilution refrigeration is based on the properties of 3He-4He mixtures at low temperatures



Example: a container filled with a 50:50 mix of ^{3}He and ^{4}He



As we approach absolute zero, the upper phase approaches 100% ^{3}He. The lower concentration reaches an *asymptotic limit of 6.6% ^{3}He*.

Dilution Refrigeration

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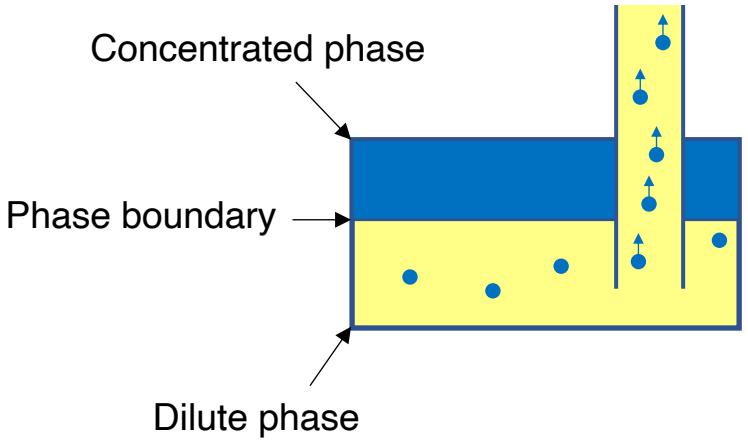
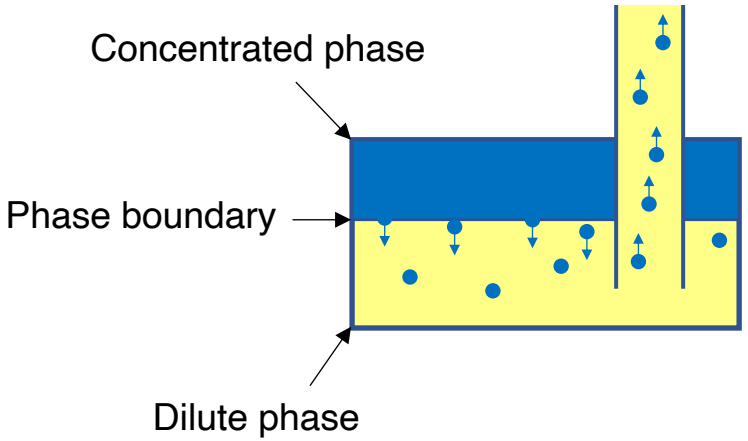


If the concentration of ^{3}He atoms in the lower phase is reduced…

Dilution Refrigeration

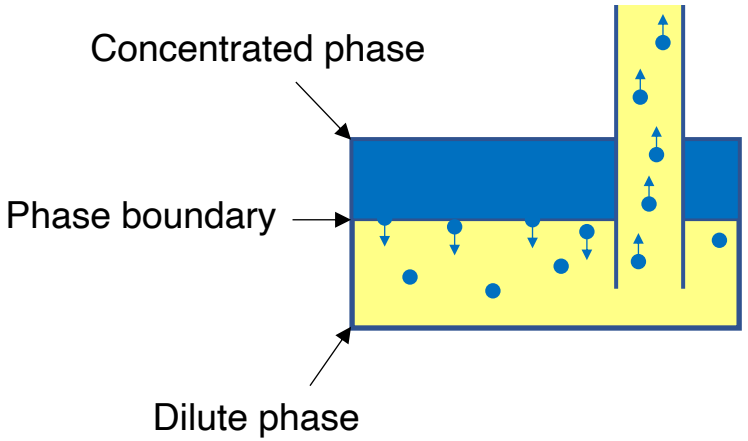
Dilution refrigeration is based on the properties of 3He-4He mixtures at low temperatures



… atoms from the upper phase will cross the phase boundary to restore the equilibrium concentration.

Dilution Refrigeration

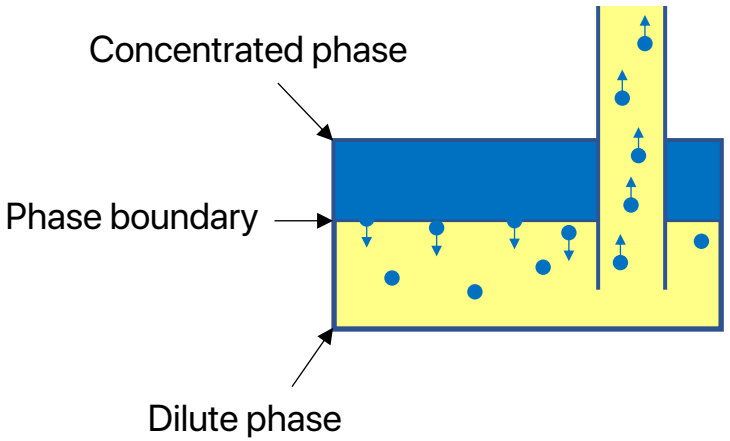
Dilution refrigeration is based on the properties of 3He-4He mixtures at low temperatures



The ^{3}He atoms absorb heat as they enter the dilute phase, cooling their surroundings.

Dilution Refrigeration

Dilution refrigeration is based on the properties of 3He-4He mixtures at low temperatures



The process is akin to the upside-down evaporation of atoms from liquid to gas, with the latent heat of *dilution* replacing the latent heat of *evaporation.*

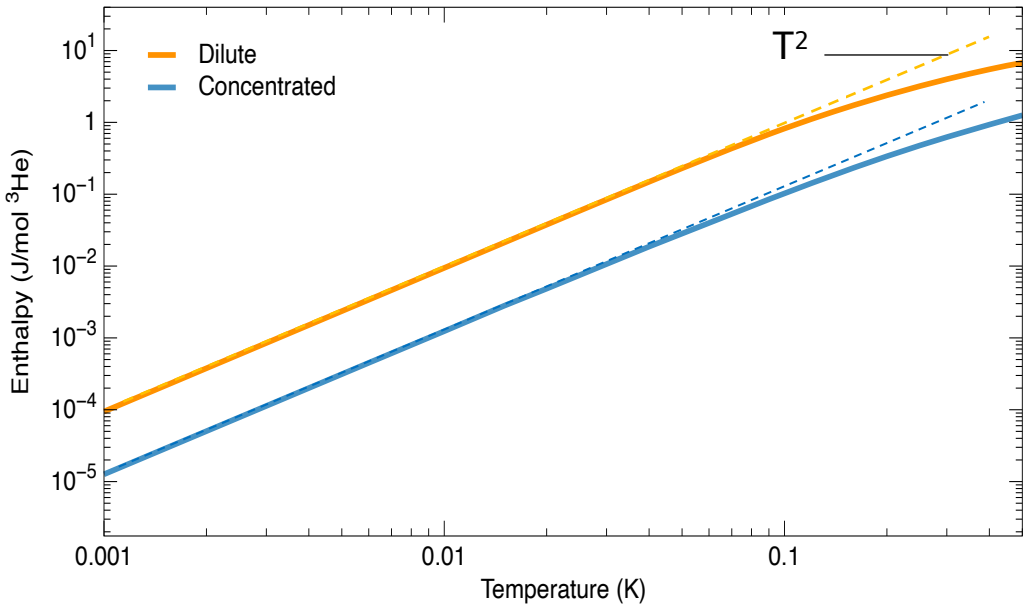
In an evaporation refrigerator, the vapor pressure goes to zero at T = 0. In a dilution refrigerator, the ^{3}He concentration never goes below 6.6%.

➔ Dilution fridges cool to MUCH lower temperatures!
(The world record is 1.7 mK)

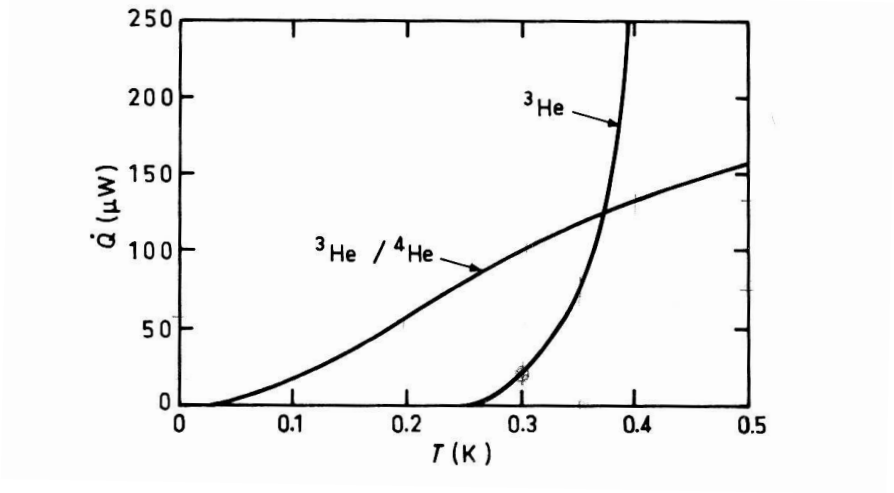
Dilution Refrigeration

The cooling power is proportional to the difference in enthalpy of ^{3}He atoms in the dilute and concentrated phases.

$$\dot{Q} = \dot{n}(H_d - H_c)$$
$$\approx \dot{n}\left[95T_m^2 - 11T_i^2\right]$$



The cooling power of an evaporation refrigerator decreases exponentially with temperature. It only decreases quadratically for a dilution refrigerator.



Available cooling power of ^{3}He evaporation and ^{3}He-^{4}He dilution, both using 5 l/s pump.

Dilution Refrigeration

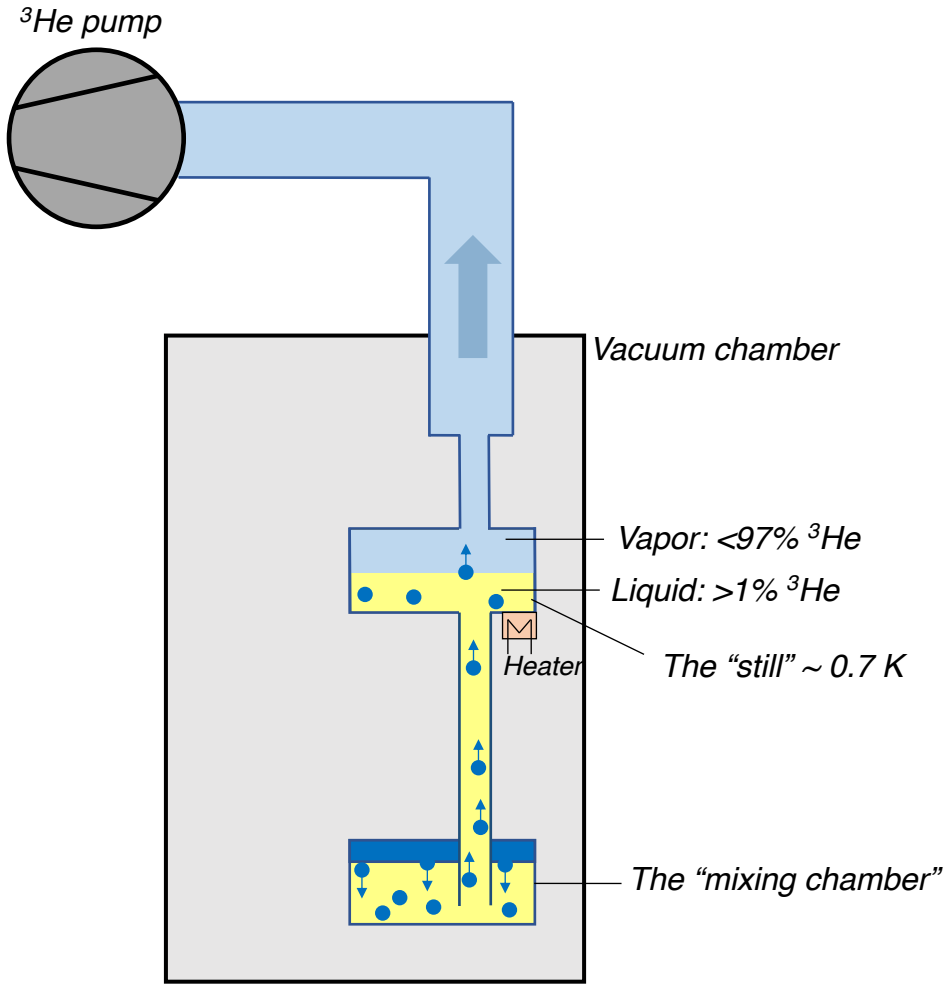
^{3}He is removed from the dilute phase by fractional distillation at a higher temperature

The still is heated to temperature of about 0.7 K, where the vapor pressure is reasonably high, ~0.1 mbar.

The vapor phase is 97% ^{3}He, so pumping mostly removes ^{3}He from the liquid.

Decreasing the ^{3}He concentration in the still creates an osmotic pressure gradient that sucks ^{3}He from the mixing chamber.

The circulation rate of ^{3}He, $\dot{n}$, is varied by adjusting the still temperature.

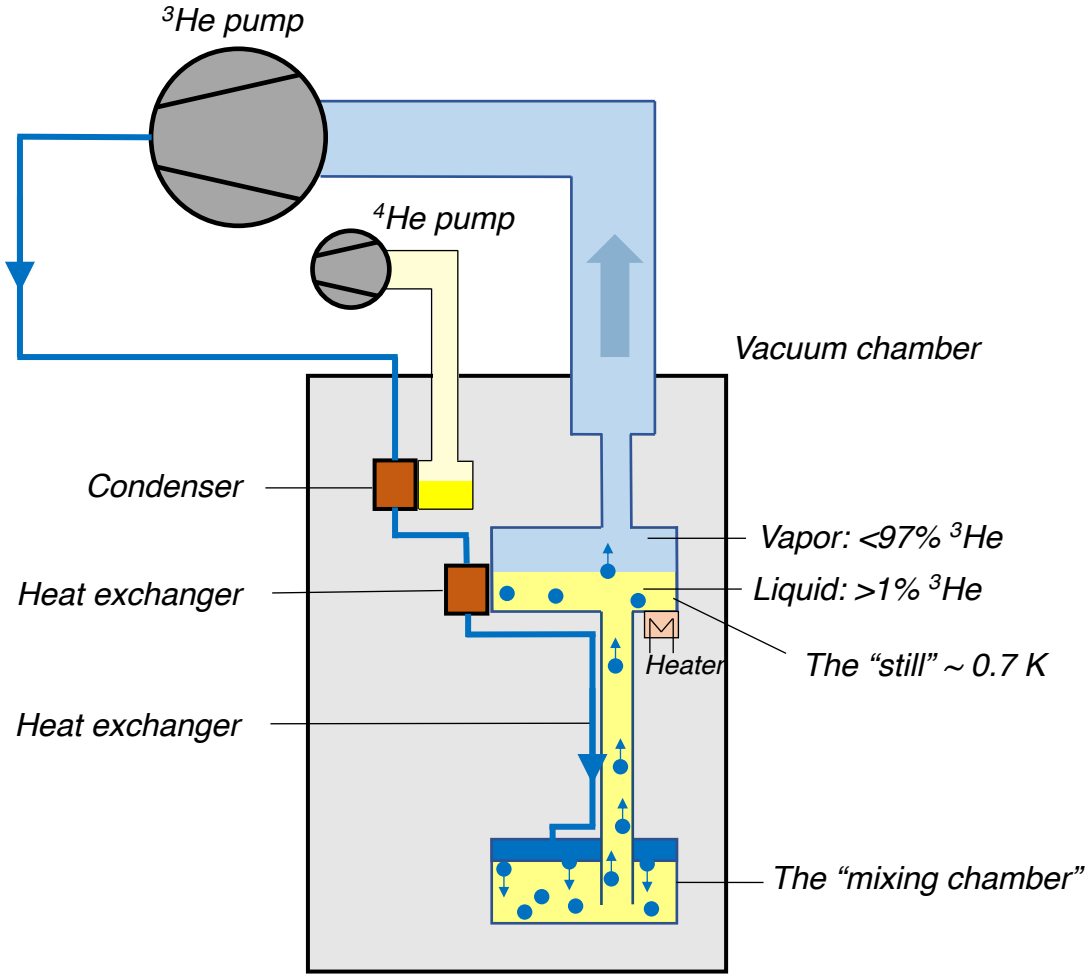


Dilution Refrigeration

The cooling process is made continuous by condensing the 3He gas and injecting it into the concentrated part of the mixing chamber.

"WET Fridge"

^{3}He pumped from the still is returned to the cryostat where it is condensed below 3 K by a ^{4}He evaporation refrigerator

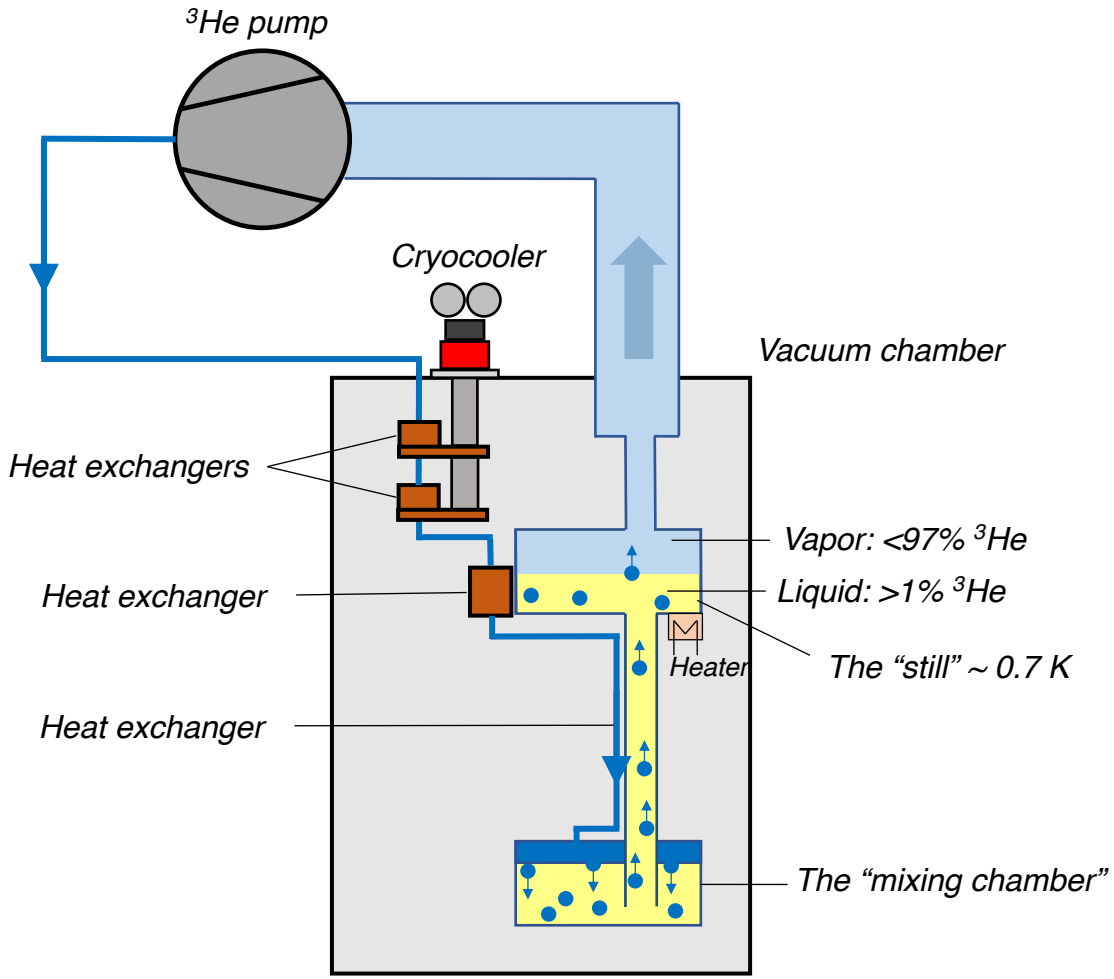


Dilution Refrigeration

Or by a cryocooler at a temperature around 3 K.

➔ “DRY Fridge”

“Cryogen-free fridge”



Dilution Refrigeration

In a recirculating dilution refrigerator, the cooling power equation must be modified to include the temperature of the ^{3}He at the *inlet of the mixing chamber*, T_i

$$\dot{Q} = \dot{n}[H_d(T_m) - H_c(T_i)]$$

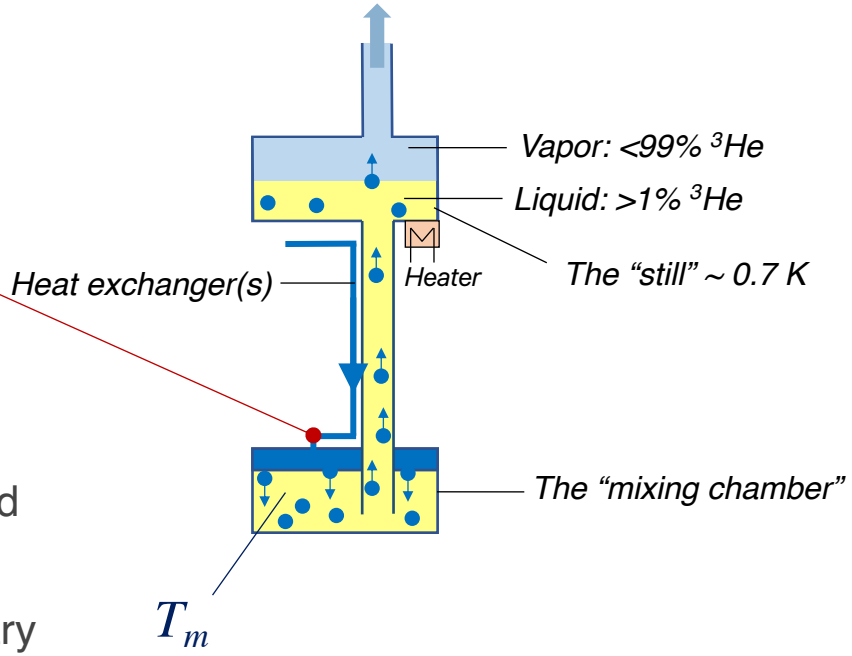
$$\approx \dot{n}\left[95T_m^2 - 11T_i^2\right]$$

The final heat exchanger (HX) between the concentrated and dilute phases deterrnines T_i.

The performance of this HX is limited by the thermal boundary ("Kapitza") resistance between its walls and the liquid helium,

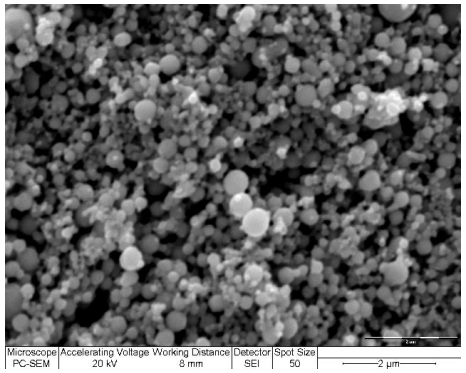
$$R_K = \frac{\rho}{A}T^{-3}$$

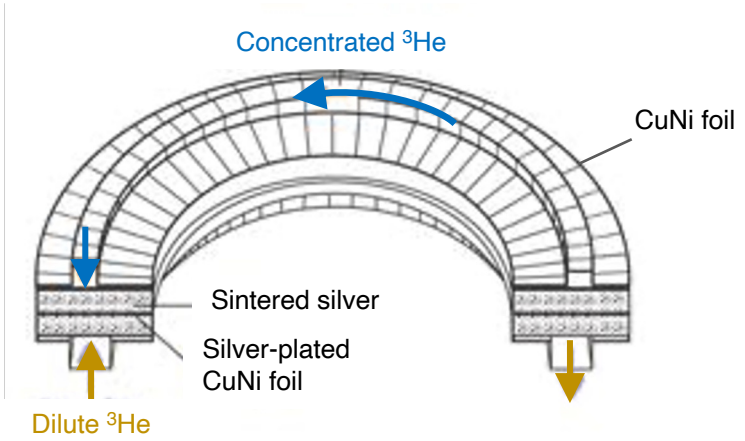
and its efficiency drops quickly with temperature.



Dilution Refrigeration

The solution is to coat the HX walls with a sinter of very fine copper or silver powder to increase the surface area *A* to a value of several square meters.

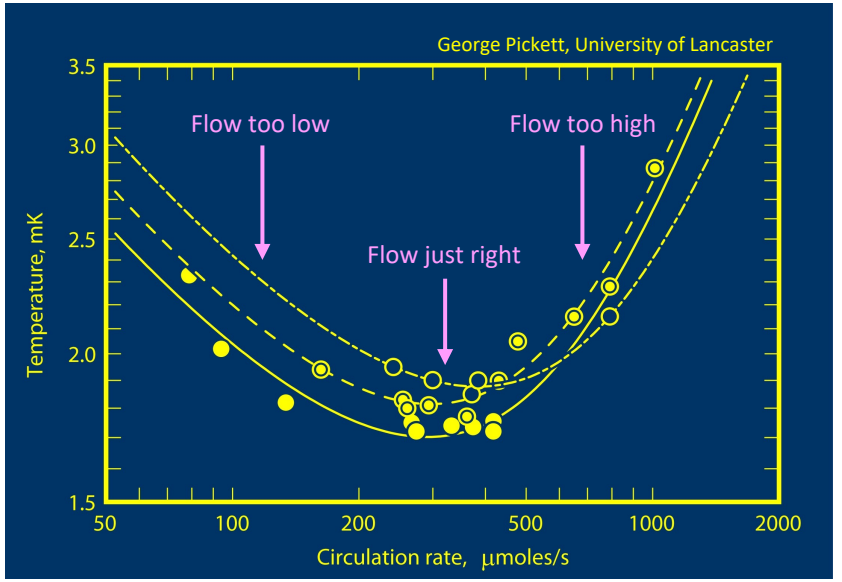




The heat exchanger must also be designed to optimize two competing sources of heat

- Axial thermal conduction (long & skinny)
- Viscous heating of the flowing liquids (short & fat)

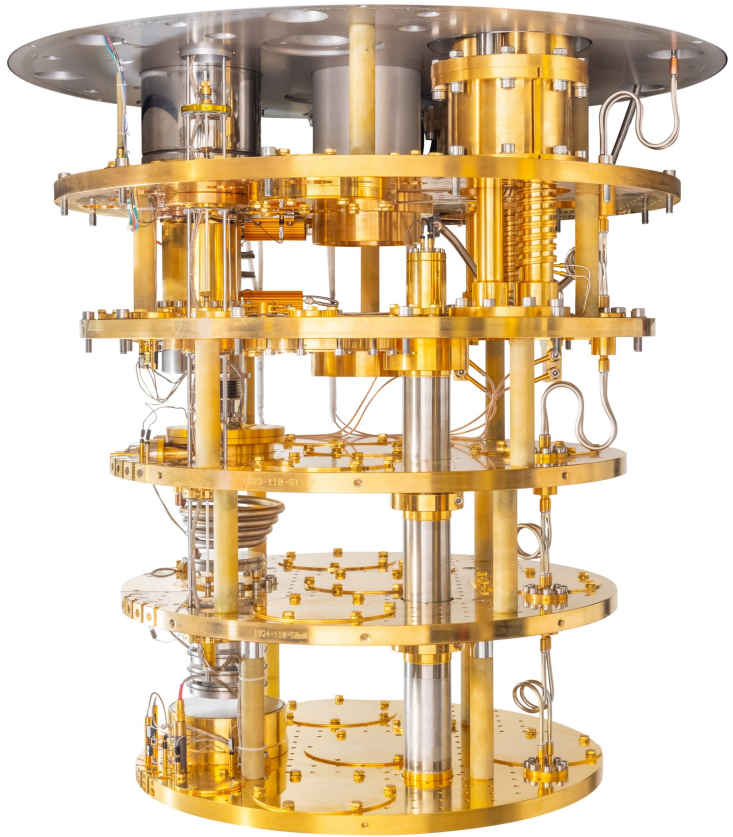
As a result, the lowest temperature is found at an optimum circulation rate that balances the total heat load (applied + viscous) with the cooling power available from the dilution process.



Dilution Refrigeration

Example: Leiden Cryogenics CF-CS110-1500 Maglev-2PT

- <7 mK base temperature
- Cooling power: >1.5 mW @ 100 mK



A Leiden Cryogenics DR cools the 1 ton CUORE neutrinoless double beta decay detector to 10 mK.

Dilution Refrigeration

The **global cryogen free dilution refrigerators market was valued at USD 112.1 Million in 2019** and is projected to reach **USD 211.4 Million by 2027**, expanding at a **CAGR of 9.1%** during the forecast period. Source: GrowthMarketReports.com

Key Players

- BlueFors (Finland)
- Oxford Instruments (UK)
- Leiden Cryogenics (Netherlands)
- Janis ULT (now FormFactor, USA)
- CryoConcept (AirLiquide, France)

Primary Drivers

- Quantum Computing
- Astrophysics
- Dark Matter Detection
- ~~Polarized Targets~~

Dilution Refrigeration

SOVIET PHYSICS

JETP

A translation of the Zhurnal Éksperimental'noĭ i Teoreticheskoĭ Fiziki.

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A METHOD OF PRODUCING VERY LOW TEMPERATURES BY DISSOLVING He^3 *IN* He^4

B. NEGANOV, N. BORISOV,[1)] and M. LIBURG[1)]

Joint Institute for Nuclear Research

Submitted to JETP editor December 10, 1965

J. Exptl. Theoret. Phys. (U.S.S.R.) **50**, 1445–1457 (June, 1966)

A method for the steady production of very low temperatures is described, based on the continuous dilution of liquid He^3 with liquid He^4 and the continuous separation of the isotopes at a higher temperature. At a He^3 circulation rate of about 1.84×10^{-4} mole/sec the temperature 0.1° K can be maintained when heat is added at the rate 1800 erg/sec. In the absence of an external heat input the temperature of the solution is maintained at ~ 0.056° K. Under constant external conditions the temperature fluctuations do not exceed ~ 0.001° K.

1. INTRODUCTION

THE existing method of producing very low temperatures by the adiabatic demagnetization of paramagnetic salts can be used, in principle, for the continuous production of very low temperatures by repeating periodically the magnetization and demagnetization of the salts. However, the practical realization of cyclical magnetic apparatus (which is, to begin with, very complicated) encounters fundamental difficulties and this technique can hardly be expected to yield refrigerators having a cooling capacity much above 1 erg/sec at ~0.01°K. An example is the refrigerator of Daunt et al.,[1] in which adiabatic demagnetization was repeated every two minutes. The lowest temperature attained with this apparatus was 0.2°K, and its refrigerating capacity was 50 erg/sec at 0.26°K.

The basic difficulty lies in the poor heat transfer at the interface between two media, where the thermal resistance increases proportionally to $1/T^3$. One can therefore hardly expect to achieve great success using this technique, and another direction must be investigated.

A new method of producing very low temperatures, which was proposed by London, Clarke, and Mendoza in 1960,[2] utilizes the latent heat of dilution of liquid He^3 by He^4. It has become clear that this technique enables the construction of apparatus possessing several advantages over the existing magnetic refrigerators. These advantages include simplicity, reliability, greater cooling capacity, temperature stability and controllability, and the possibility of cooling any material in any form without requiring the metallic cooling ducts that have interfered seriously with many measurements. This new method should therefore be utilized more extensively than the magnetic method, and will permit many investigations under pure conditions, at least at temperatures of the order of hundredths of a degree, which were previously unattainable for many solid state investigations.

Development of the solution (or dilution) method began in the Laboratory for Nuclear Problems at the end of 1961, when the need arose for a simple refrigerator to be used in nuclear polarization at

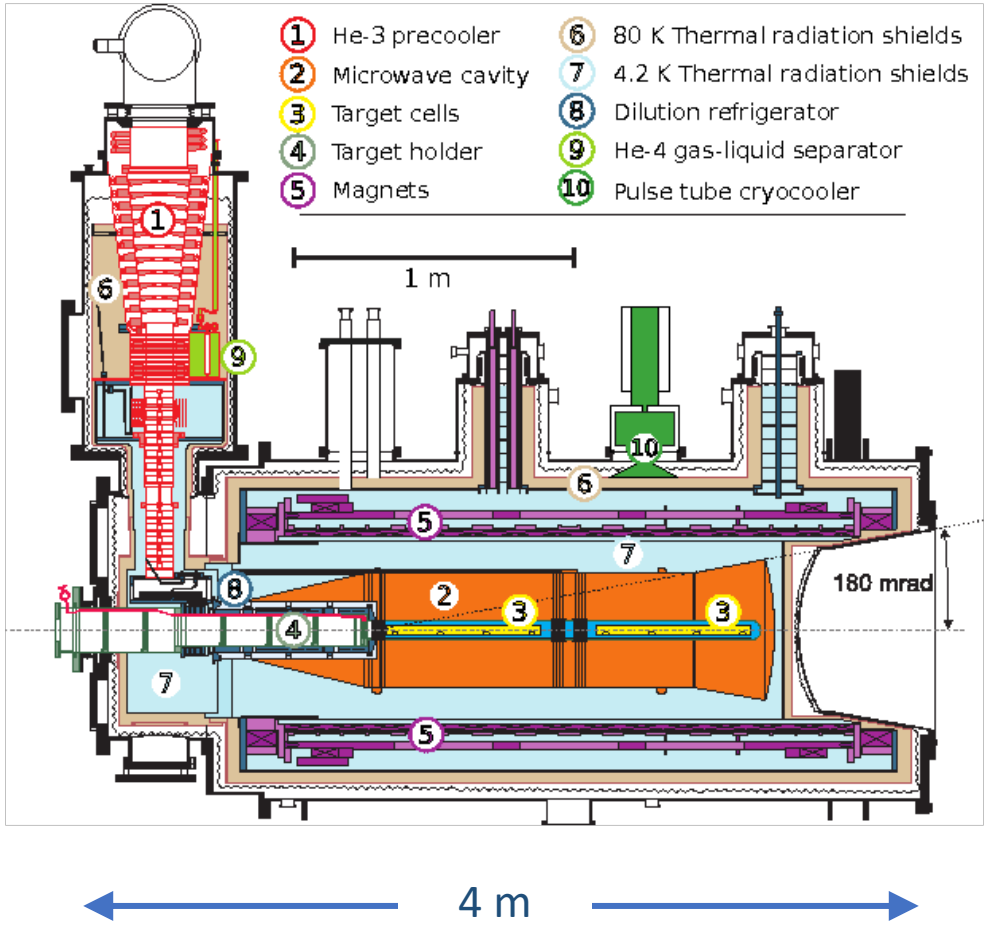
[1)]M. V. Lomonosov Moscow state University.

The very first working dilution refrigerator was built at Leiden in 1964. It cooled to 0.22 K.

The second DR (Dubna, 1966) was four times colder, 0.056 K. It was built for a dynamically polarized target…

Dilution Refrigeration

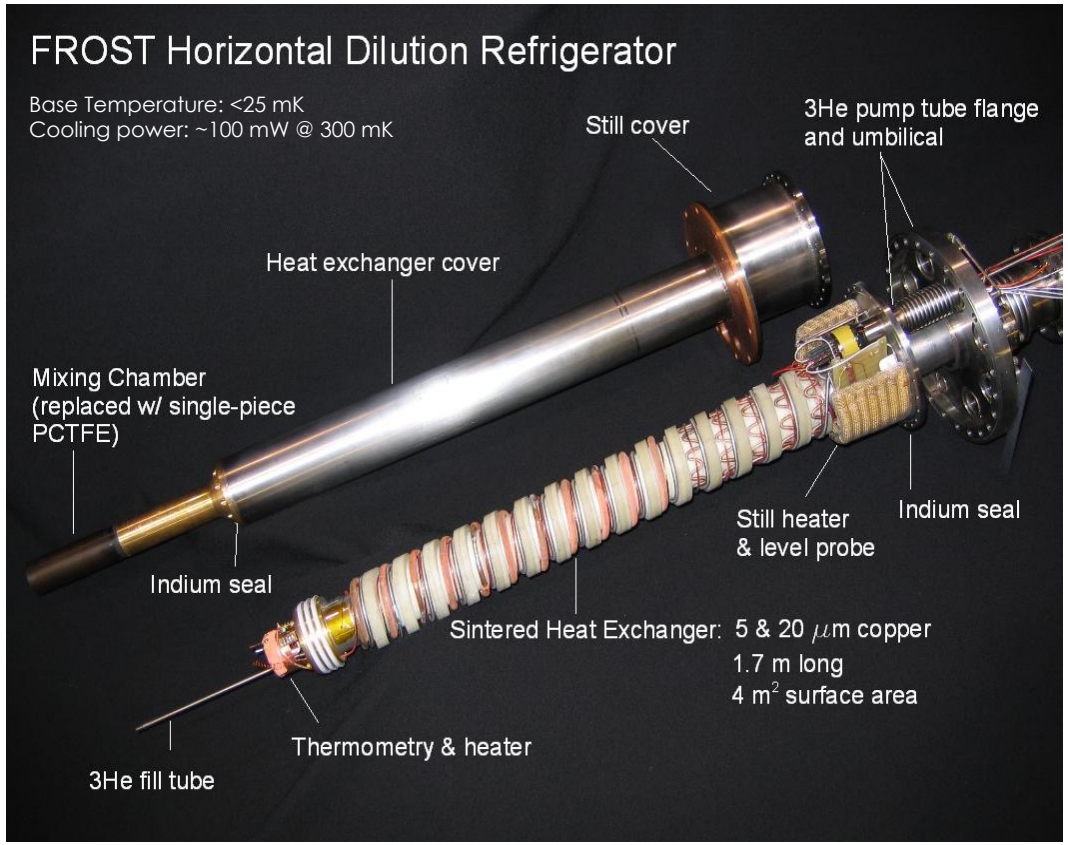
The most powerful (not coldest) dilution refrigerators have been built for polarized targets.



The SMC/COMPASS fridge (CERN, 1999) has a cooling power of 400 mW at 0.3 K.

Dilution Refrigeration

The most powerful (not coldest) dilution refrigerators have been built for polarized targets.



The FROST dilution refrigerator (JLab, 2012) has a cooling power of 10 mW at 0.1 K.

Summary

- Four “bench-top” refrigeration schemes for temperatures below 4 K were reviewed.
  1. Cryocoolers
  2. Helium Evaporation
  3. Adiabatic Demagnetization
  4. ^{3}He - ^{4}He Dilution
- With the exception of ADR, each has been used to cool targets at Jefferson Lab
- Each has its advantages and disadvantages.
- The choice has to made for each application based on the necessary cooling requirement at the desired temperature.
- Dilution Refrigeration remain the only option for continuous cooling below about 300 mK
- Most commercial DRs are designed to provide a very low base temperature (<10 mK)
- DRs built for polarized targets are usually optimized to provide much more power at elevated temperatures (>100 mK) at the cost of a higher base temperature (~20 mK)

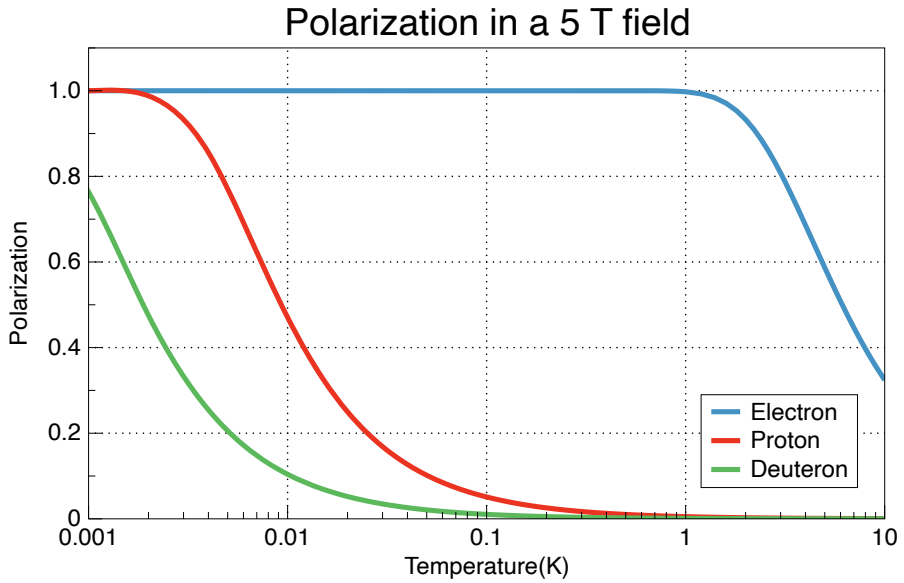
Ultralow temperatures & targets

Polarized solid targets are a dense source of nuclear spins and are in frequent use at JLab

- Used in all three halls during the 6 GeV era
- Approved for experiments in all *four* halls during 12 GeV era

Brute Force Polarization

The easiest way to polarize the nuclei in a solid is to cool the solid to an ultra-low temperature and use a high magnetic field to align its spins



Unfortunately, reasonable nuclear polarizations can only be achieved at extremely low temperatures.

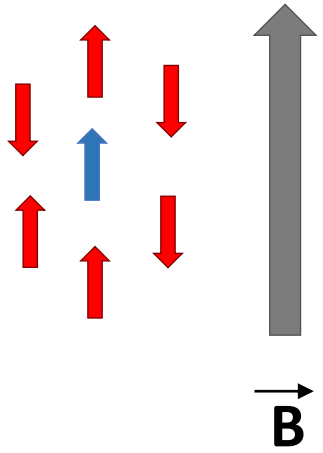
Heating limits these targets to very low intensity beams.

Notice that *electrons are far easier to polarize!*

Ultralow temperatures & targets

Dynamic Nuclear Polarization

A smarter way to polarize nuclear spins in an insulating solid
Unpaired electron spins (paramagnetic radicals) are highly polarized at high B/T, e.g. 5T/1K
Microwaves near ESR frequency transfer electronic polarization to the nuclei



Sample is prepared with **paramagnetic radicals** (~10^{19} cm^{-3})

Radicals are polarized at **low temperature and high field**, $\tanh(\mu_e B/kT) > 99\%$

Nuclei remain **unpolarized,** < 1%

Ultralow temperatures & targets

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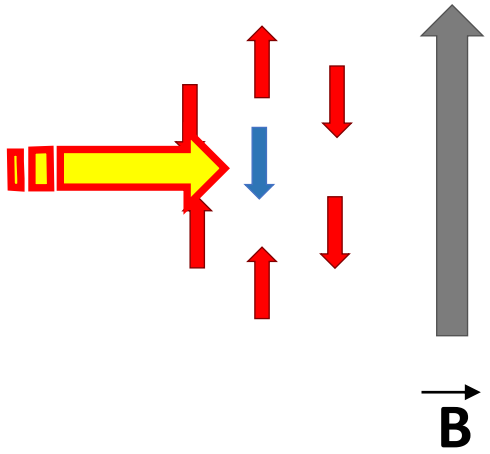

Microwaves near the radicals resonance frequency induce mutual electron/nuclear spin flips.

Freq. matching: $\nu = \nu_e \pm \nu_N$

The electron flips back quickly to its equilibrium orientation (short T_1).

The nucleus does not (long T_1).

Ultralow temperatures & targets

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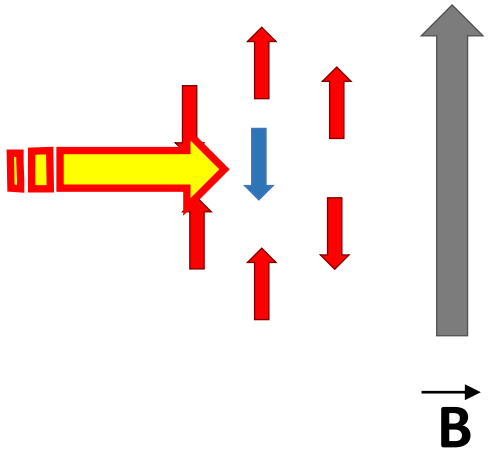

Microwaves near the radicals resonance frequency induce mutual electron/nuclear spin flips.

Freq. matching: $\nu = \nu_e \pm \nu_N$

The electron flips back quickly to its equilibrium orientation (short T_1).

The nucleus does not (long T_1).

Eventually, the nuclei in the vicinity of the radical become polarized.

Ultralow temperatures & targets

Dynamic Nuclear Polarization

With DNP we can achieve proton polarizations > 90%
and deuteron polarizations > 80%

Typical operating conditions for electron beam experiments at JLab are 5 T and 1 K

In an extreme case, we have operated a polarized target at 0.5 T and 0.02 K.