Vacuum techniques (down to 1 K)

- For isolation (deep Knudsen regime)
  - liquid helium dewar / inner vacuum jacket
- Leak testing at level 10^{-11} Pa m^3/s (10^{-10} mbar l/s = 3ℓ NTP in 10^6 a)
  - liquid helium dewar & transfer syphon
  - inner vacuum jacket
  - helium evaporator (pot)
  - dilution refrigerator
  - helium sample chamber (if exists)
  - ^{3}He circulation (tubing, pumps, valves, storage tanks, pressure gauges, cold traps, ...)
- Refrigeration by evaporation cooling
  - helium evaporators
  - dilution refrigerator
  - (– pulse tube coolers)

System design has to permit required flow rate at given vapor pressure

- ^{4}He: ~ 1 … 10 mmol/s @ p ~ 1 mbar
- ^{3}He: ~ 0.1 … 5 mmol/s @ p ~ 0.01 … 0.1 mbar

- Heat engines/refrigerators

Basic vacuum concepts I

Vacuum	Pressure (torr)	Number Density (m^{-3})	M.F.P. (m)	Surface Collision Freq. ($m^{-2} \cdot s^{-1}$)	Monolayer Formation Time (s)
Atmosphere	760	2.7×10^{25}	7×10^{-8}	3×10^{27}	3.3×10^{-9}
Rough	10^{-3}	3.5×10^{19}	0.05	4×10^{21}	2.5×10^{-3}
High Rubber O-rings	10^{-6}	3.5×10^{16}	50	4×10^{18}	2.5
Very high Copper gaskets	10^{-9}	3.5×10^{13}	50×10^{3}	4×10^{15}	2.5×10^{3}
Ultrahigh Baking	10^{-12}	3.5×10^{10}	50×10^{6}	4×10^{12}	2.5×10^{6}

Basic vacuum concepts II

$$R = N_A k_B$$

$$PV = N_{moles} RT \Longrightarrow PV = N_{molecules} k_B T \Rightarrow N_{molecules} = \frac{PV}{k_B T}; n = \frac{P}{k_B T}$$

molecular mean speed

of the magnitude of the velocity

$$\langle v \rangle = \sqrt{\frac{8 k_B T}{\pi m}}$$

$$\langle v \rangle = \sqrt{\frac{3 k_B T}{m}}$$

root mean square of the total velocity

$$RT: \langle v \rangle_{H2} = 1761 \frac{m}{s}$$

$$LHe: \langle v \rangle_{H2} = 213 \frac{m}{s}$$

impingement rate

$$\varphi = \frac{1}{4} n \langle v \rangle = \frac{1}{4} n \sqrt{\frac{8 k_B T}{\pi m}}$$

$$\mathrm{RT} - 10^{-8} Pa - N_2$$

$$\varphi = 1.1\ 10^{11} \frac{collisions}{cm^2 s}$$

mean free path

$$l = \frac{1}{\sqrt{2}\, n\, \pi\, \delta^2}$$

$$\mathrm{RT} - 10^{-8} Pa - N_2$$

$$l \cong 700\ km$$

Knudsen number $\Rightarrow$ $K_n = \frac{l}{D}$

D is a characteristic dimension of a vacuum system

Basics of flow regimes

K_n range	Regime	Description
$K_n > 0.5$	Free molecular flow	The gas dynamics are dominated by molecular collisions with the walls of the system
$K_n < 0.01$	Continuous (viscous) flow	The gas dynamics are dominated by intermolecular collisions
$0.5 < K_n < 0.01$	Transitional flow	Transition between molecular and viscous flow

Outgassing/Degassing

Metals

After state-of-art surface cleaning:

- If not heated in situ: mainly **H_2O** for the first months in vacuum, then also H_2.

$$q_{H_2O} \approx \frac{3 \times 10^{-9}}{t[h]} \left[\frac{mbar\ l}{s\ cm^2}\right]$$

  Source of H_2O is recharged after each venting to air (**adsorption/desorption**).

- If heated in situ (baked-out): mainly **H_2**. The outgassing rate can be assumed as constant; it depends on the accumulated effect of the previous thermal treatments.

H atoms travel in average 4 μm per day in austenitic stainless steels

Organics (Polymers)

- High solubility of gas in the bulk, in particular **H_2O.**
- In general, the outgassing process is dominated by H_2O release.
- In the initial phase of pumping:

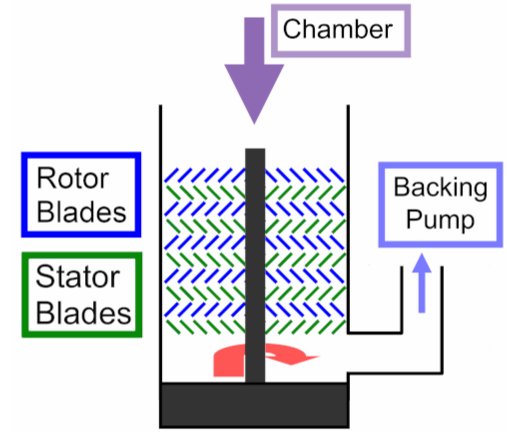
$$q_{H_2O} \propto \frac{1}{\sqrt{t}}$$

- Heavier gas molecules can be outgassed (remnant of polymerization, fraction of polymeric chains).
- The **permeation** of light molecules is not negligible, in particular He.

H_2O molecules move along about 20 μm per day in a high-performance polymer.

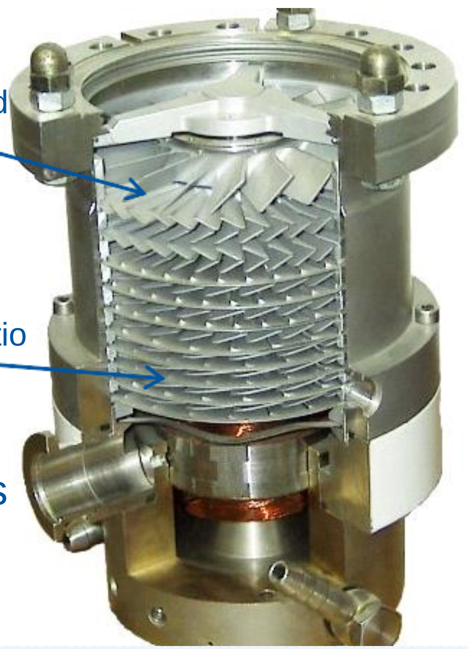
https://outgassing.nasa.gov/

Turbomolecular pumps

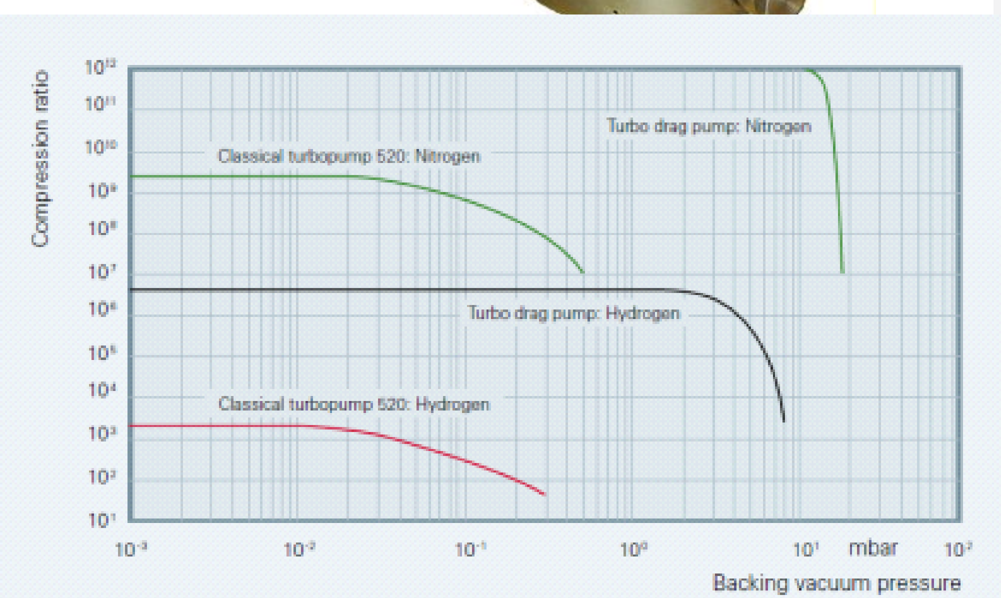


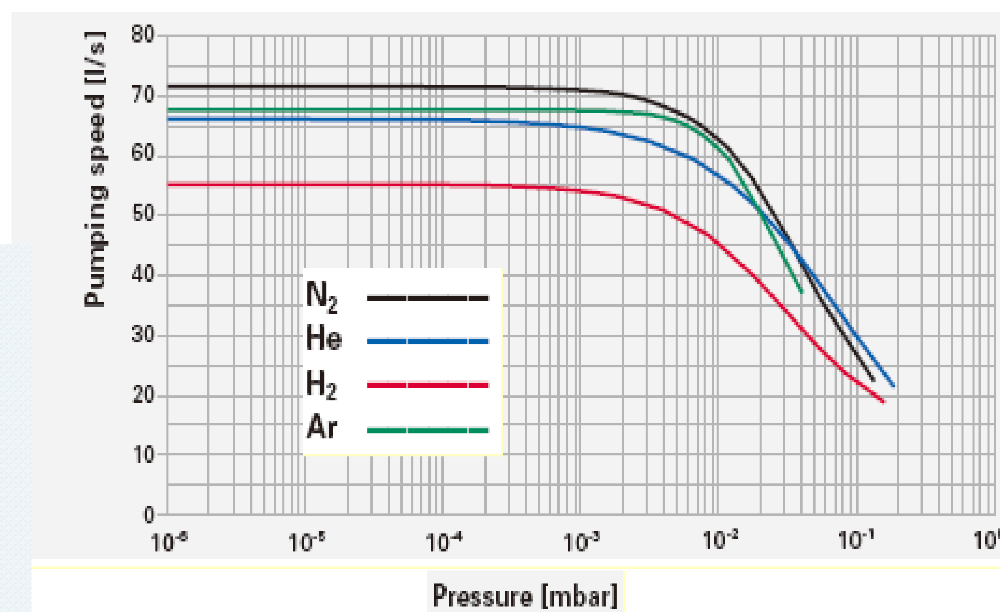
TMP pumping speeds are in the range from 10 l/s to 25,000 l/s.

Their ultimate pressure (H_2) is of the order of 10^{-10}, 10^{-11} mbar



- mean free path has to be larger than the blade distances.

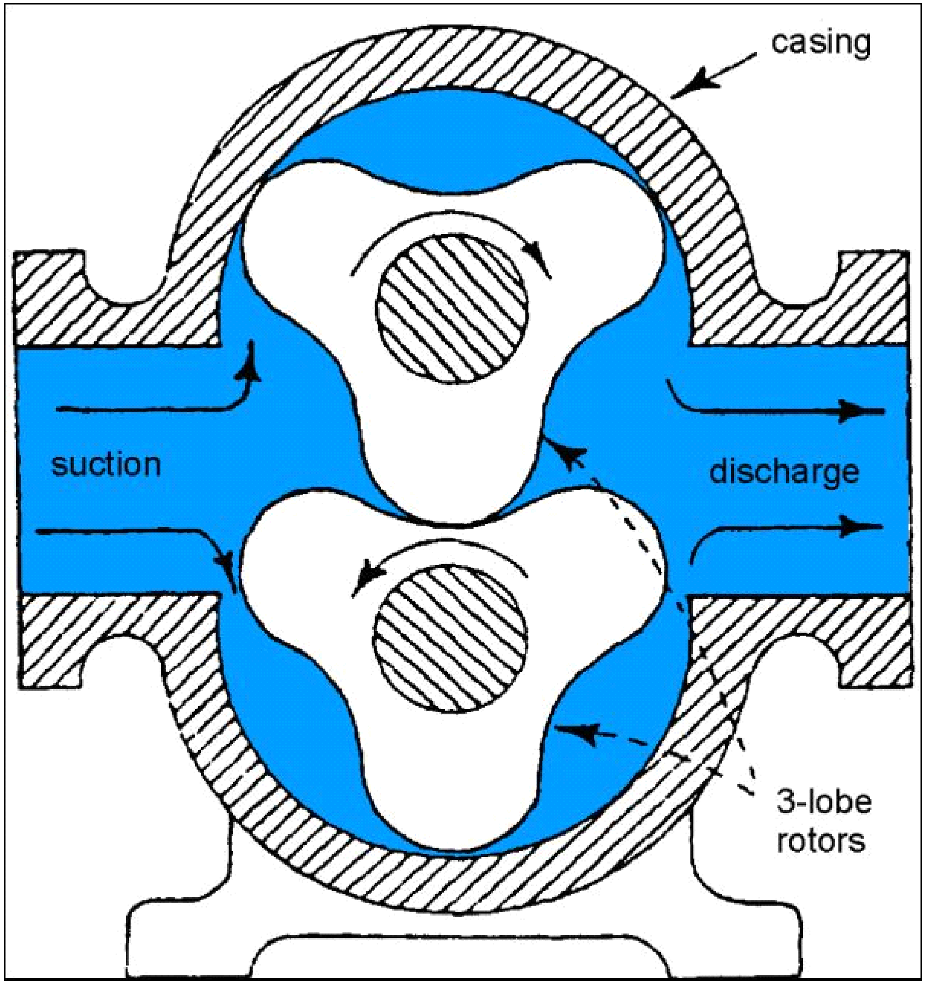




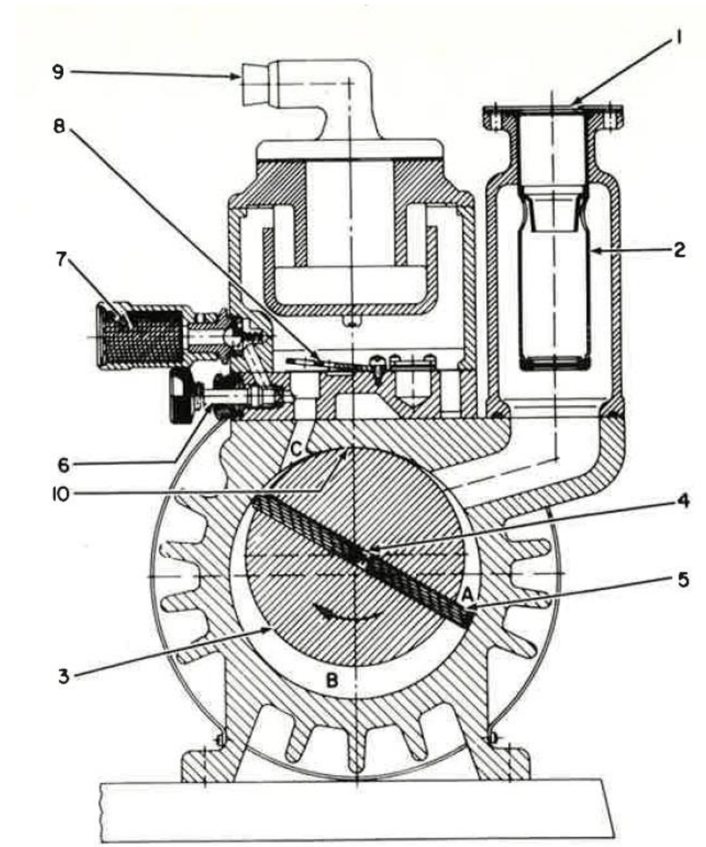
Courtesy of Pfeiffer Vacuum
http://www.pfeiffer-vacuum.com

$Q = pS$ Throughput

Roots blowers



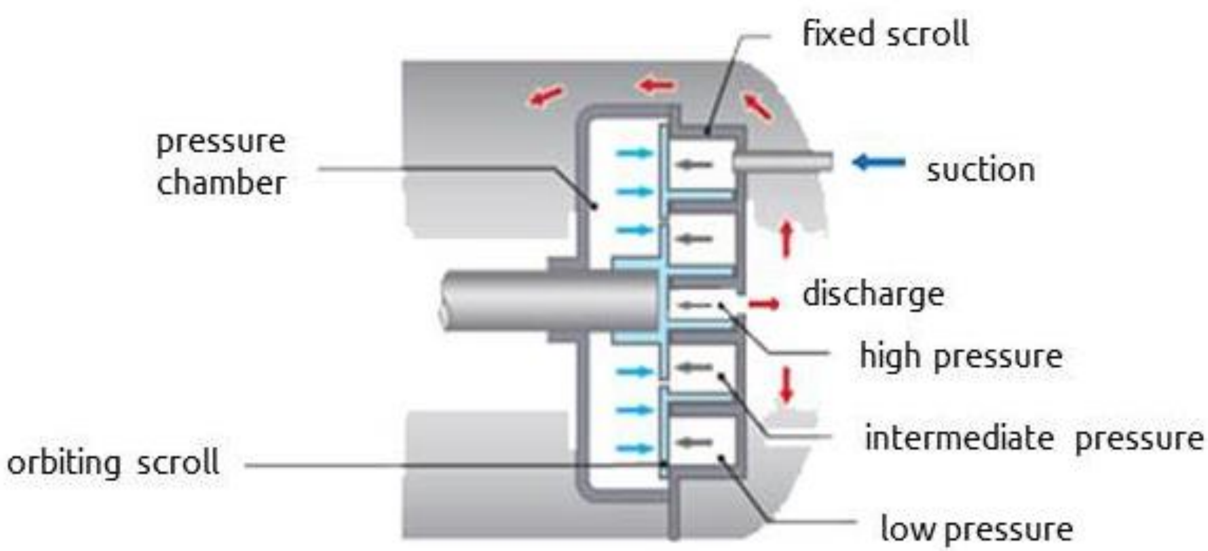
- Compression ratio not as good as for mechanical rotary pumps
- Requires a good backing pump

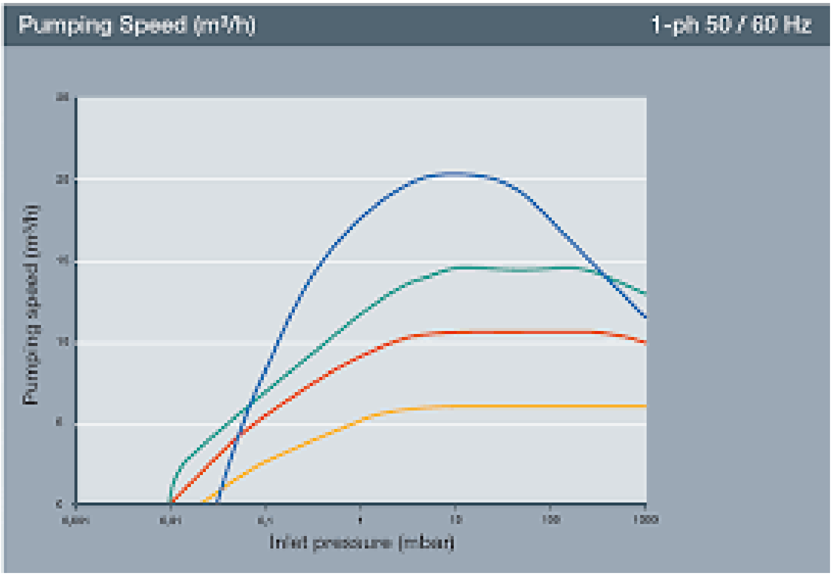


- Can move air very rapidly
- Often no oil seal
- Used often as backing of huge booster pumps

- Multistage roots pumps replaced by scroll pumps in LTL

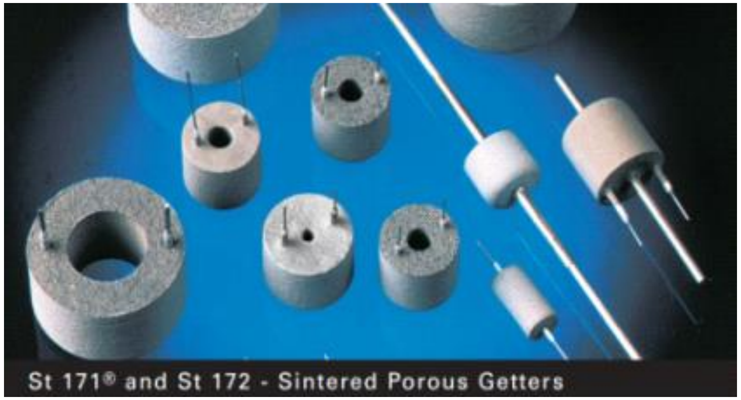
Scroll pumps





Modern getter pump

Full pumping speed after heating at 400°C for 45’ or 300°C for 24h

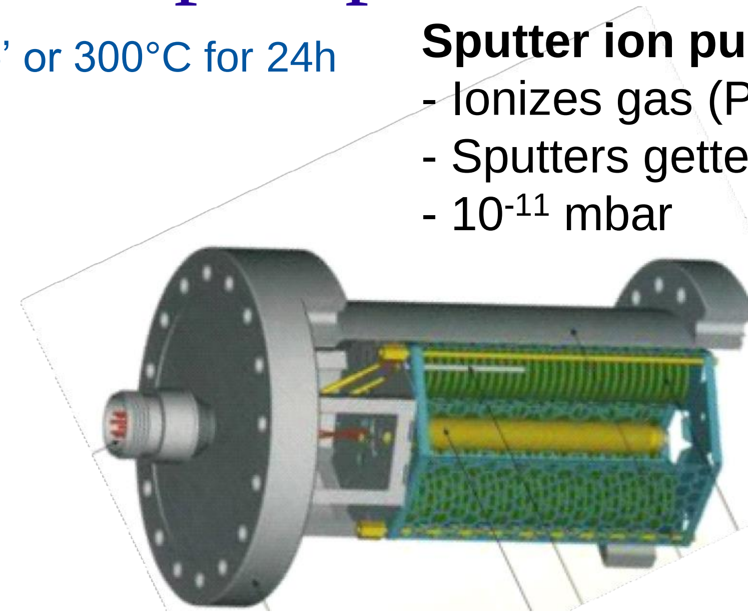


Getter materials do not pump rare gases and methane at room temperature.

Sputter ion pump

- Ionizes gas (Penning trap)
- Sputters getter metal
- 10^{-11} mbar

Element
Zr
V
Fe



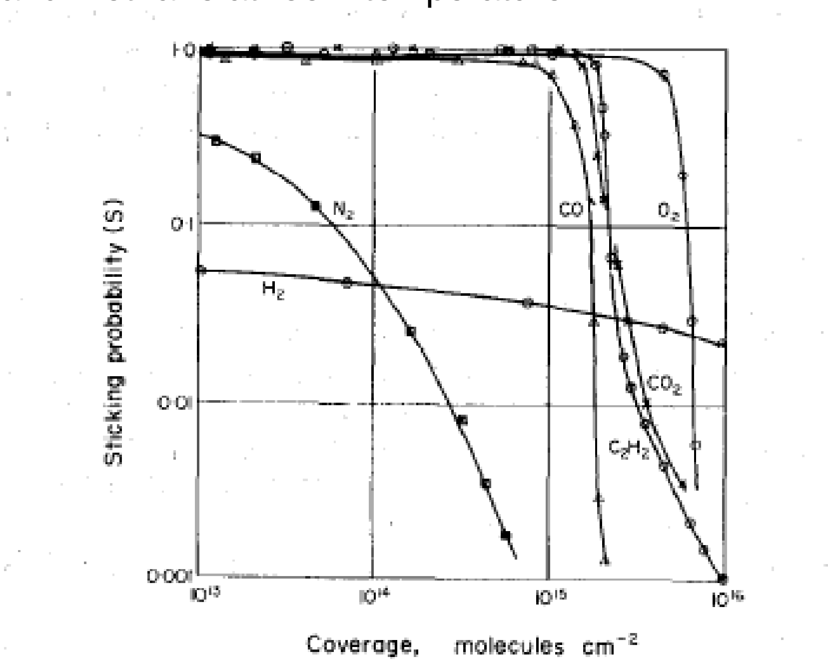
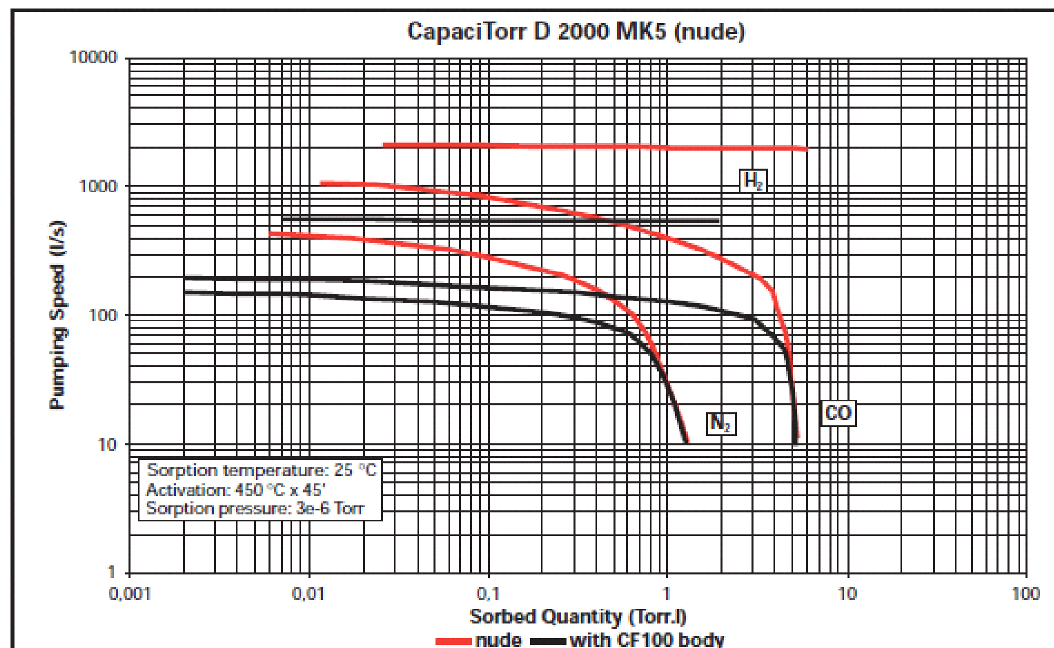


Figure 4. Room temperature sorption characteristics for pure gases on batch evaporated clean titanium films.

Ti sublimation pump

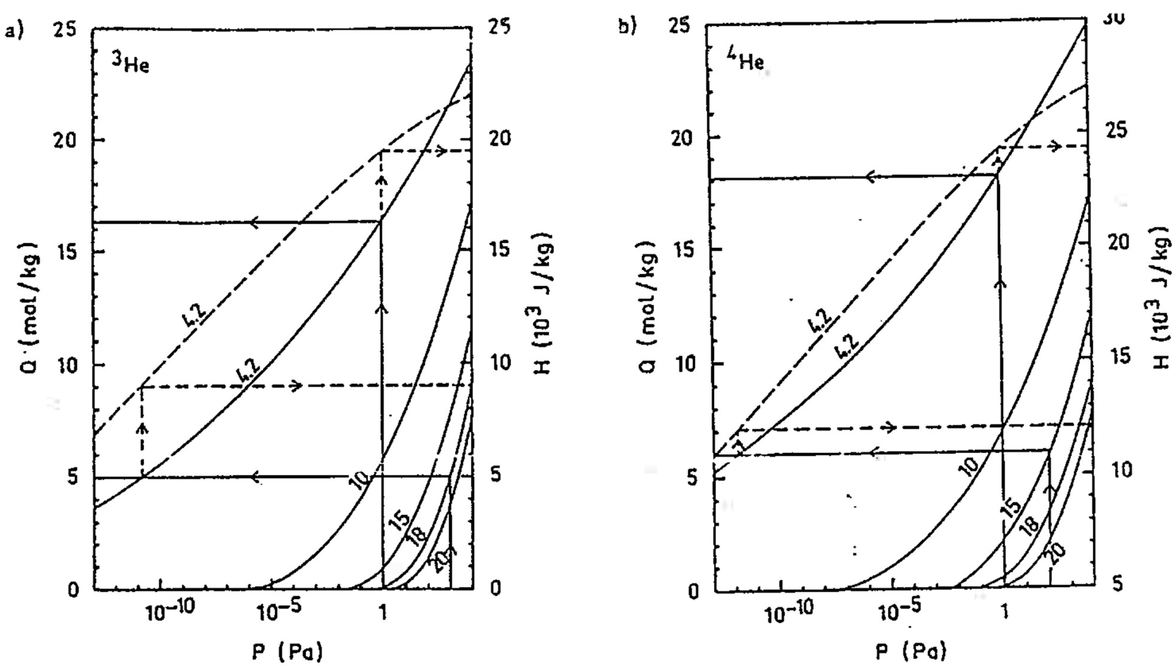


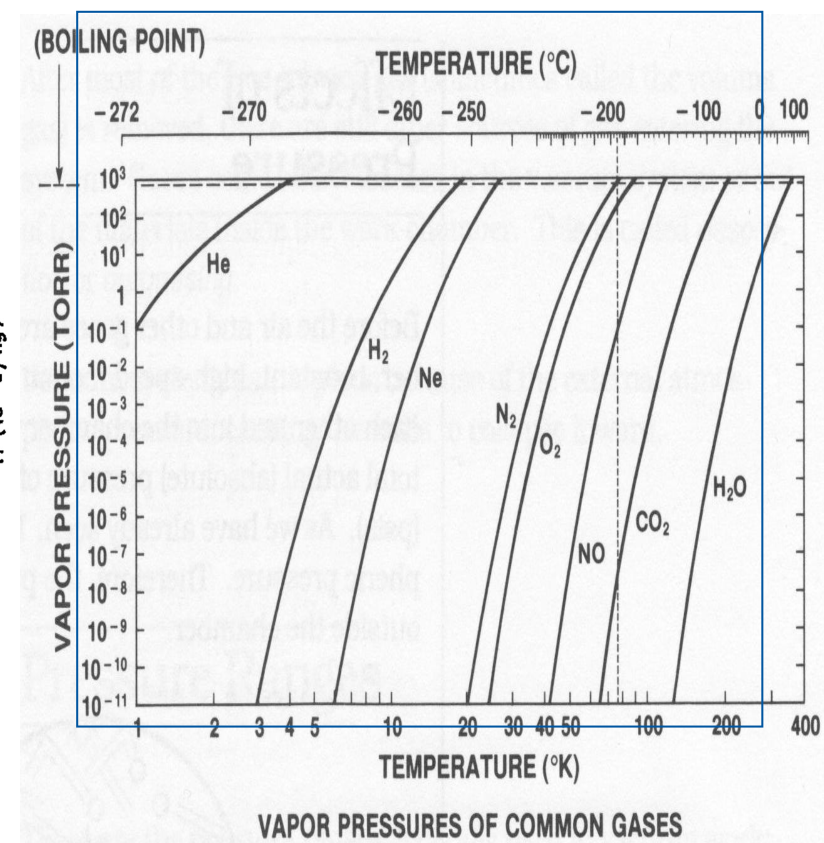
- Chemisorption
- Physisorption (lighter gases)

Cryoadsorption pumps

Best possible vacuum is obtained by **cryoadsorption on charcoal**

- activated charcoal has strong binding potential on surface for gas molecules
- huge surface area per mass unit (~ **600 m^2/g**)
- when it is cold (< 10 K) it adsorbs anything, also He (but saturates)
- heat to room temperature => release gases He, H_2, N_2, O_2 (regeneration)
- used for securing vacuum in storage dewars
- used also for purifying circulating He in cryostats (kept in LN2)
- can be used to take care of SMALL leaks in inner vacuum jacket
- heat of adsorption is considerable





Helium leak detector

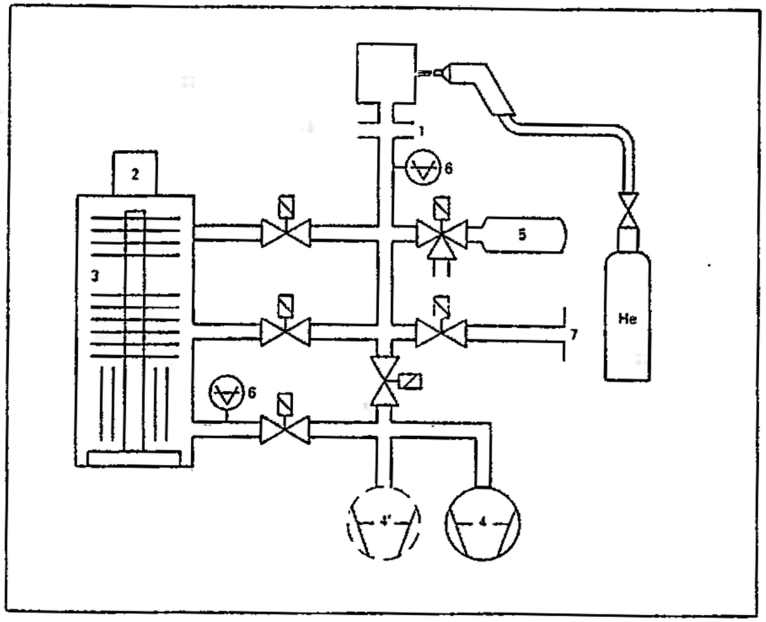
Helium is used for leak detection because

- inert gas does not react in the system
- small atom, penetrates easily through the tiniest holes
- does not appear in natural environment (low background)
- light, easy to determine with a mass spectrometer

Note the diffusion of He through plastics & rubber at room temperature

Modern units have several modes of operation for small and big leaks

- **direct mode**
- **counterflow mode**
- rough mode
- sniffing mode



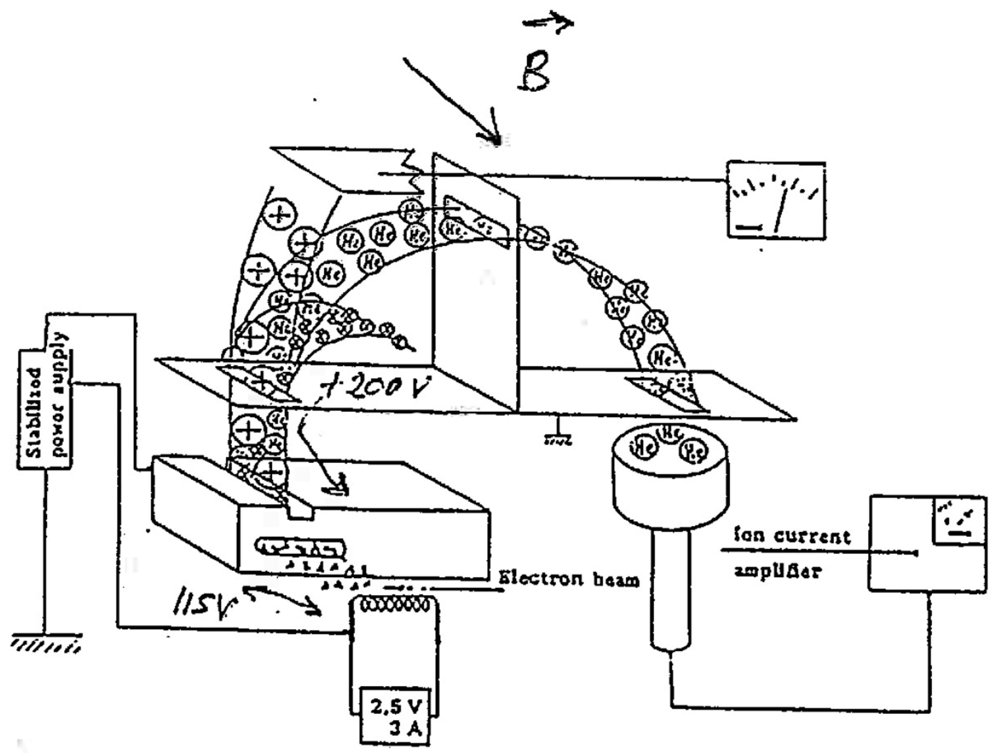
1. Twin-Flow™ test connection
2. Mass spectrometer
3. Special turbopump
4. Internal pump: rotary vane for HLT 260, membrane for HLT 270
4' External pump: scroll for HLT 275, customer-side for HLT 265
5. Test leak
6. Pirani gauge
7. Venting connection

Helium leak detector (cont.)

Mass spectrometer for leak detectors:

- hot cathode emits electrons, which ionize the surrounding gas
- ions are accelerated by electric field
- accelerated ions are deflected by magnetic field
- can be set to detect mass numbers 2 (H_2), 3 (^3He), or 4 (^4He)
- calibrate by means of a standard leak:

  He gas diffusing through
  a sealed Pyrex-glass ampule



Leak testing

1) **Huge leaks**: impossible to get below 10^{-3} mbar or p returns above 10 mbar – bubble test, soap test (if applying overpressure is possible)
– solvent exposure test (monitor pressure when putting alcohol on suspect spot)
– blue stick test (cover the hole by blue stick and see if the rest is ~ OK)

2) **Big leaks**: pump OK, pressure stays low, but leak detector level is high – blue stick method may work
– sniffing technique, if test chamber can be filled with He

3) **Small leaks**: leak signal low (< 10^{-8} mbar l/s), but reacts to He exposure
– injection needle search; expose to He minimum area possible at a time
– bag test, put parts of the system (if possible) into a bag filled by He

4) **Tiny leaks**: leak signal low (< 10^{-9} mbar l/s), no response at room temperature – warm systems are OK at this level
– cold systems must be tested at LN2 temperature and 10 K; tiny leaks show up

5) **Superleaks**: only appear when superfluid helium is present in the system
– nightmare (remake the faulty component or seam)

Evaporation cooling

Ideal gas assumption:

1) particle diameter a << distance between particles $1/n^{1/3}$
2) no interactions between the particles
3) collisions between the particles are elastic

- – mean free path $l = 1/(2^{1/2}\,\pi\, a^2\, n)$
- – pressure $p = n\, k_B T$
- – viscosity $\eta = [8\, m\, k_B T/(9\,\pi^3\, a^4)]^{1/2}$
- – average velocity $\bar{v} = [8\, k_B T/(\pi\, m)]^{1/2}$

These apply well to helium gas ($a \sim 0.22$ nm) in all considered cases
Efficiency of evaporation cooling from Clausius-Clapeyron relation:

$$\frac{dp}{dT} = \frac{\Delta S}{\Delta V} = \frac{L}{T\Delta V}$$

- – **latent heat** $\boldsymbol{L = T\Delta S}$ depends weakly on temperature
- – $\Delta V = V_g - V_l \approx V_g = N_A k_B T/p = R\,T/p$

$dp/p \approx L/R\; dT/T^2 \;\Rightarrow\; \boldsymbol{p \sim e^{-L/(RT)}}$

$\left\{ \begin{array}{l} {}^3\mathrm{He}:\; L_3/R \sim 2.5\ \mathrm{K} \\ {}^4\mathrm{He}:\; L_4/R \sim 8.5\ \mathrm{K} \end{array} \right.$

Sustainable mass flow depends on

– dimensions of the tubing; p_p (pump) <~ p_v (vapor)

– capacity of the vac pump; $\mathbf{dN/dt} = p_p/k_B T\, \mathbf{dV/dt}$ (pump) $\propto$ $\mathbf{p_p}$ (pump)

Cooling power is given by $\mathbf{dQ/dt = dN/dt\, L/N_A \sim e^{-L/(RT_v)}}$ **(vapor)**

Mechanical rotary pump (10 ... 100 m^3/h) is enough for a decent ^{4}He evaporator
Small dilution fridge can be run by a good turbo pump + mech. rotary pump (scroll)
Powerful dilution fridge requires heavy duty pumps:

– oil vapor booster
+ roots pump
+ mechanical rotary => **50 000 m^3/h**

Viscous regime:

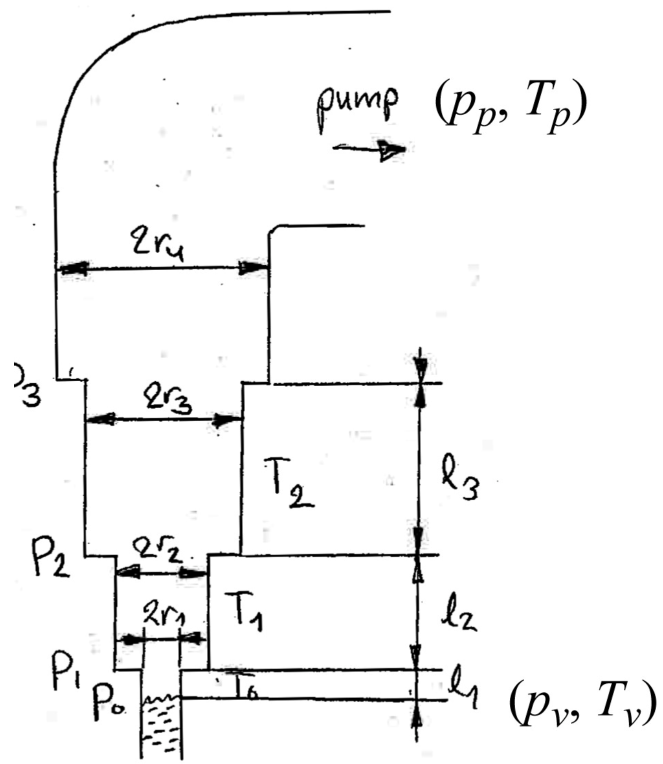
viscosity: $\tau = \eta\, dv_y/dx$

$$p_{i-1}^2 - p_i^2 = \frac{16\,\eta_i\, l_i\, \dot{n}\, R T_i}{\pi r_i^4}$$

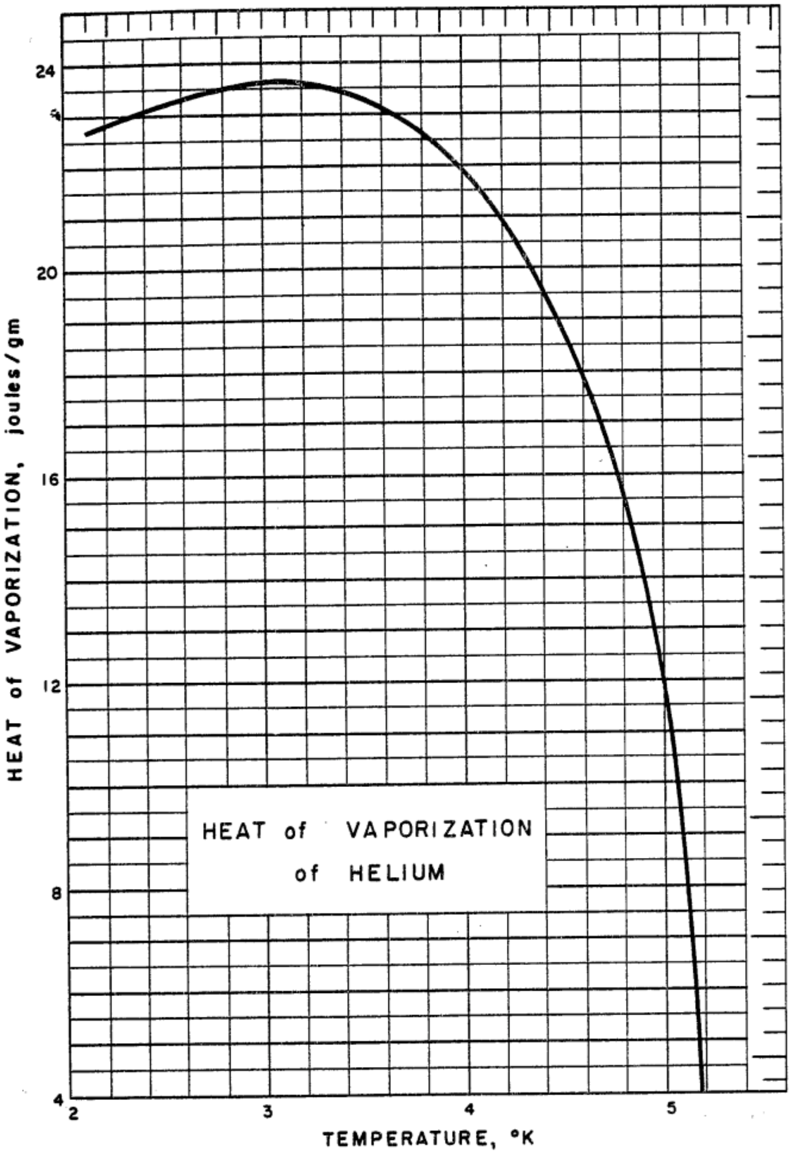
Conductance: $$G = \frac{\pi d^4}{256 \eta L}(p_1 + p_2) \quad [m^3/s]$$

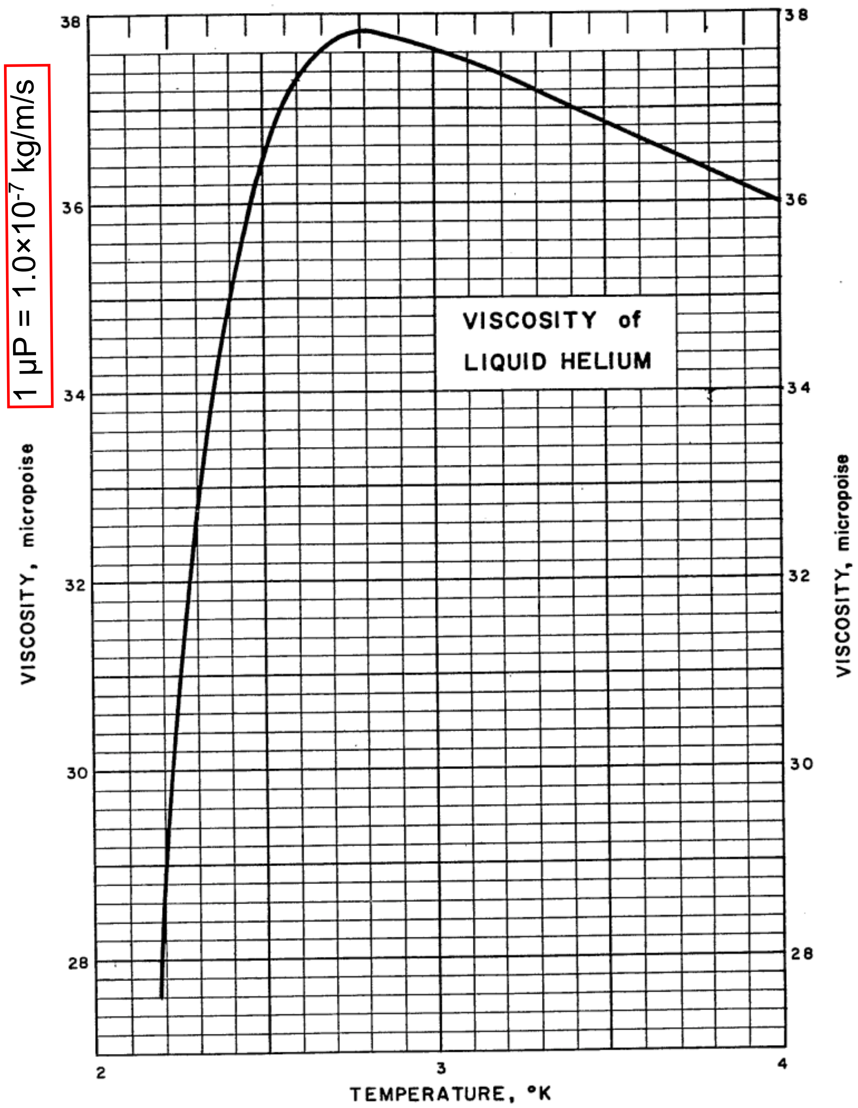
Molecular regime:

$$p_{i-1} - p_i = \frac{3\, l_i\, \dot{n}\, R}{4 r_i^3}\sqrt{\frac{m T_i}{2\pi k_B}}$$



Data on liquid ^{4}He





Air: 180 μP

^{4}He evaporator (pot)

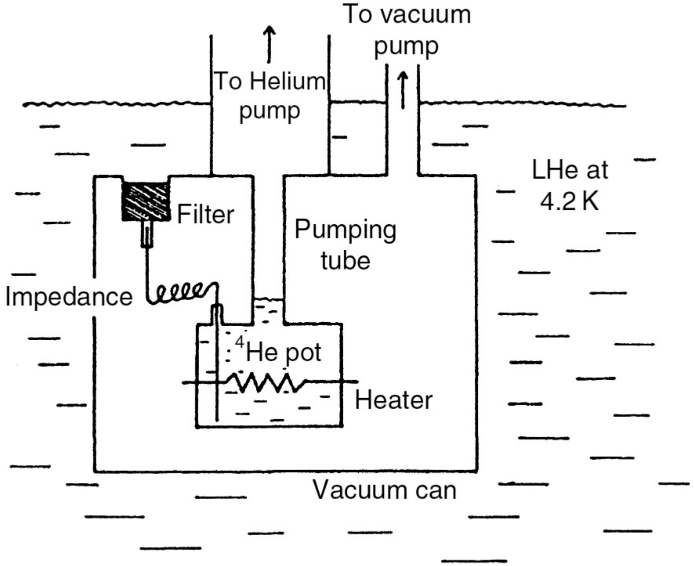
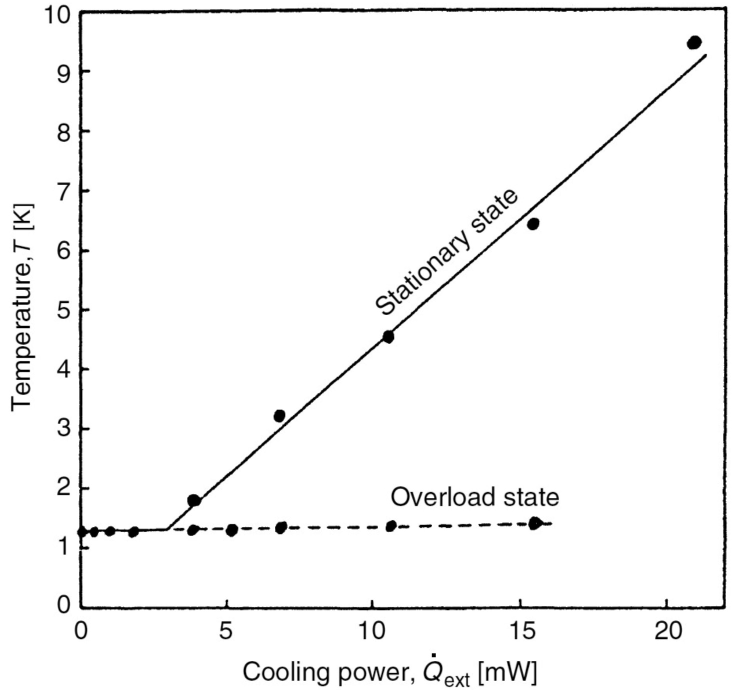
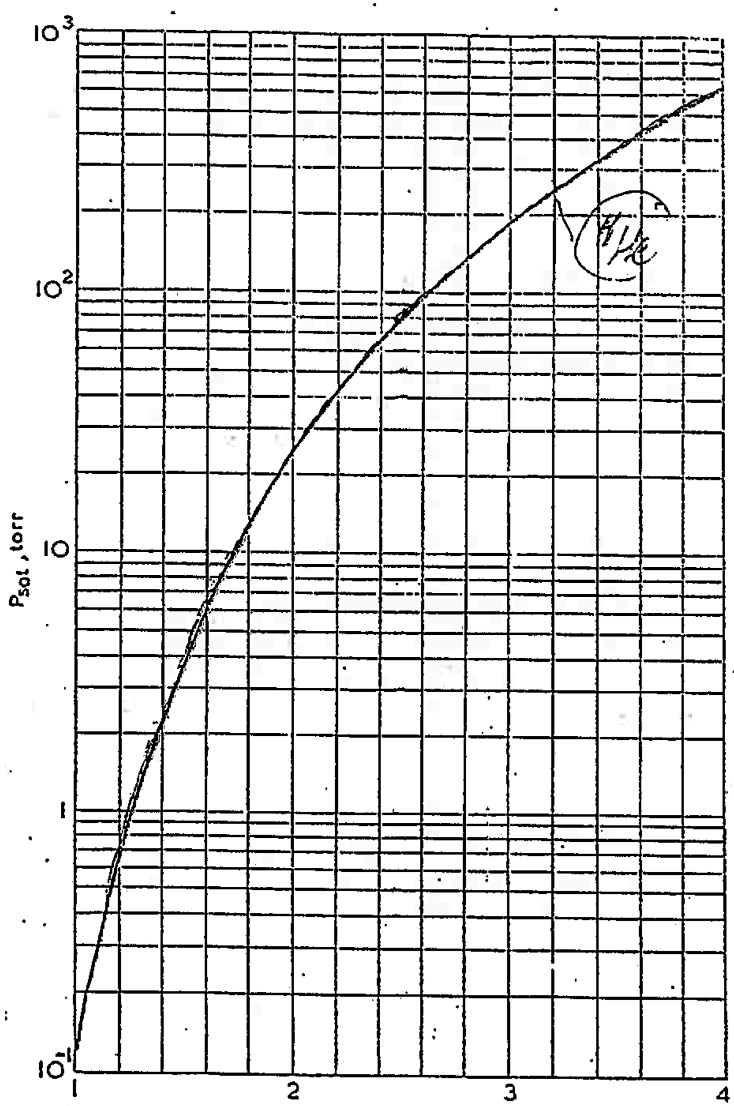


Fig. 5.4. A continuously operating ^{4}He refrigerator for the temperature range between 1.3 and 4.2 K

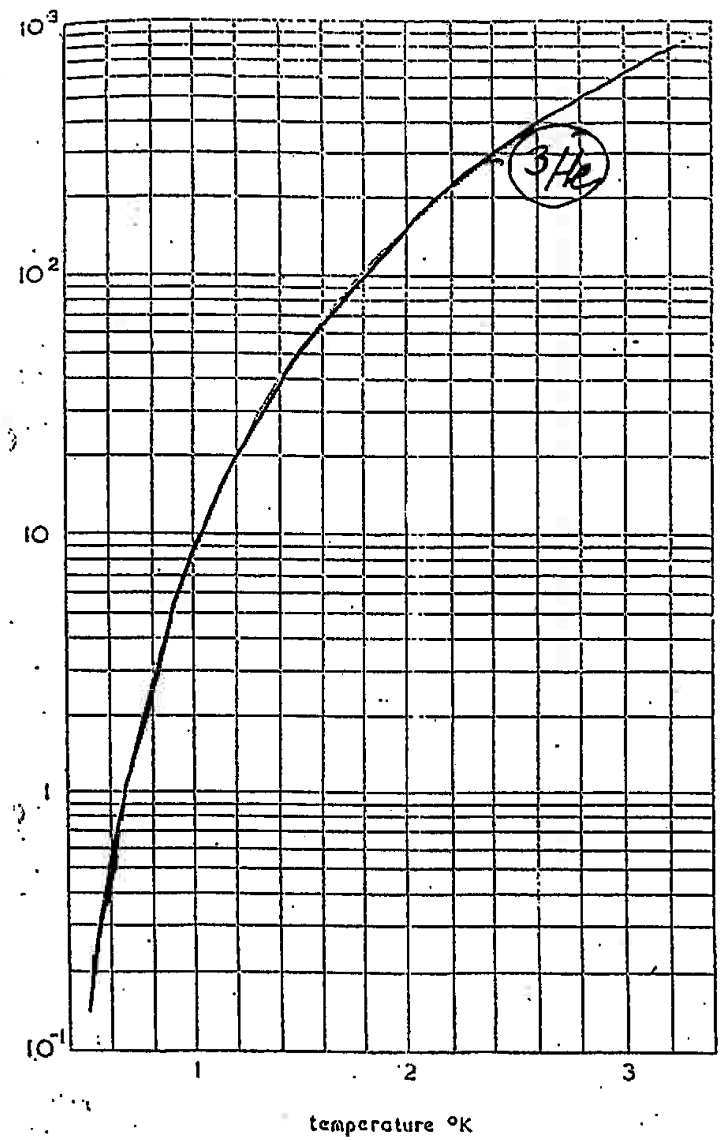


– continuous operation
– T_{min} ~ 1 K
– roughly 40% of evaporating liquid is needed to cool down incoming fluid, the rest is available for cooling => ***dQ/dt* ~ 0.6 *dn/dt L***
– flow impedance (~ 1 m of 50 µm capillary)

1 Torr = 1,33 mbar = 133 Pa

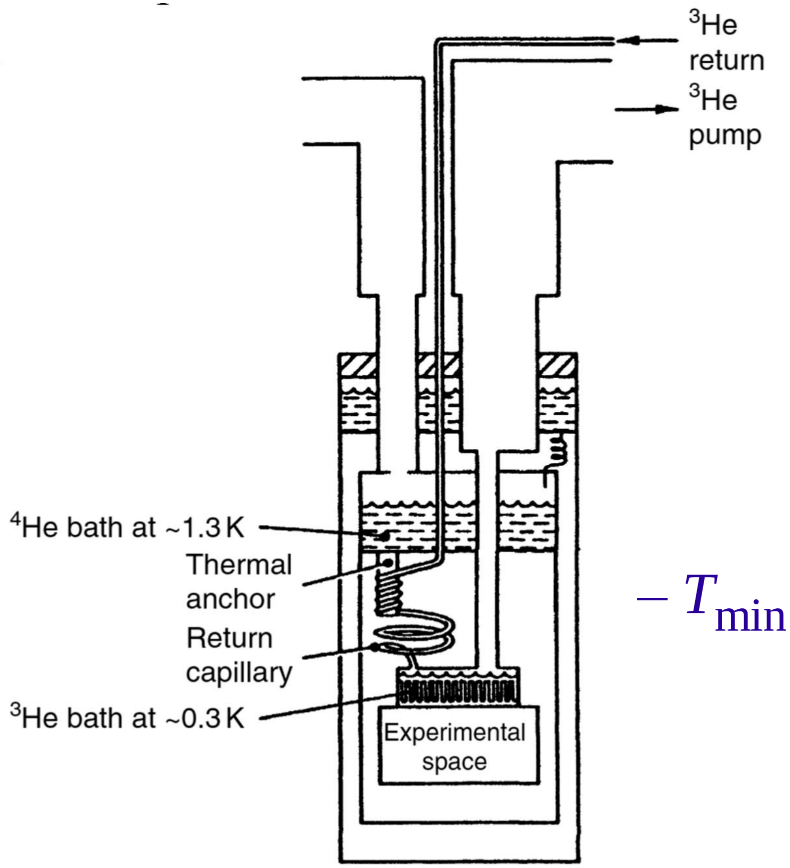


Vapour pressure of liquid Helium[4]



Vapour pressure of liquid Helium[3]

^{3}He evaporators



– T_{min} ~ 0.3 K

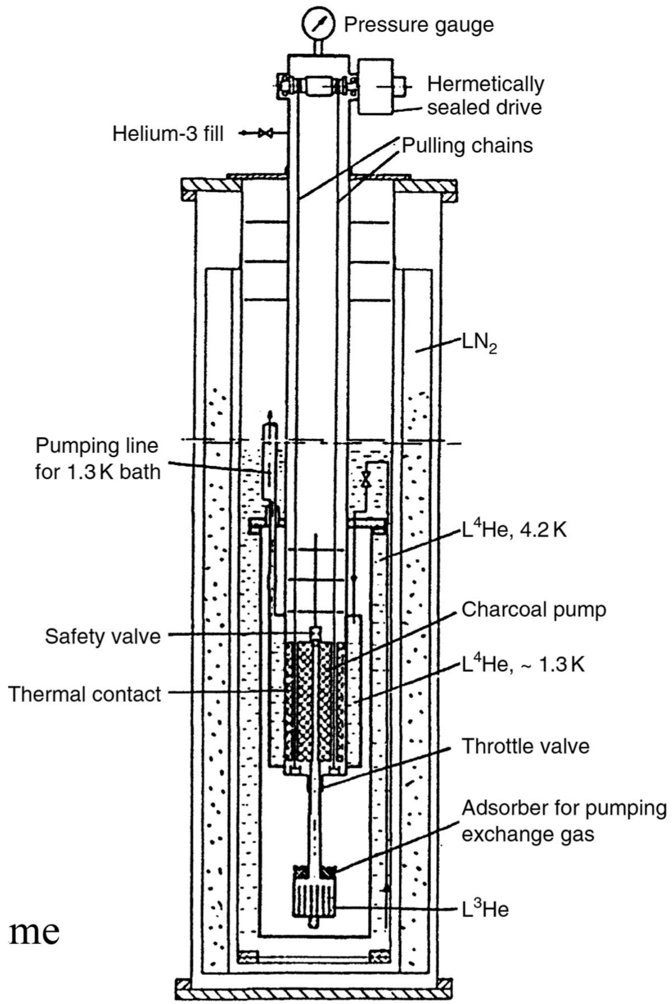


Fig. 6.2. Hermetically sealed, charcoal pumped ^{3}He refrigerator. The charcoal pump can be raised or lowered by means of a chain drive operated via a hermetically closed rotating seal [6.11]

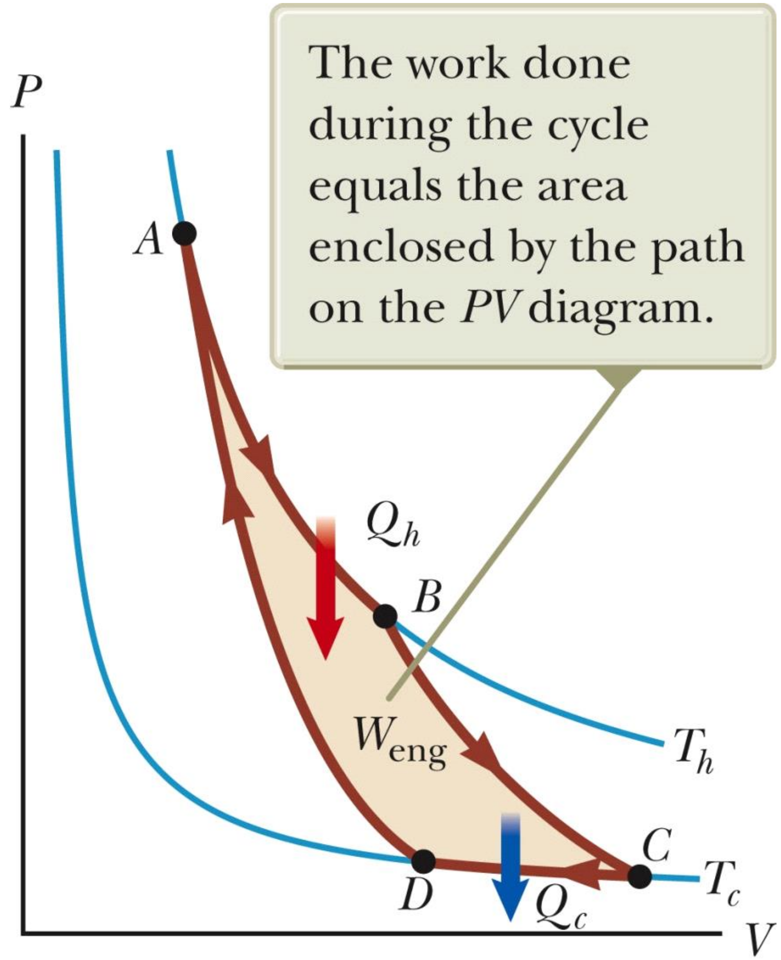
"Single-shot" version (0.25 K)
[Wiedemann & Smolic, 1968]

– closed cycle, hermetic pumps, minimum volume
– combine with ^{4}He pot, since T_{c3} = 3.3 K
– impedance ~ 10^{12} cm^{-3}
– circulating gas must be cleaned
(LN2 charcoal trap)
– nearly as complicated in construction as a simple dilution fridge with base-T ~ 50 mK

me

Heat engines

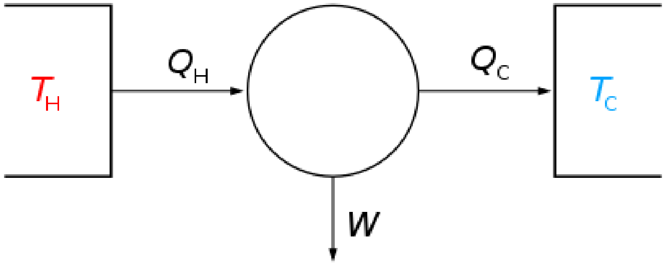
Sadi Carnot 1796-1832



Efficiency of Carnot cycle

$$\varepsilon = \frac{Q_{in} - |Q_{out}|}{Q_{in}} = 1 - \frac{|Q_{out}|}{Q_{in}}$$

$$\varepsilon = 1 - \frac{T_c}{T_h}$$



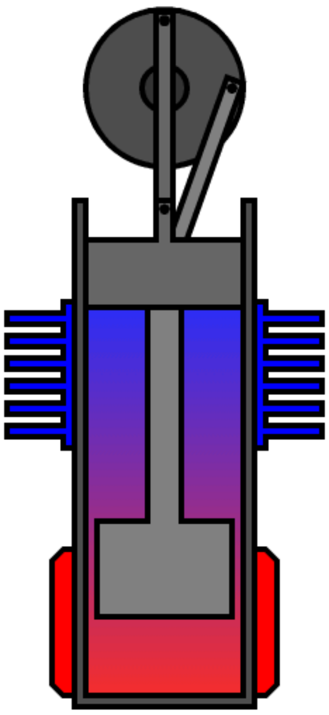
A→B Isothermal at T_h
B→C Adiabatic
C→D Isothermal at T_c
D→A Adiabatic

The working gas is compressed in the colder portion of the engine and expanded in the hotter portion **resulting in a net conversion of heat into work**

$$\frac{pV}{T} = const.$$

$$\frac{pV^{\gamma}}{T} = const. \qquad \gamma = \frac{C_p}{C_V} = \frac{5}{3} \text{ ideal monoatomic gas}$$

Cryocoolers



Stirling cycle:

Isochoric compression

Isothermal expansion

Regenerator:

1. heat capacity of the matrix is larger than that of gas
2. thermal contact between gas and the matrix is perfect
3. gas behaves as ideal gas
4. the flow resistance within the matrix is negligible
5. the axial thermal conductivity is negligible

Coefficient of performance: $\xi = T_L/(T_H - T_L)$

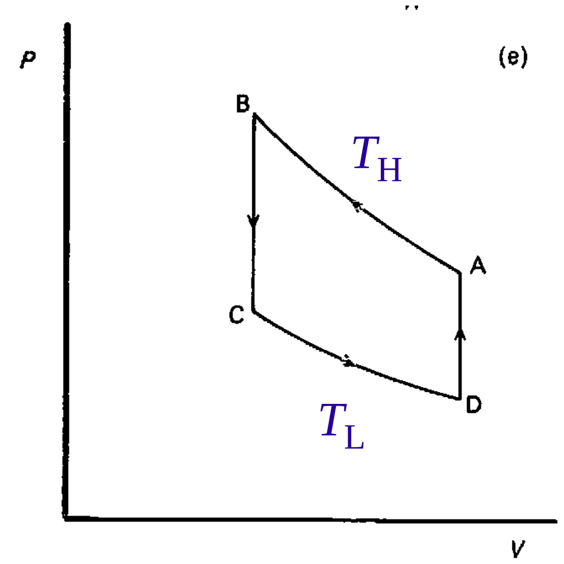
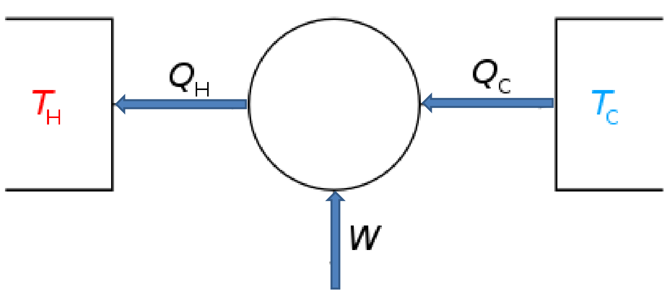
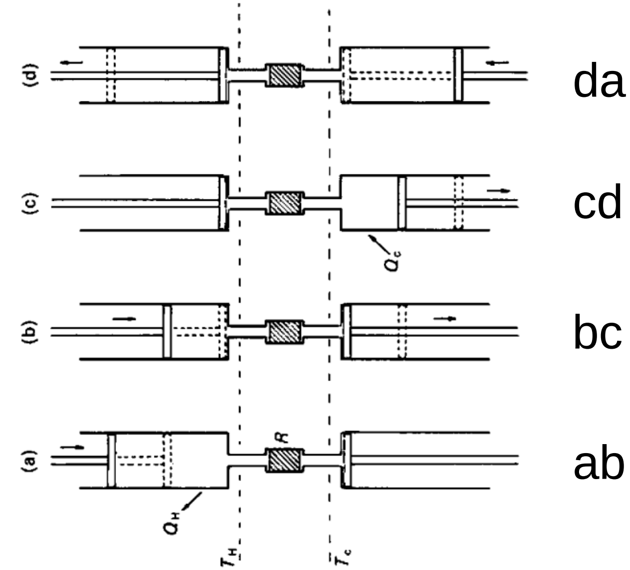


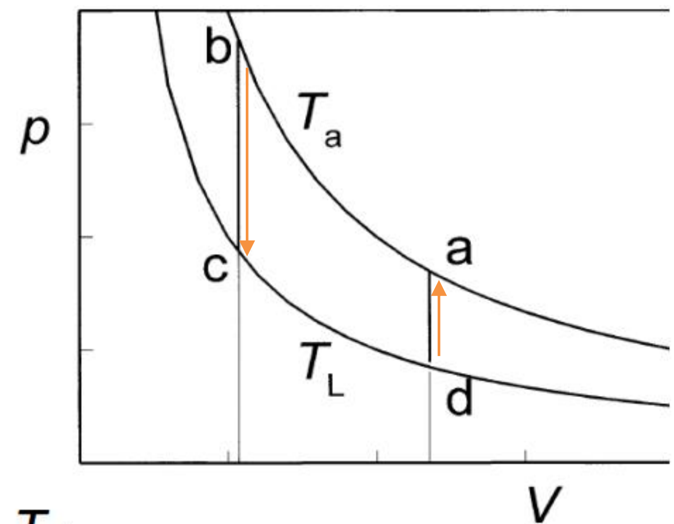
Figure 1.4 The Stirling refrigeration cycle: (*a*)–(*d*) represent different positions of the pistons and (*e*) is the corresponding pressure-volume indicator diagram for the cycle.

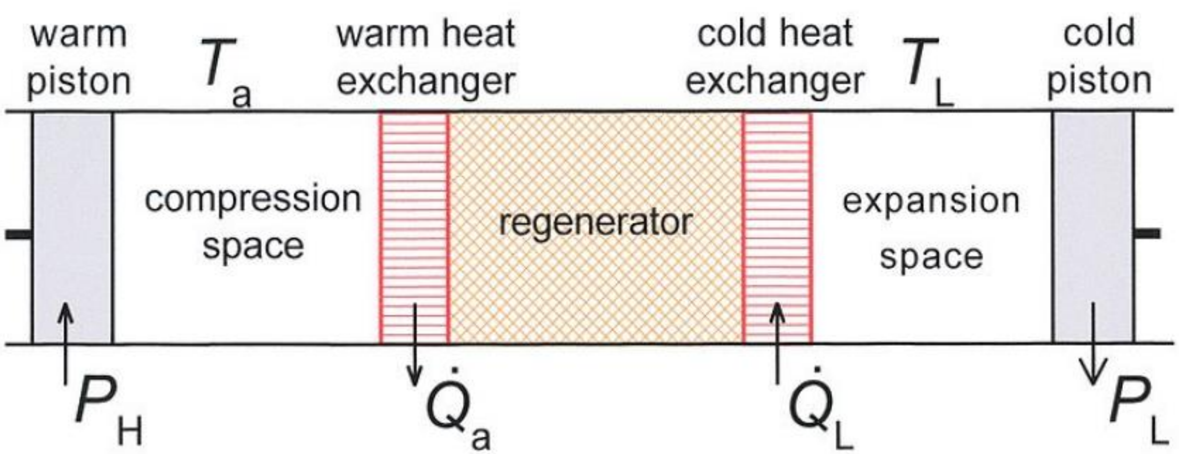


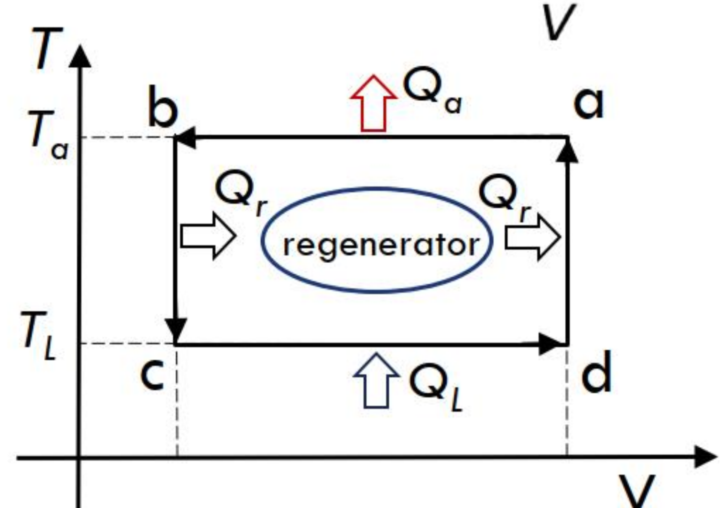
Stirling cooler



1 (cd) expand low T: p decreases
2 (da) move pistons at V=const.: heat from regenerator, p grows, T up
3 (ab) compress high T: p grows
Remove heat to environment
4 (bc) move pistons at V=const.: Heat to regenerator, p decreases, T down

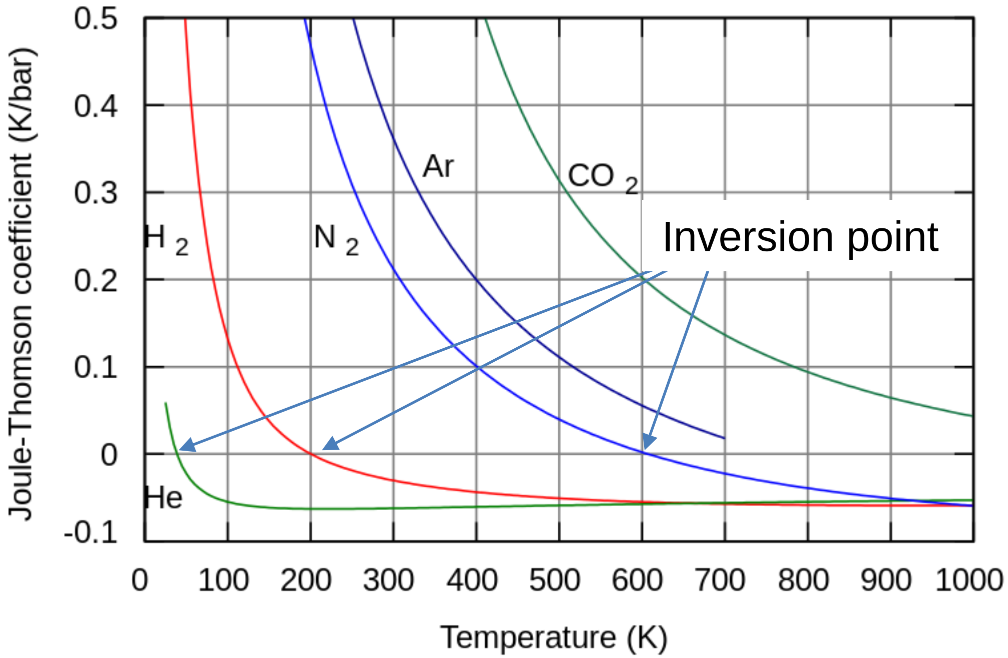




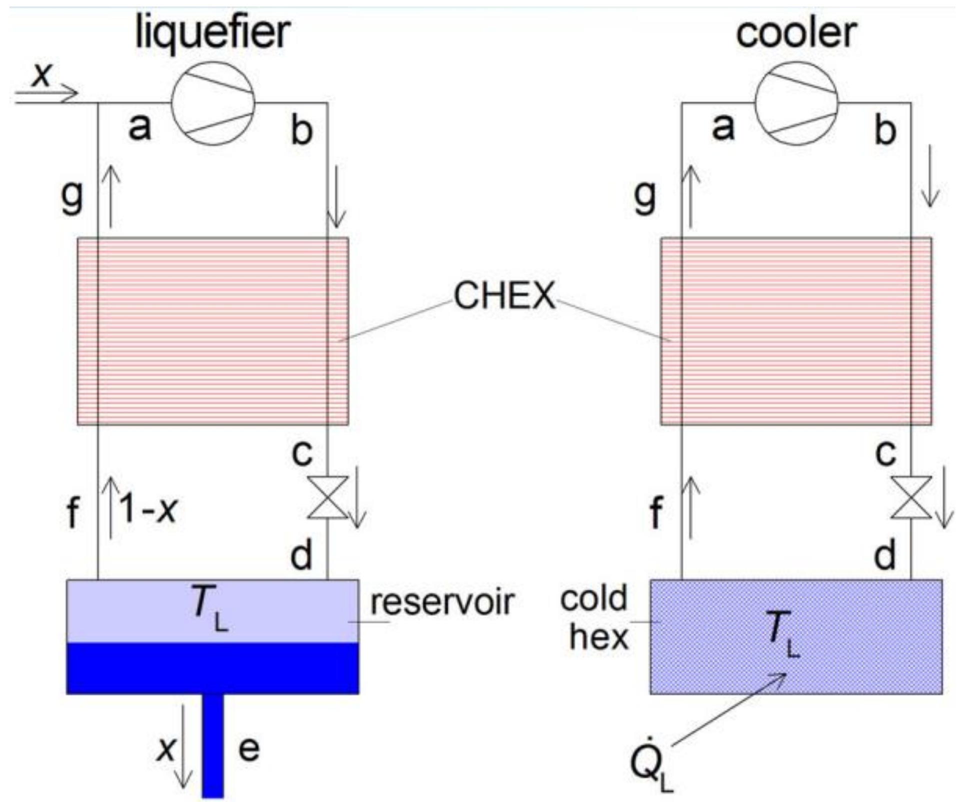


The thermal contact with the surroundings at the temperatures T_a and T_L is supposed to be perfect so that the compression and expansion are isothermal.

JOULE–THOMSON CRYOCOOLERS



JT liquefier (left) and JT cooler (right). In the liquefier, the mass flows in the CHEX at the two sides differ while in the cooler they are equal.



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

Cooling when $\mu_{JT} > 0$

Ideal gas: $\alpha T = 1$

Joule–Thomson cycle provides cooling only **with real gases** where the enthalpy is a function of pressure (Nitrogen, argon, mixed refrigerants).

Pulse tube refrigerator

Components:

– piston (or compressor + valves)

– heat exchangers X_1, X_2, X_3

– regenerator

– tube

– orifice

– reservoir volume

Working substance is helium gas at $p \sim 20$ bar

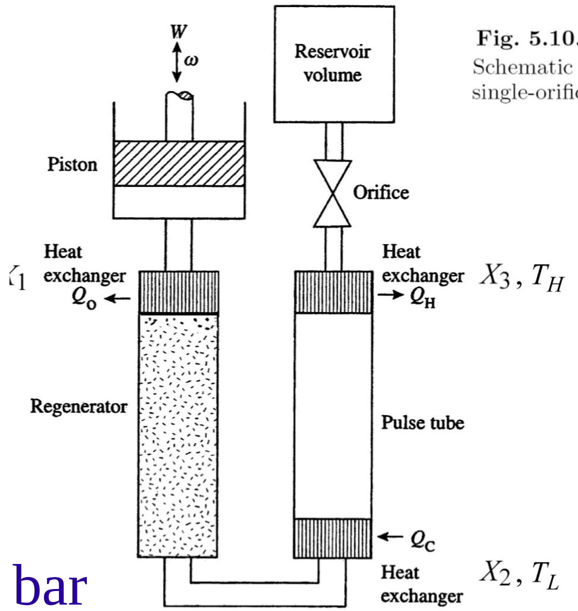
– Every other process is reversible except flow through the orifice

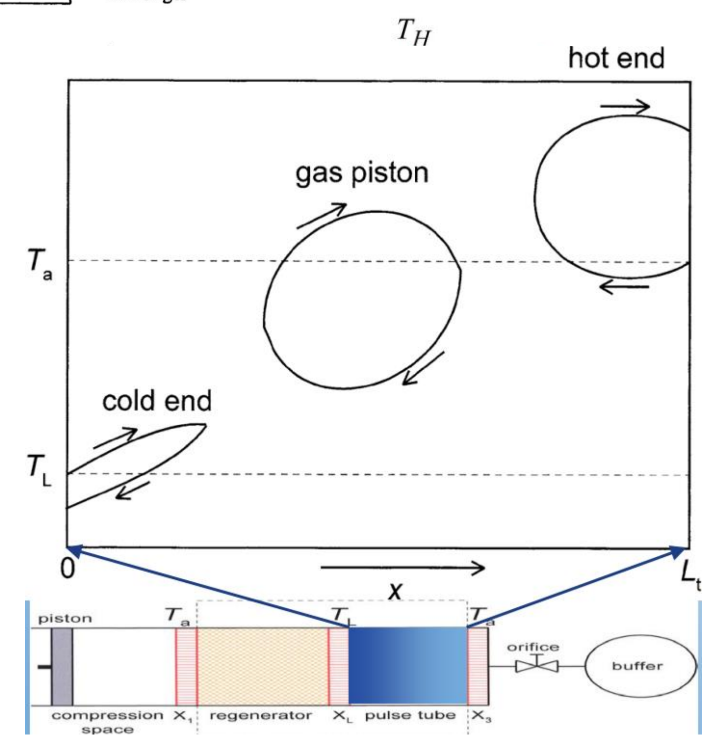
– Pressure in the reservoir is practically constant

– Heat produced by the piston is taken out at X_1

– Heat absorbed at the low temperature junction X_2 is taken out at X_3

Coefficient of performance: $\xi = T_L/T_H$





To produce $T < 4$ K, two pulse tubes must work in series

Ideal extracted power:

$$dQ/dt = 1/2\ RT_0\ dn/dt\ \Delta p/p_0 \cos\theta$$

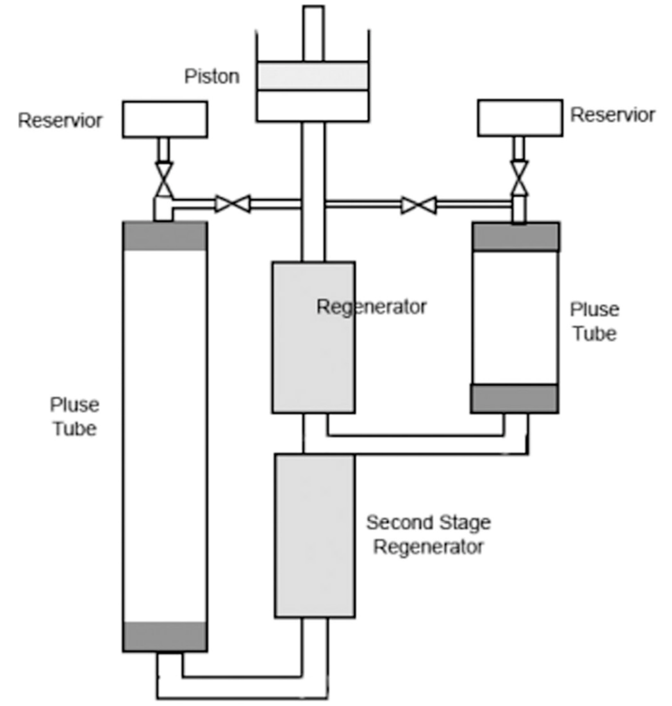
T_0: temperature of isothermal expansion

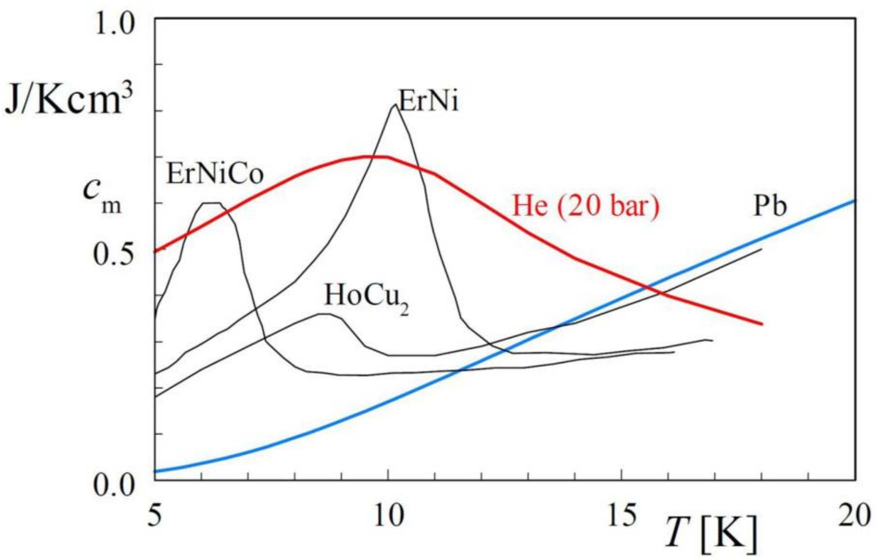
phase θ between the gas flow and pressure can be tuned by ~ 10% flow through a bypass orifice

Regenerator material has to be chosen according to the operation range

Low-*T* stage uses **magnetic heat capacity** of some substances

Regenerator operation degrades with time





Ray Radebaugh, Proc. Institute of Refrigeration (London) 96, 11 (1999-2000)

Real example:

Cooling capacity:	1st	30 W @ 65 K
	2nd	0.5 W @ 4.2 K
Lowest temperature:	2.5 K	
Weight:	25 kg	
Cool down time:	100 min