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# Superconductivity and Low Temperature Physics II



**Lecture Notes  
Summer Semester 2023**

**R. Gross  
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# Chapter 3

## Cryogenic Techniques Generation and Measurement of Low Temperatures



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# Superconductivity and Low Temperature Physics II



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**Lecture No. 11**

**R. Gross**

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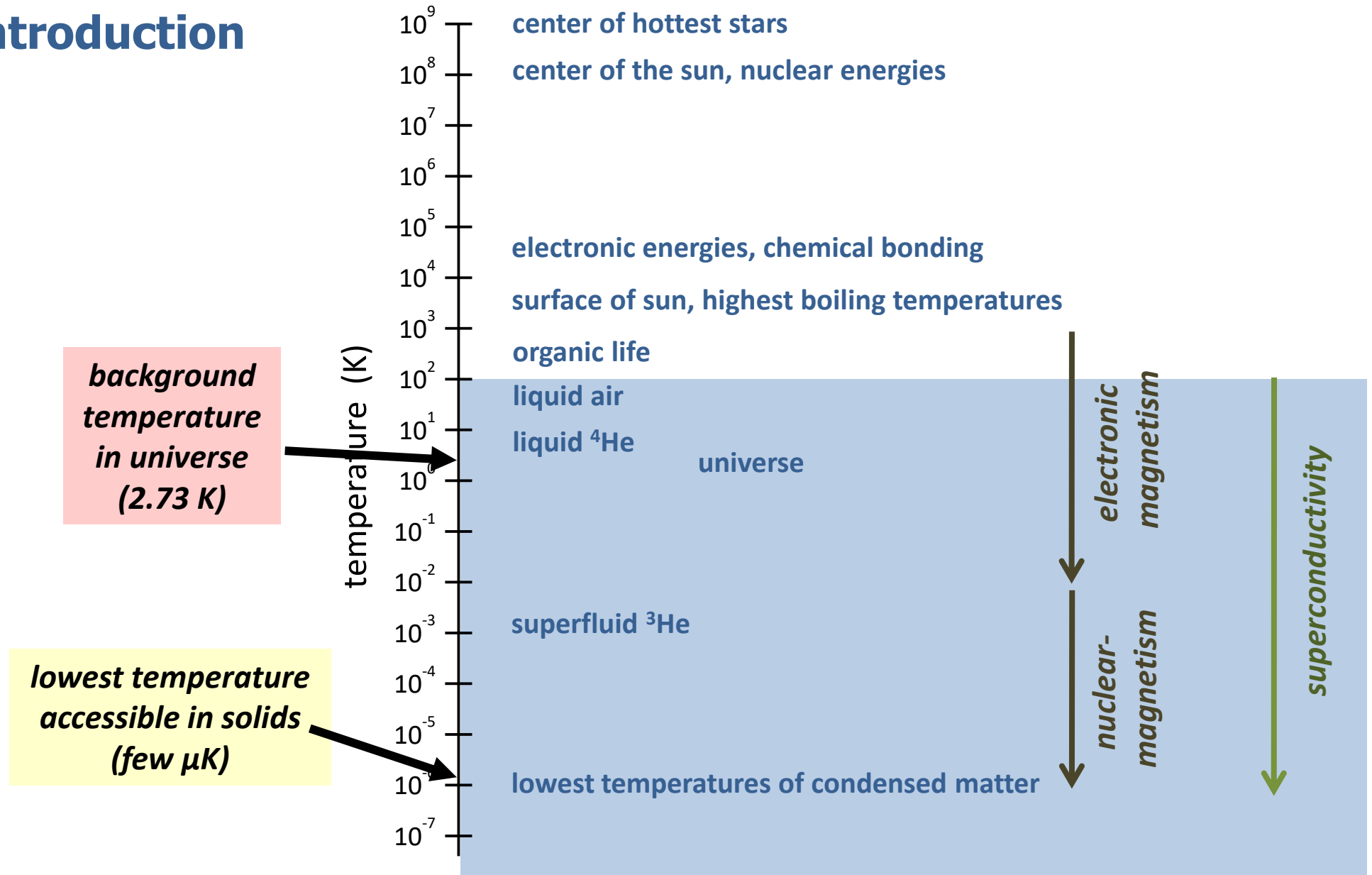
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## *Literature:*

- 1. Tieftemperaturphysik**  
**Enss, Hunklinger**  
Springer (2000)
- 2. Matter and Methods at Low Temperatures**  
**F. Pobell**  
Springer, 2nd edition (1996)
- 3. Experimental Low-Temperature Physics**  
**Anthony Kent**  
American Institute of Physics (1993)
- 4. Cryogenic Systems**  
**Randall F. Barron**  
Oxford University Press, Oxford (1985)

# III.1 Generation of Low Temperatures

## III.1.1 Introduction



# III.1.1 Introduction

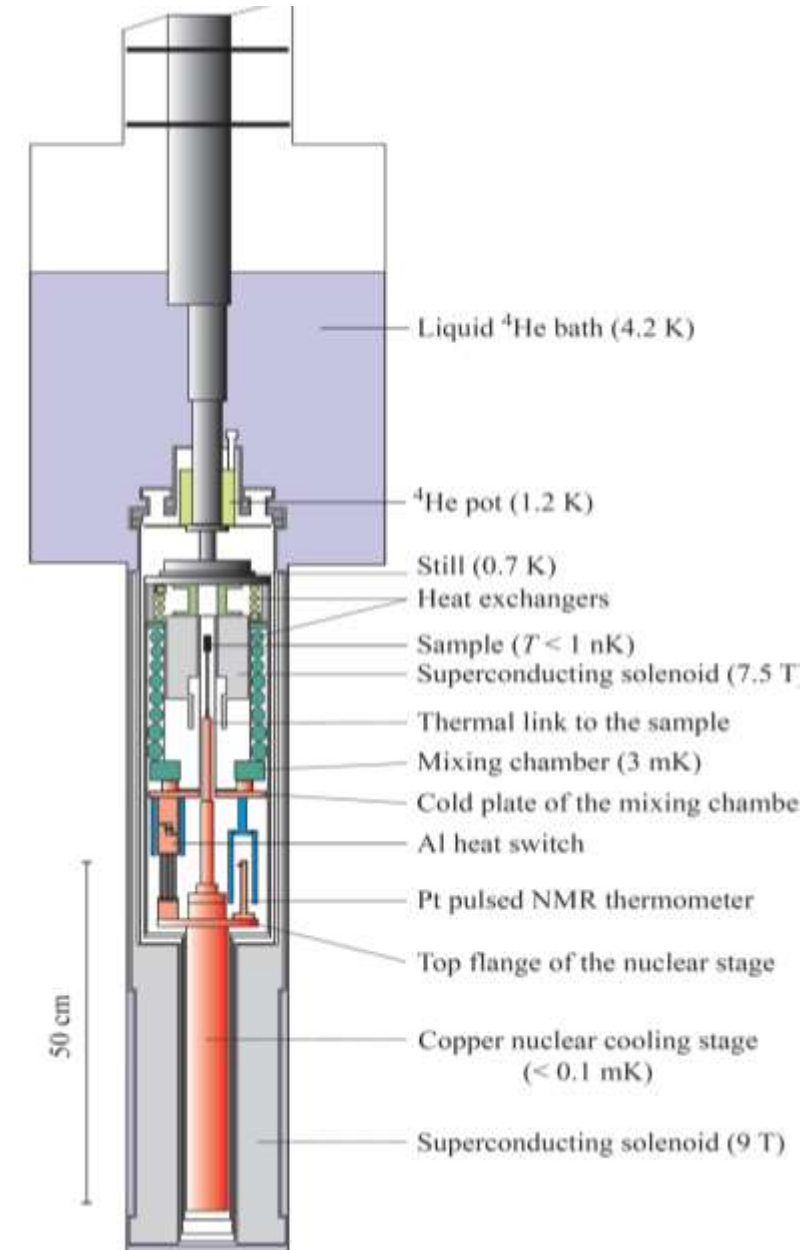
- lowest temperatures (in solid-state systems)

*low temperature record  
for nuclear spin system:*

- experimental setup according to Tauno Knuuttila (2000)
- lowest temperature: about **100 pK** by demagnetization of Rhodium nuclei („temperature of nuclear spins“)

*PhD Thesis, Helsinki University of Technology  
(Espoo, Finland)*

- **problem:** spin temperature cannot be transferred to lattice of solid



# III.1.1 Introduction

- generation of low temperatures by using cryo-liquids

**19<sup>th</sup> century:** liquefaction of various gases by pressure except for “permanent gases” (O<sub>2</sub>, H<sub>2</sub>, He)

**1877:** liquefaction of O<sub>2</sub> by thermal expansion

(L. Cailletet, C.R. Acad. Sci. Paris 85, 1213 (1877); R. Pictet, C.R. Acad. Sci. Paris 85, 1214 (1877))

**1884:** liquefaction of H<sub>2</sub> (precooling with liquid O<sub>2</sub>)

(K. Olszewski, Ann. Phys. u. Chem. 31, 58 (1887))

**1898:** significant amounts of LH<sub>2</sub> for physical experiments

(J. Dewar, Proc. R. Inst. Gt. Br. 15, 815 (1898))

**1908:** liquefaction of last “permanent gas” He by Kamerlingh Onnes

(H. Kammerlingh Onnes, Leiden Commun. 105, Proc. Roy. Acad. Sci. Amsterdam 11, 168 (1908))

**1922:** Kammerlingh Onnes reaches  $T < 1$  K

(H. Kammerlingh Onnes, Leiden Commun. 159, Trans. Faraday Soc. 18 (1922))

**1926:** adiabatic demagnetization of electron spins in paramagnetic salts by Debye and independently (P. Debye, Ann. Phys. 81, 1154 (1926))

**1927:** by Giaque (W.F. Giaque, J. Am. Chem. Soc. 49, 1864 (1927))

**since 1950s:** <sup>3</sup>He available

<sup>3</sup>He cryostat

<sup>3</sup>He-<sup>4</sup>He dilution refrigerator



Sir James Dewar,  
(1842-1923)



Peter J. Debye  
1884 - 1966



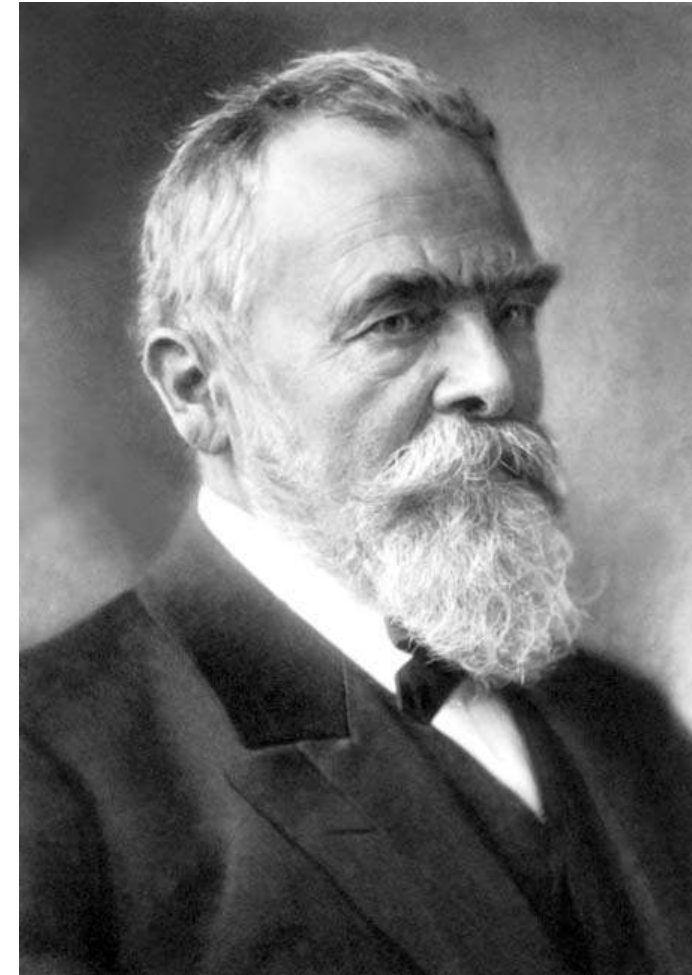
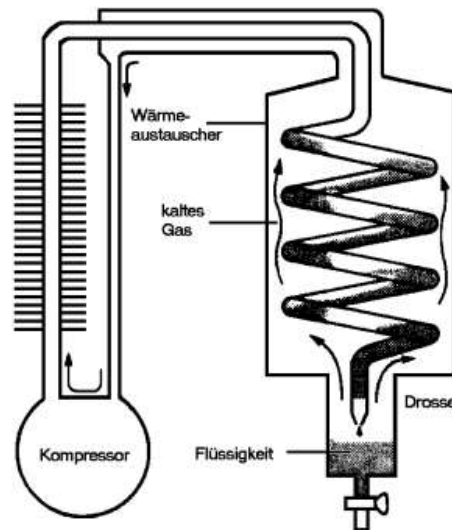
Heike Kamerlingh Onnes  
(1853 – 1926)  
Nobelpreis für Physik: 1913



# III.1.1 Introduction

- 1868** offer of chair at the Polytechnische Schule München (now TUM)
- 1873** development of cooling machine allowing the temperature stabilization in beer brewing
- 21. 6. 1879** foundation of „*Gesellschaft für Linde's Eismaschinen AG*“ together with two beer brewers and three other co-founders
- 1892 – 1910** re-establishment of professorship

- 12.5.1903** patent application: „*Lindesches Gegenstrom-verfahren*“  
liquefaction of oxygen  
( $-182^{\circ}\text{C} = 90\text{ K}$ )

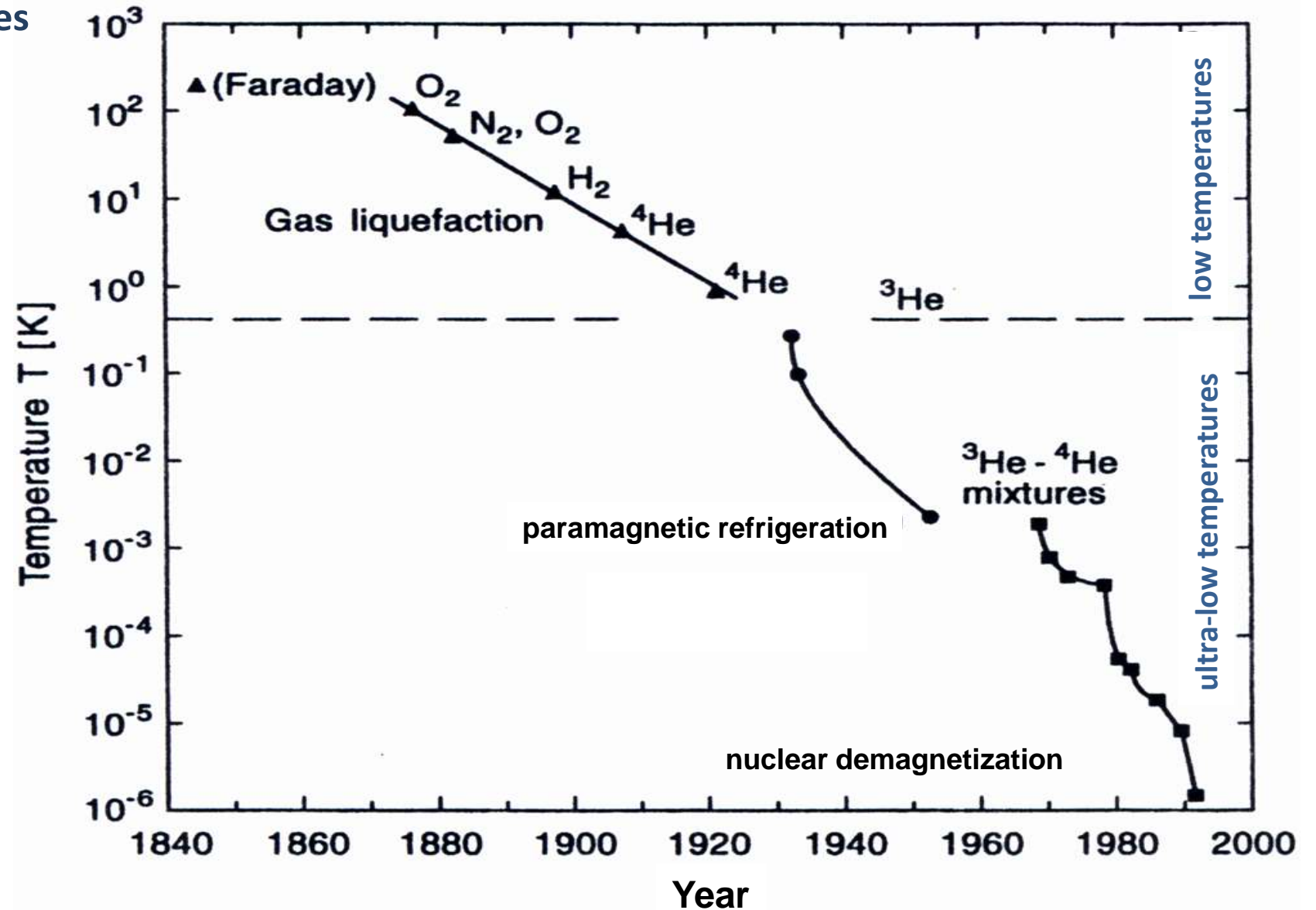


**Carl Paul Gottfried von Linde**

\* 11. Juni 1842 in Berndorf, Oberfranken  
† 16. November 1934 in Munich

# III.1.1 Introduction

- refrigeration techniques



# III.1.1 Introduction

- refrigeration techniques

temperature range	refrigeration technique	available since	typical $T_{\min}$	record $T_{\min}$
Kelvin	universe			2.73 K
	$^4\text{He}$ evaporation	1908	1.3 K	0.7 K
	$^3\text{He}$ evaporation	1950	0.3 K	0.25 K
millikelvin	$^3\text{He}$ - $^4\text{He}$ dilution	1965	10 mK	2 mK
	Pomeranchuk cooling	1965	3 mK	2 mK
	electron spin demagnetization	1934	3 mK	1 mK
microkelvin	nuclear spin demagnetization	1956	50 $\mu\text{K}$	100 pK

# III.1.1 Introduction

- cooling techniques
  - expansion of an ideal gas
    - expansion machine
    - regenerative machine
    - *work against outside world*
  - expansion of a real gas
    - Joule Thomson cooler
    - *work against internal interactions*
  - evaporation of a real gas:
    - *work against internal interactions*
  - dilution cooling ( $^3\text{He}/^4\text{He}$ )
    - *work against ordering in momentum space*
  - adiabatic demagnetization (electronic/nuclear moments)
    - *work against magnetic ordering*

# III.1.1 Introduction

- liquefaction of gases

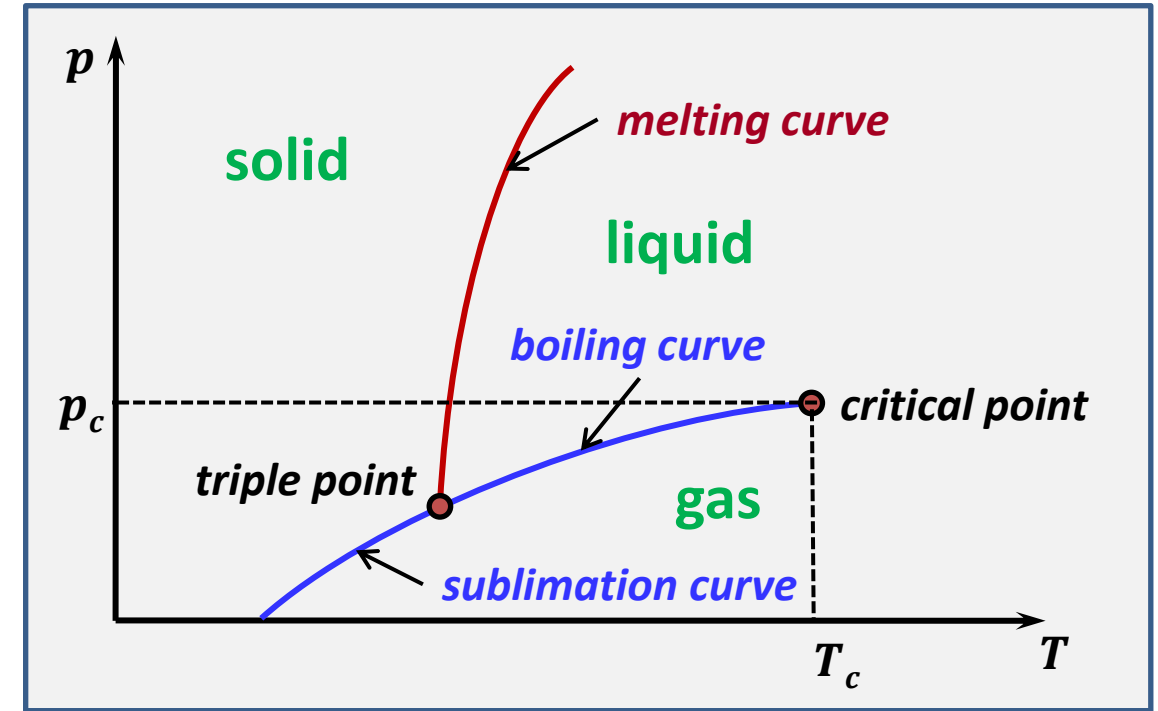
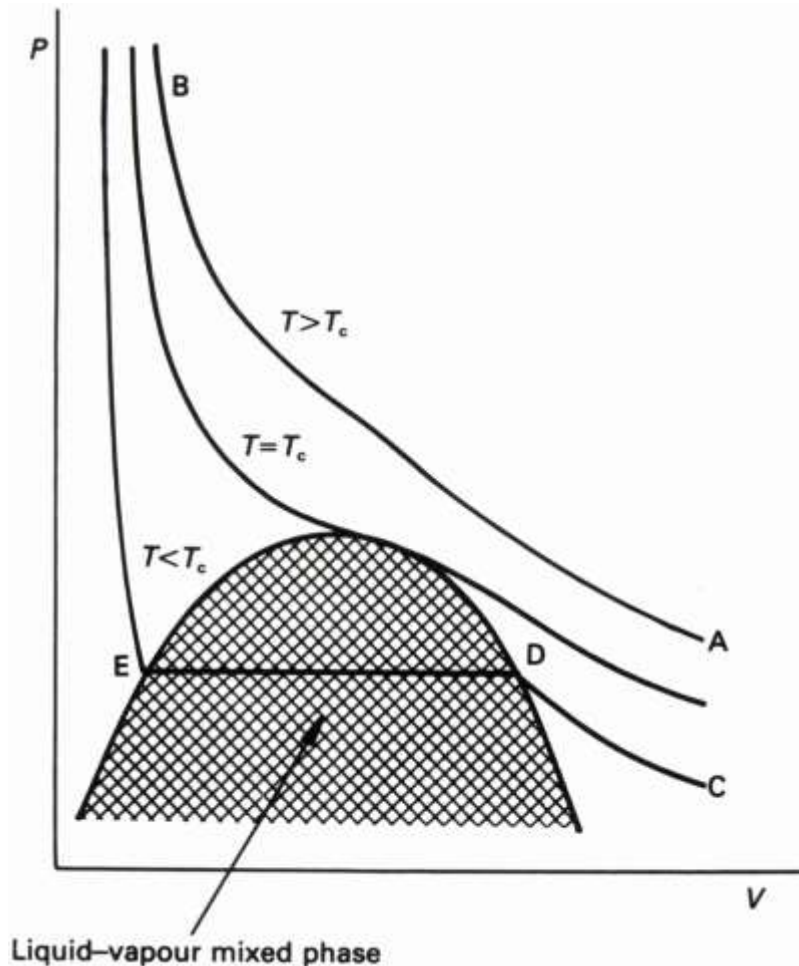
## three useful methods:

1. direct liquefaction by *isothermal compression*
2. letting the gas perform *work against external forces* at the expense of its internal energy
  - ➔ cooling and eventual liquefaction
3. making the gas perform *work against its own internal forces* by Joule-Kelvin or Joule-Thomson expansion
  - ➔ cooling and eventual liquefaction

# III.1.1 Introduction

- direct liquefaction of gases by isothermal compression

→ *starting temperature must be smaller than critical temperature  $T_c$*



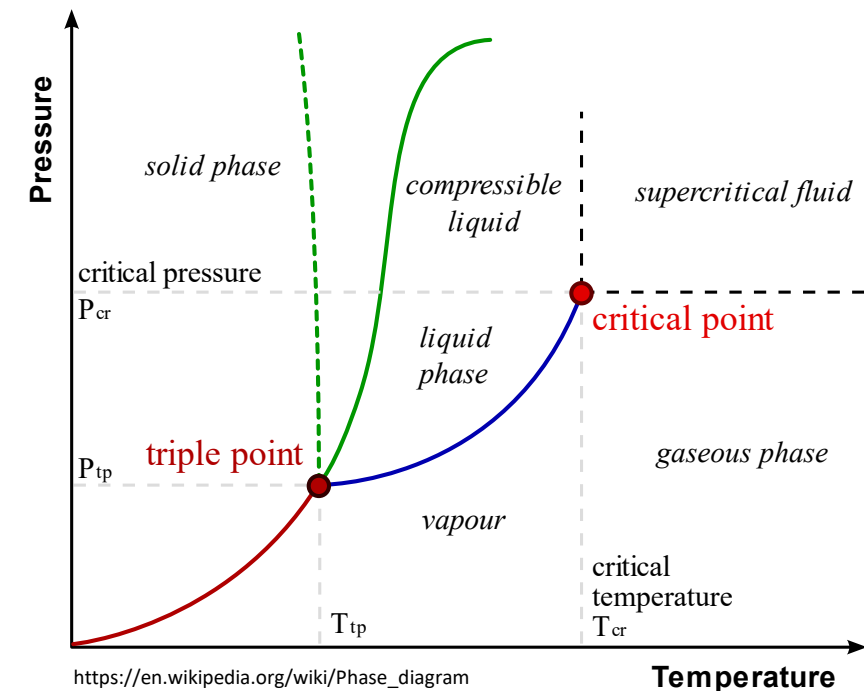
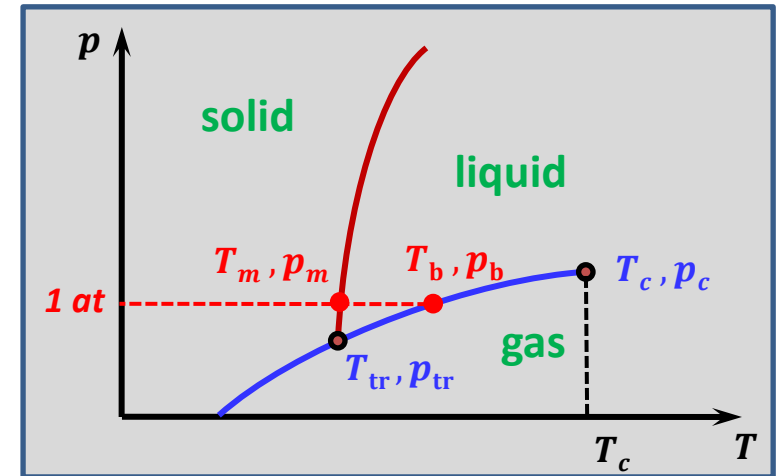
*critical temperatures  $T_c$  in K of selected liquid cryogenes*

ammonia (NH <sub>3</sub> )	406
O <sub>2</sub>	154.5
N <sub>2</sub>	126
H <sub>2</sub>	33.2
<sup>4</sup> He	5.2
<sup>3</sup> He	3.32

# III.1.1 Introduction

- properties of cryogenic liquids

Substance	$T_b$ [K]	$T_m$ [K]	$T_{tr}$ [K]	$P_{tr}$ [bar]	$T_c$ [K]	$P_c$ [bar]
@ 1 bar						
H <sub>2</sub> O	373.15	273.15	273.16	0.06	647.3	220
Xe	165.1	161.3	161.4	0.82	289.8	58.9
Kr	119.9	115.8	114.9	0.73	209.4	54.9
O <sub>2</sub>	90.2	54.4	54.36	0.016	154.3	50.4
Ar	87.3	83.8	83.81	0.67	150.9	48.7
N <sub>2</sub>	77.4	63.3	63.15	0.12	126.0	33.9
Ne	27.1	24.5	24.56	0.43	44.5	27.2
D <sub>2</sub>	23.7	18.7	18.72	0.17	38.3	16.6
H <sub>2</sub>	20.3	14.0	13.80	0.07	33.3	13.0
<sup>4</sup> He	4.21	--	--	--	5.20	2.28
<sup>3</sup> He	3.19	--	--	--	3.32	1.16



[https://en.wikipedia.org/wiki/Phase\\_diagram](https://en.wikipedia.org/wiki/Phase_diagram)



# III.1.1 Introduction

- direct liquefaction of gases by expansion (Joule-Thomson-Effect)  
 → *starting temperature must be smaller than inversion temperature* (definition follows later)

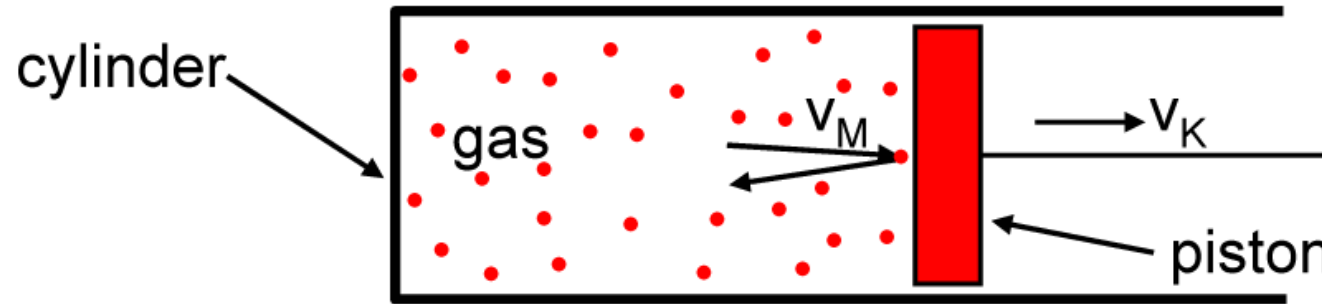
cryogen	boiling point [K]	liquefaction	latent heat [kJ/liter]	inversion temp. [K]
oxygen	90.2	1877: Cailletet and Pictet	240	762
nitrogen	77.3	1883: Wroblewski and Olszewski	160	625
hydrogen	20.4	1898: Dewar	30	203
<sup>4</sup> Helium	4.2	1908: Onnes	2.6	43.2
<sup>3</sup> Helium	3.2		0.5	-

- **liquid oxygen and hydrogen have potential hazards**
- liquid nitrogen and <sup>4</sup>He are the most widely used cryogenes
- **liquid <sup>3</sup>He is very expensive**



# III.1.1 Introduction

- liquefaction of gases by performance of external work



***gas molecules are reflected at the moving piston-surface:***

incoming:	laboratory system: $v_M$
	piston system: $v_M - v_K$
outgoing:	piston system: $-(v_M - v_K)$
	laboratory system: $-(v_M - v_K) + v_K = 2v_K - v_M = v'_M$

i.e.:  $|v'_M| = v_M - 2v_K$  **molecule is slower, i.e. colder**

average momentum transfer per time to piston = force, ***force · distance = work***

**→ external work at the expense of internal energy → cooling**

# III.1.1 Introduction

- liquefaction of gases by performance of external work

- in practice,

1. gas is **isothermally compressed** (involves removing of heat corresponding to work done on gas)
2. and then allowed to **expand adiabatically** (resulting in cooling)

- 1st law of thermodynamics

$$dU = dQ + dW = dQ - pdV = C_V dT$$

$$C_V = \left. \frac{dQ}{dT} \right|_V = \left. \frac{dU}{dT} \right|_V$$

$$C_p = \left. \frac{dQ}{dT} \right|_p = \left. \frac{dH}{dT} \right|_p$$

- adiabatic expansion:  $dQ = 0$

$$\Rightarrow dQ = C_V dT + pdV = 0$$

- for ideal gas:  $pV = RT$

$$\Rightarrow pdV + Vdp = R dT$$

- elimination of  $dT$

$$\Rightarrow 0 = C_V \left( \frac{pdV + Vdp}{R} \right) + pdV \stackrel{C_p - C_V = R}{=} C_V (pdV + Vdp) + pdV (C_p - C_V)$$

$$\Rightarrow 0 = C_V Vdp + C_p pdV = C_V \frac{dp}{p} + C_p \frac{dV}{V} \stackrel{\frac{C_p}{C_V} = \kappa}{=} \kappa \frac{dV}{V} + \frac{dp}{p}$$

- integration yields  $\kappa \ln V + \ln p = \text{const.}$

$$\Rightarrow pV^\kappa = \text{const.}$$

$\kappa$  = isentropic coefficient

# III.1.1 Introduction

- **Carnot process:**
  - counterclockwise: **heat pump** (conversion of mechanical work into heat)
  - clockwise: **heat engine** (conversion of heat into mechanical work)

- **$pV$  diagram:**

expansion cooling: adiabats

$$(pV^\kappa = \text{const}, \quad dQ = 0) \quad \kappa = \frac{c_p}{c_v} > 1)$$

heat exchange: isotherms

$$(pV = \text{const}, \quad dT = 0)$$

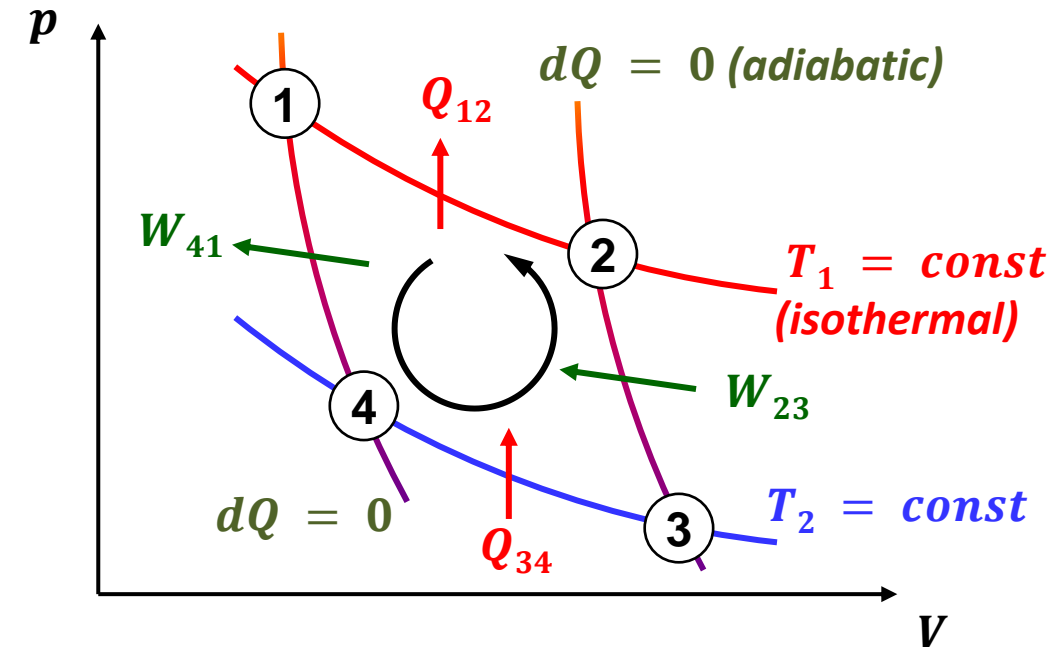
work per cycle:  $W = \oint p dV = \text{area}$

- **efficiency:**  
**heat engine**

$$\frac{W}{\Delta Q} \equiv \eta = \frac{\Delta T}{T_{\text{warm}}} \quad (\text{Carnot})$$

**thermodynamic definition of temperature**

- **Carnot process:** *technologically difficult* to realize  
 → better: **gas circulation, compressor and expansion machine are spatially separated**



# III.1.2 Expansion Machine

- medium: He gas

*Brayton method (reverse Brayton cycle)*

- e.g. **liquefaction of air**:
  - condensation on cold head
  - distillation in separation columns
    - N<sub>2</sub> (77.4 K) cooling
    - Ar (87.3 K) inert gas
    - O<sub>2</sub> (90.2 K) welding

- **temperature reduction:**

$$T_2 = T_1 \left( \frac{p_2}{p_1} \right)^{(\kappa-1)/\kappa}$$

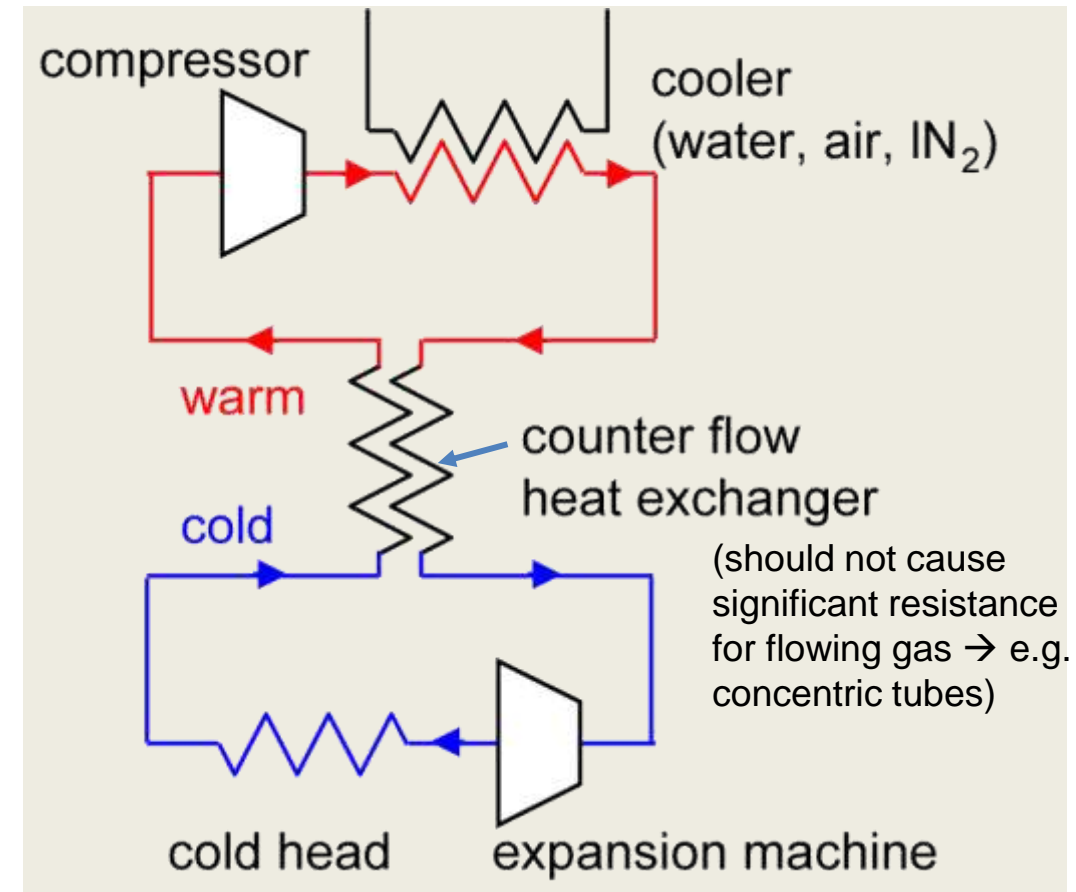
$$\kappa = C_p/C_V (= 5/3 \text{ for He})$$

$$\text{ideal, single-atomic gas: } \kappa = 1 + \left( \frac{2}{f} \right)$$

- **efficiency:**

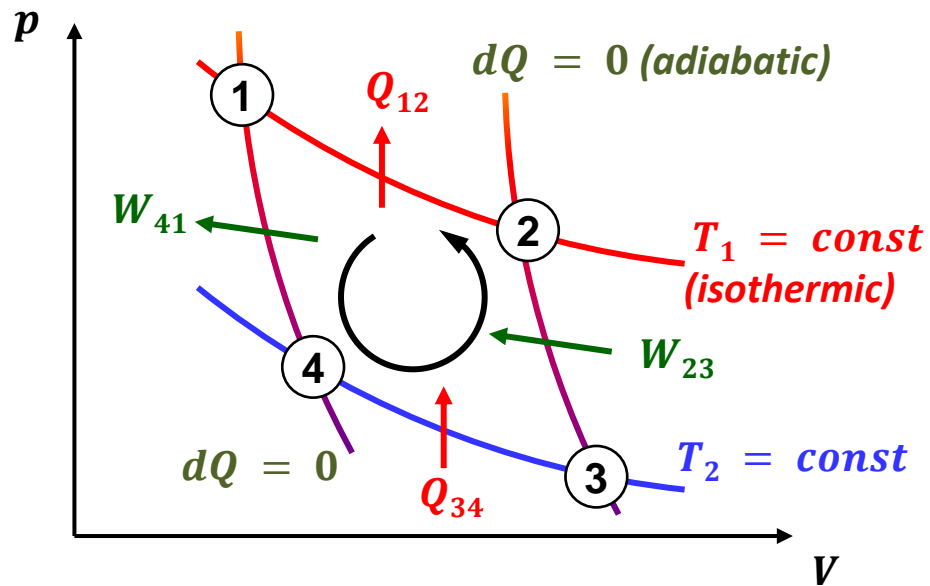
$$\eta = 1 - \frac{T_2}{T_1} = 1 - \left( \frac{p_2}{p_1} \right)^{(\kappa-1)/\kappa}$$

➔ expansion from  $p_1 = 100 \text{ bar}$  to  $p_2 = 1 \text{ bar}$  results in  **$T_2 = 50 \text{ K}$**  @  $T_1 = 300 \text{ K}$   
 $T_2 = 8 \text{ K}$  can be reached in a two-stage cycle



# III.1.2 Expansion Machine

- heat pumps: heating and refrigerating machines



- **heat pump:** *heat is generated by mechanical work*

- **efficiency:**

$$h = \frac{\text{generated heat @ } T = T_1}{\text{performed work}} = \frac{\Delta Q_1}{\Delta W}$$

- **refrigerating machine:** *removing heat (generating cold) by mechanical work*

- **efficiency:**

$$k = \frac{\text{removed heat @ } T = T_2}{\text{performed work}} = \frac{\Delta Q_2}{\Delta W}$$

- **ideal efficiency for reversible Carnot process:**

$$h_c = \frac{1}{\eta_c} = \frac{T_1}{T_1 - T_2} \geq 1$$

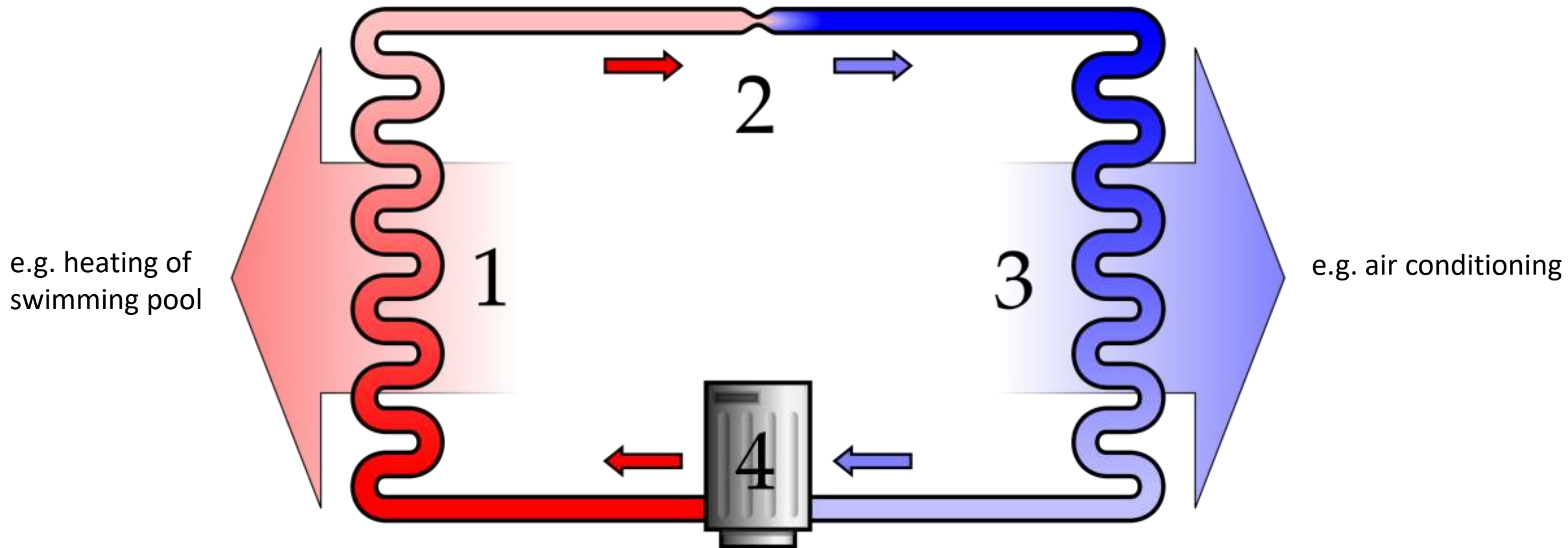
(increases with decreasing temperature difference  $T_1 - T_2$ )

$$k_c = \frac{T_2}{T_1 - T_2} \geq 0$$

(decreases with increasing temperature difference  $T_1 - T_2$ )

# III.1.2 Expansion Machine

- application examples: heating and refrigerating machines



Wikimedia Commons

schematic diagram of a heat pump's vapor-compression refrigeration cycle:

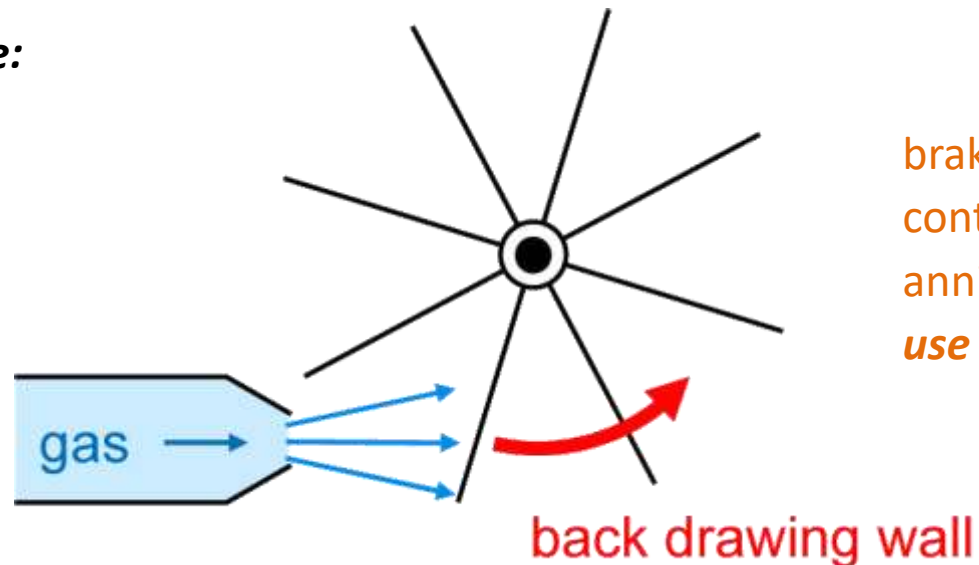
**1) condenser, 2) expansion valve, 3) evaporator, 4) compressor**

# III.1.2 Expansion Machine

- realizations of expansion machines:

- piston-cylinder machine similar to automobile engine  
crankshaft, camshaft, valve
- cooling turbine  $\Rightarrow$  *commercially relevant*  
higher efficiency for larger throughput

*principle:*

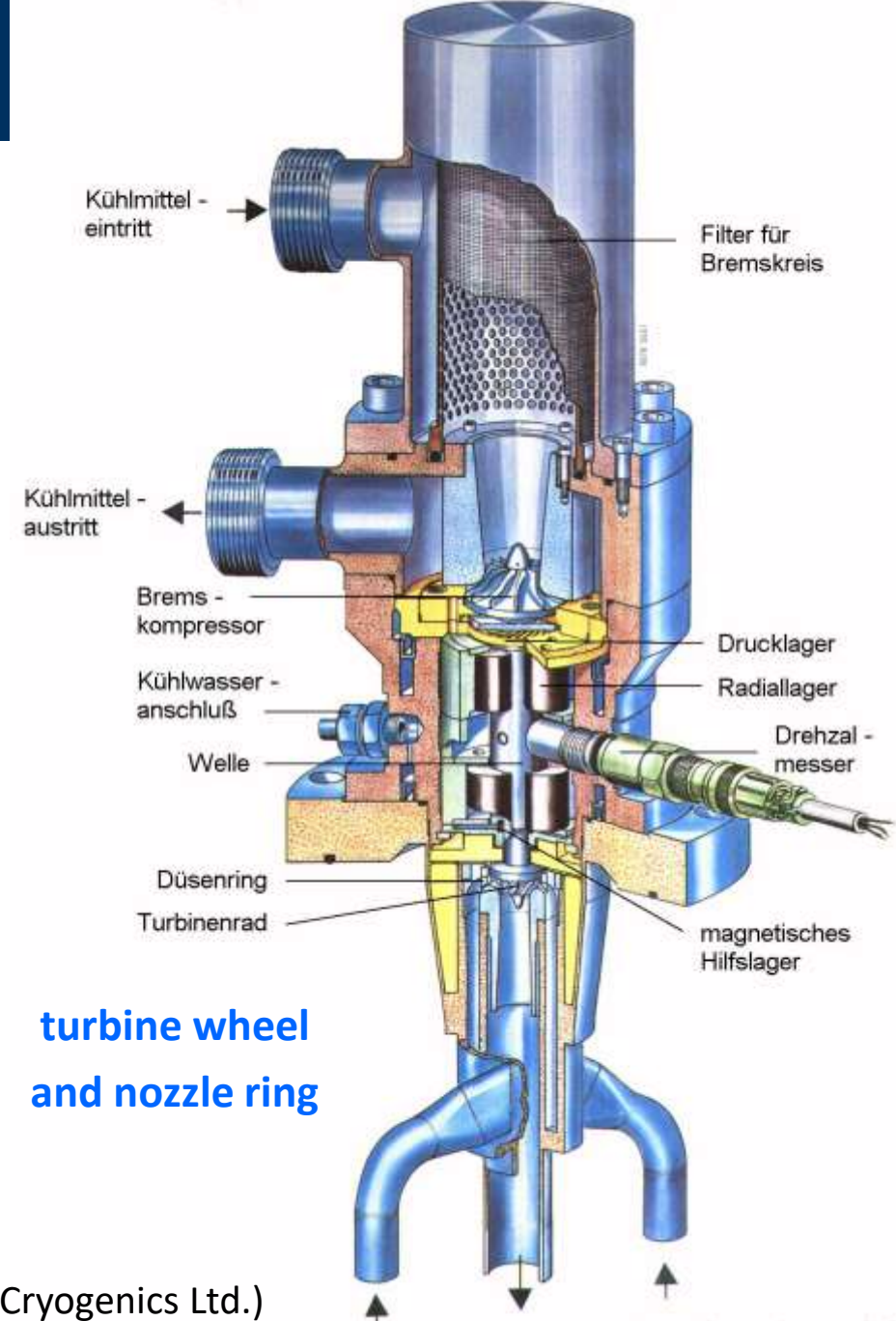
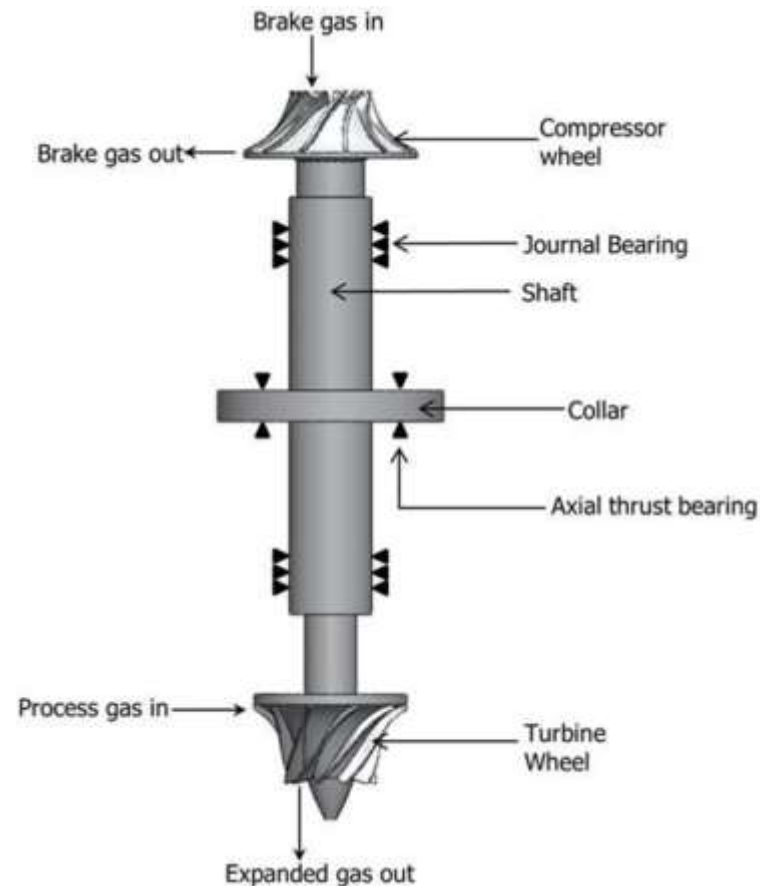


brake on turbine axis  
controls rotational speed,  
annihilates performed work,  
*use of gas bearings*



# III.1.2 Expansion Machine

- turbine cooler (Sulzer machine)



turbine wheel  
and nozzle ring

(Source: Linde Cryogenics Ltd.)




# III.1.2 Expansion Machine

- conclusions
  - expansion machines are technologically simple
  - multi-stage arrangements for lower temperatures
    - ⇒ *almost down to 4.2 K*
  - but:
    - ⇒ *efficiency* only acceptable for cooling turbines
  - no direct liquefaction of gas (mechanical problems)
    - ⇒ *liquefaction by Joule-Thomson stage*
  - for small-scale facilities:
    - ⇒ *regenerative machines* better suited

## ***Contents:***

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### **III.2 Thermometry**

- III.2.1 Introduction
- III.2.2 Primary Thermometers
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# III.1.3 Regenerative Machines

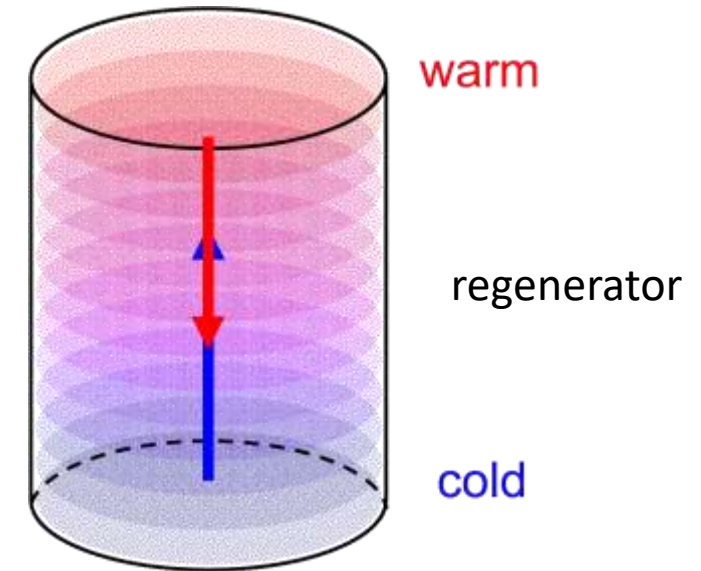
- **regenerator** replaces **heat exchanger**

⇒ column with staple of fine metal meshes (Cu, Pb)

→ *low flow resistance*

→ *high heat capacity*

→ *low longitudinal heat conductivity*



- alternating gas flow:

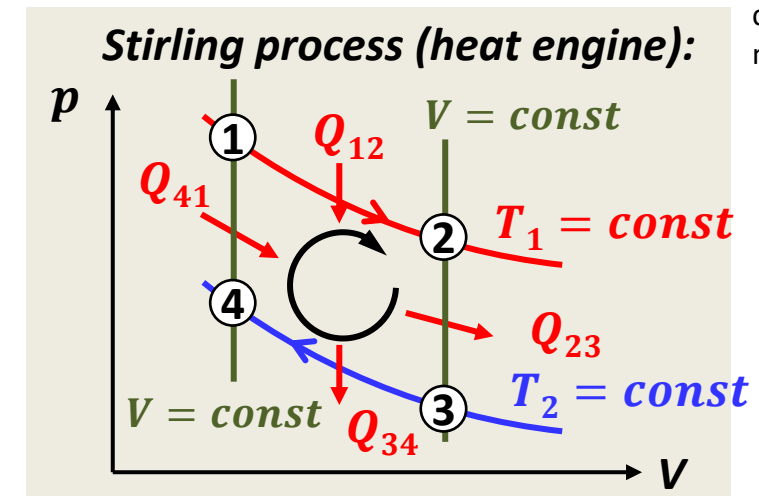
**cold** gas upward

⇒ **cooling of meshes**

**warm** gas downward

⇒ **cooling of gas**

- used in **Stirling process**

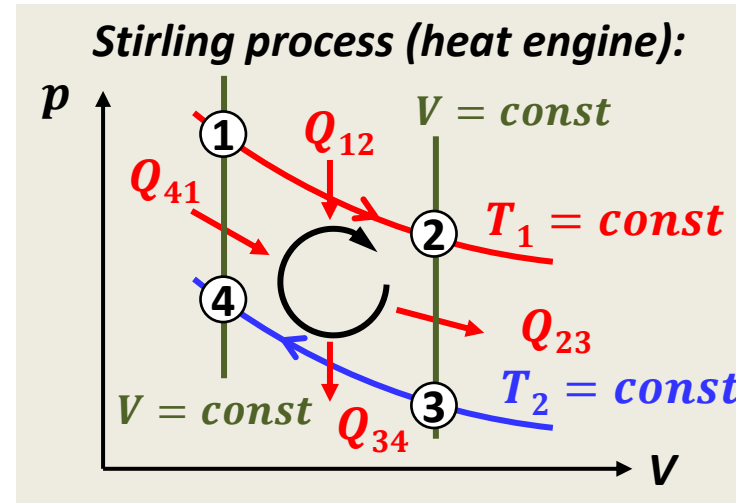


conversion of heat into mechanical work

**cold gained in step 2 → 3 has to be stored and provided in step 4 → 1**

# III.1.3 Regenerative Machines

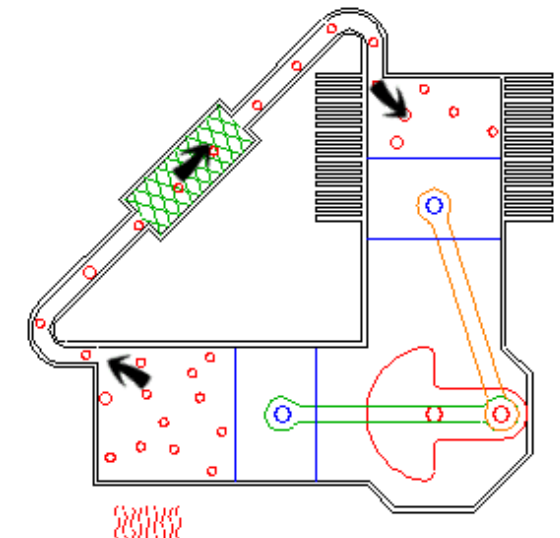
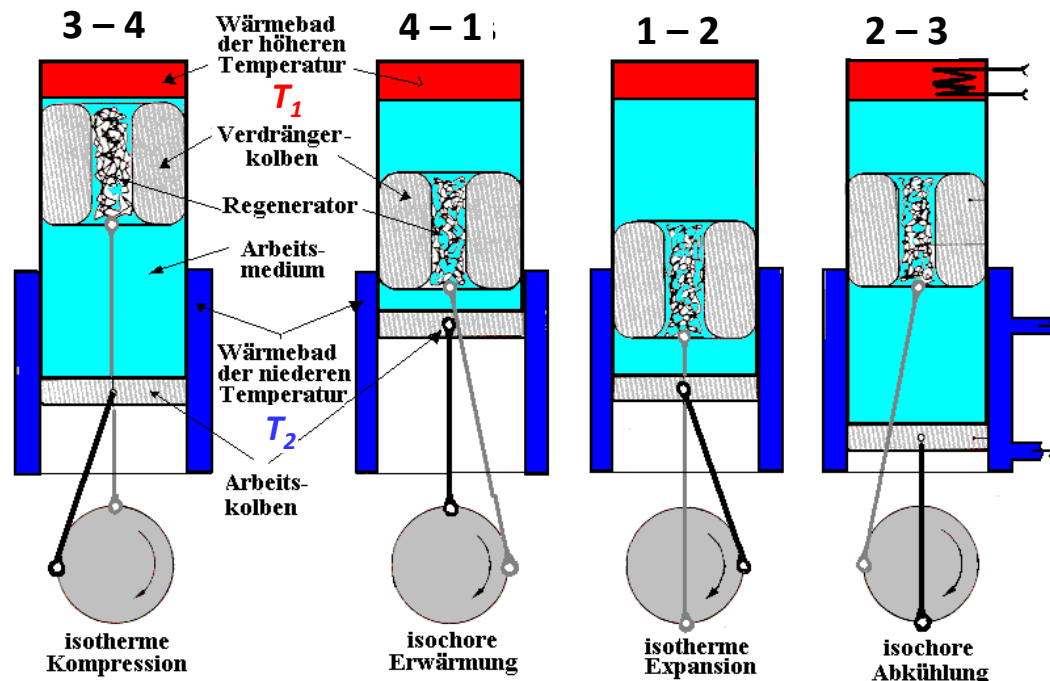
- Stirling machine  
(clockwise, heat engine)



*periodic expansion and compression of gas along two isotherms and two isochors*

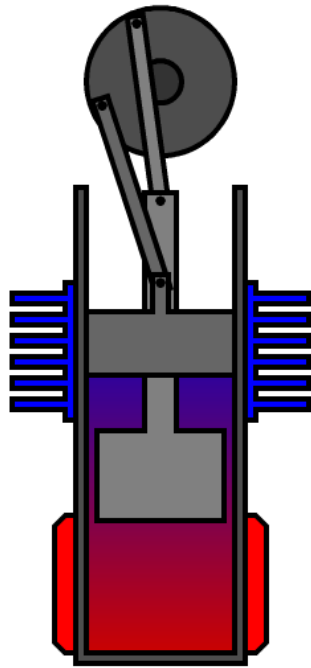
- $1 \rightarrow 2$ : isothermal expansion,  $Q_{12}$  is added
- $2 \rightarrow 3$ : isochoric cooling,  $Q_{23}$  is removed
- $3 \rightarrow 4$ : isothermal compression,  $Q_{34}$  is removed
- $4 \rightarrow 1$ : isochoric warming,  $Q_{41}$  is added
- for isochoric steps there is no mechanical work  

$$Q_{23} = -Q_{41} = C_V \Delta T$$
- goal: intermediate storage of  $Q_{23}$  in regenerator to be able to add it again in step  $4 \rightarrow 1$   
**→ use of two pistons with phase shift**

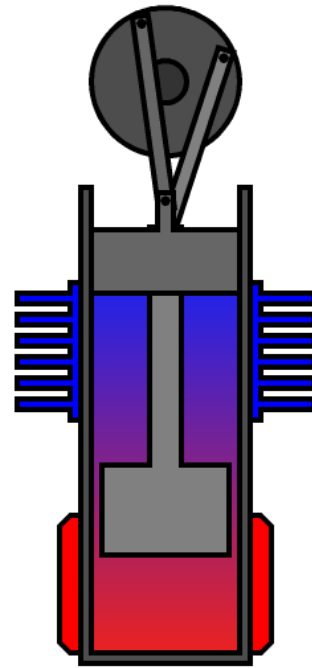


# III.1.3 Regenerative Machines

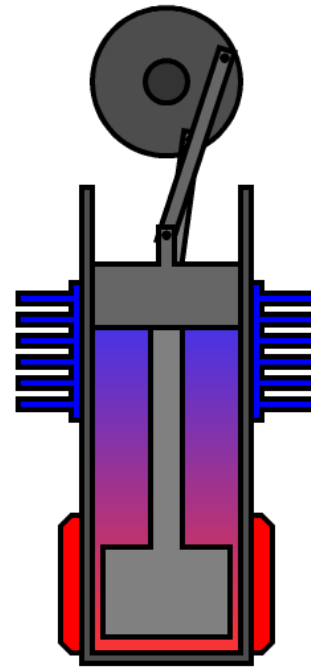
- (beta) Stirling machine



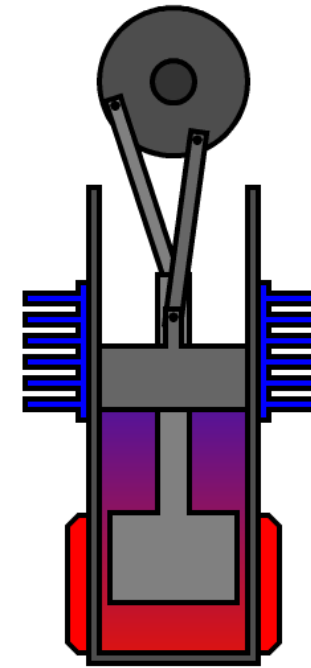
power piston (dark grey) has compressed the gas, the displacer piston (light grey) has moved so that most of the gas is adjacent to the hot heat exchanger



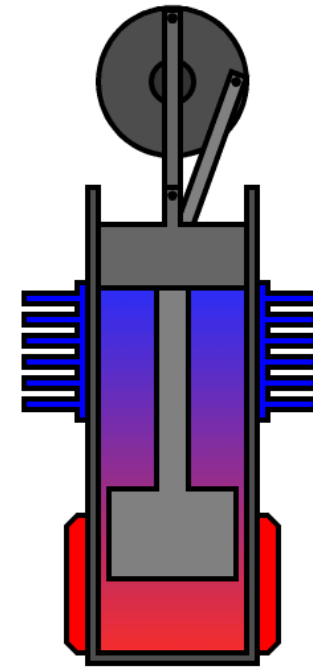
heated gas increases in pressure and pushes the power piston to the farthest limit of the power stroke.



displacer piston now moves, shunting the gas to the cold end of the cylinder.

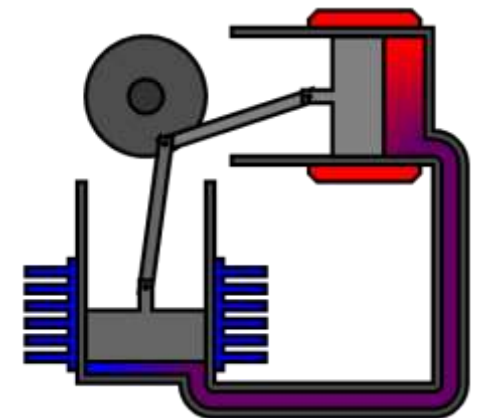
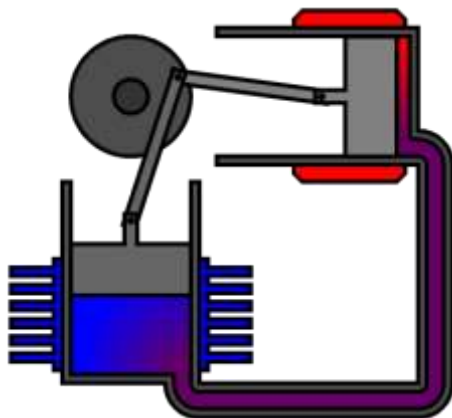
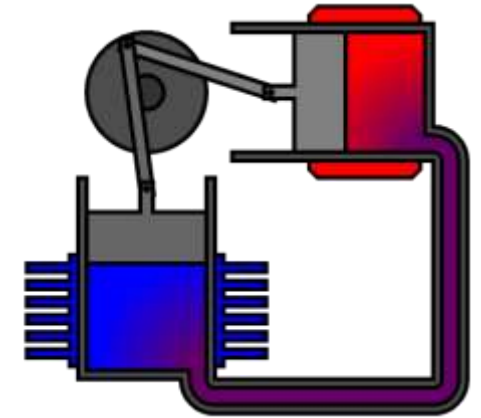
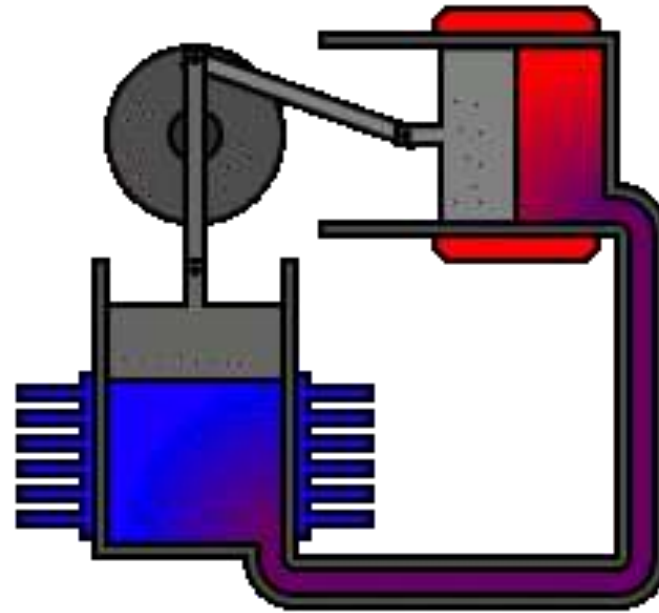
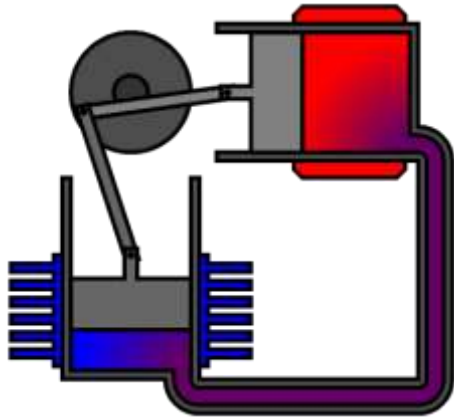


cooled gas is now compressed by the flywheel momentum. This takes less energy, since when it is cooled its pressure dropped.



# III.1.3 Regenerative Machines

- (alpha) Stirling machine



# III.1.3 Regenerative Machines

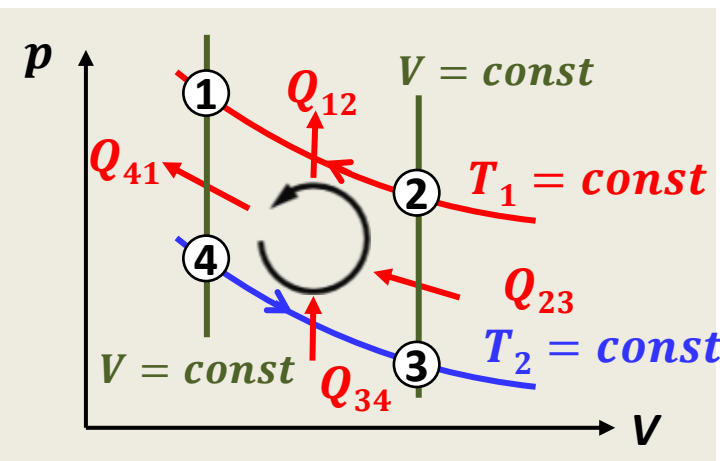
- Gifford-McMahon machine

uses compressor with switching valve instead of piston

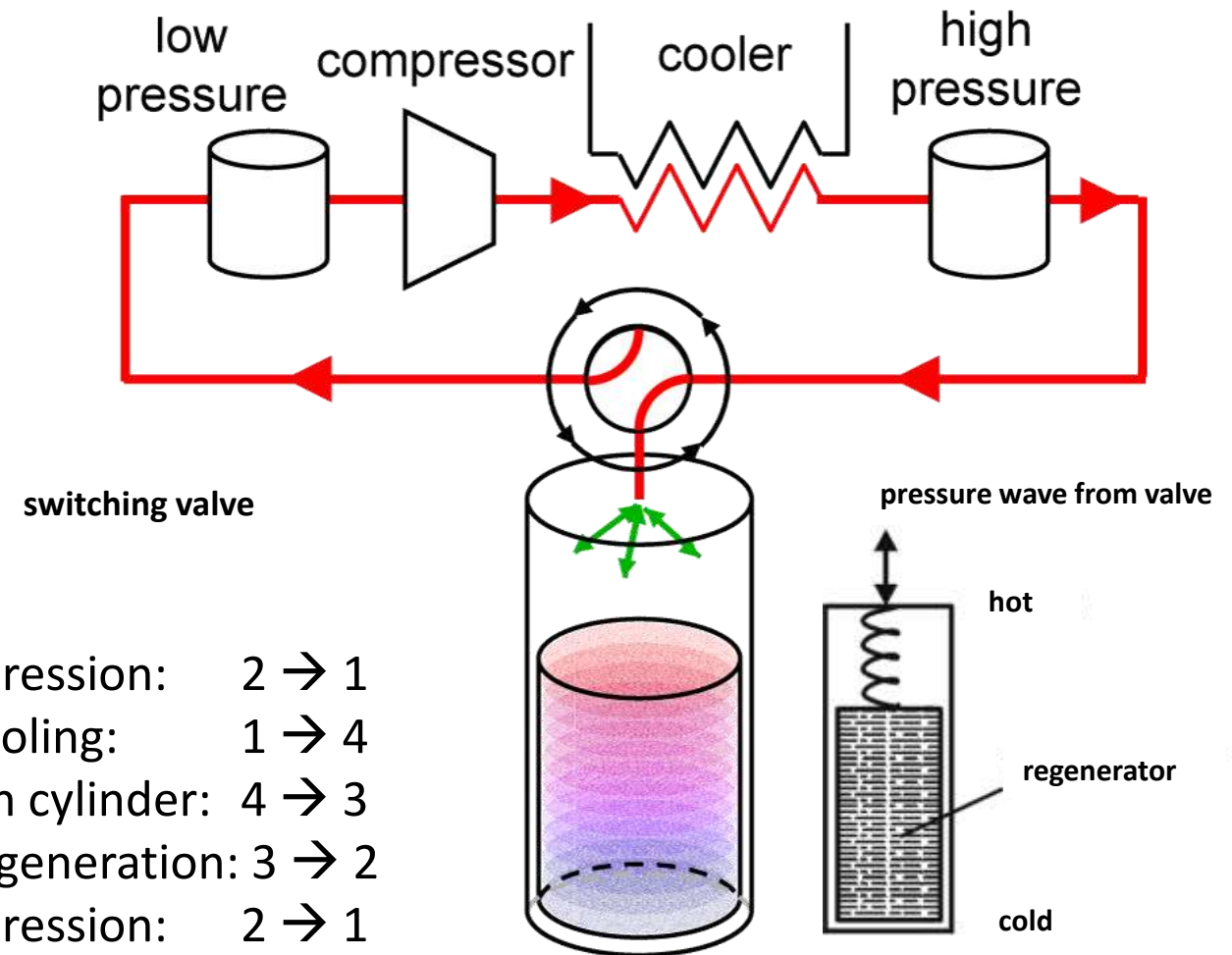
(counterclockwise, heat pump)

## heat pump

(conversion of mechanical work into heat)



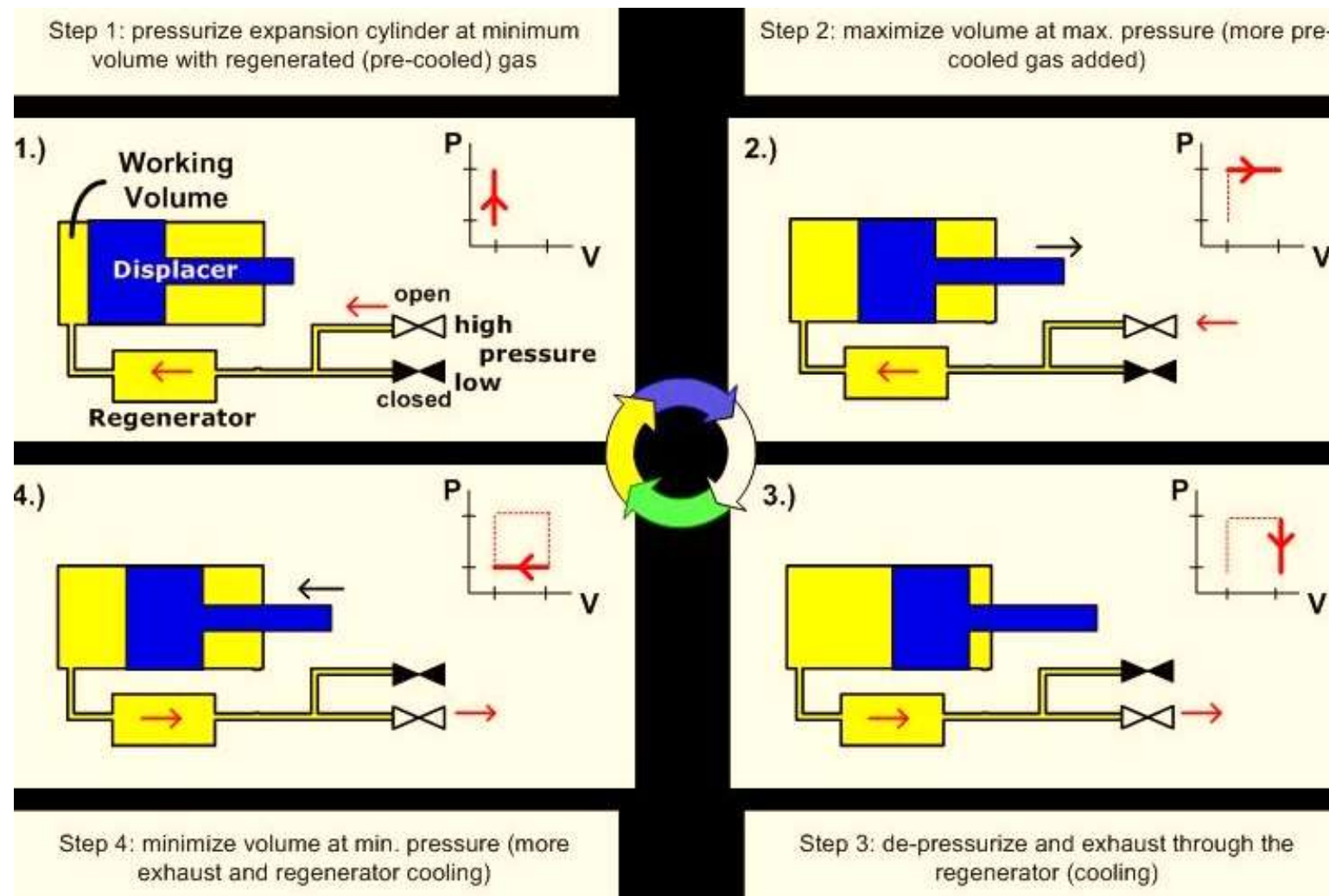
1. warm compression:  $2 \rightarrow 1$
2. isochoric cooling:  $1 \rightarrow 4$
3. expansion in cylinder:  $4 \rightarrow 3$
4. isochoric regeneration:  $3 \rightarrow 2$
5. warm compression:  $2 \rightarrow 1$





# III.1.3 Regenerative Machines

- Gifford-McMahon cycle



cryopump



two-stage cryocooler





# III.1.3 Regenerative Machines

- Pulse – Tube – Refrigerator

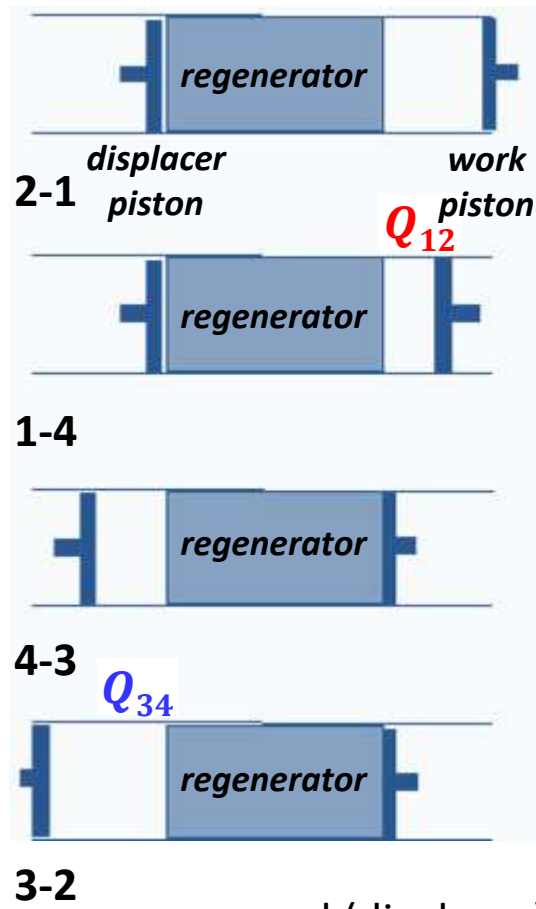
- the **pulse tube refrigerator (PTR)** or **pulse tube cryocooler** is based on the operation principle of Stirling coolers
- PTR is made without moving parts in the low temperature part (in contrast with other cryocoolers, e.g. Stirling cryocooler and Gifford-McMahon cooler)
- compact design possible → suitable for a wide variety of applications
- minimum temperature about 2.5 K (with  $^4\text{He}$ ) and 1.3 K (with  $^3\text{He}$ )

## ***applications:***

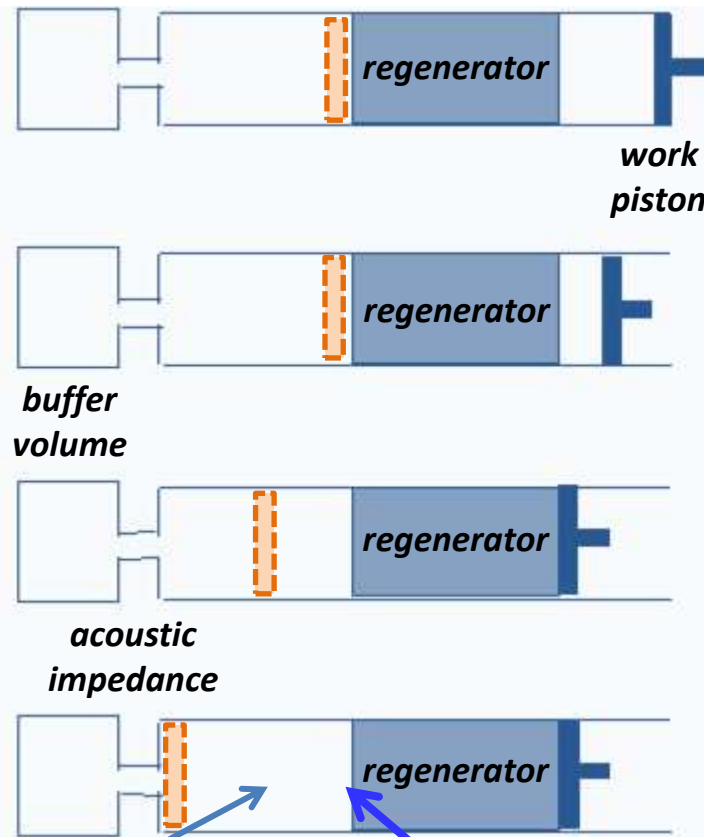
- industrial applications such as semiconductor fabrication (e.g. cryopumps)
- cooling of infrared sensors
- cooling of astronomical detectors (e.g. Atacama Cosmology Telescope or the QUBIC experiment – an interferometer for cosmology studies)
- **precoolers of dilution refrigerators**  
Kurt Uhlig (WMI), “Dry” dilution refrigerator with pulse-tube precooling, Cryogenics 44, (2004), pp. 53–57
- suggested to be used to liquefy oxygen on Mars

# III.1.3 Regenerative Machines

*Stirling Cooler*

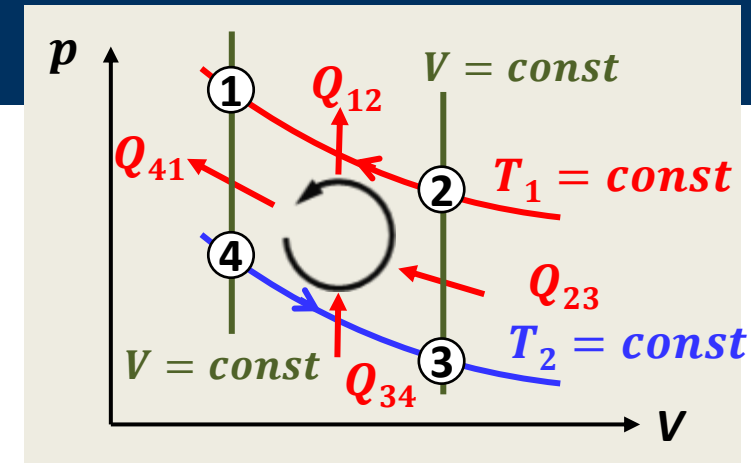


*Pulse – Tube – Refrigerator*



second (displacer) piston is replaced by pulse tube (gas piston)

coldest spot between regenerator and pulse tube



(counterclockwise, heat pump)

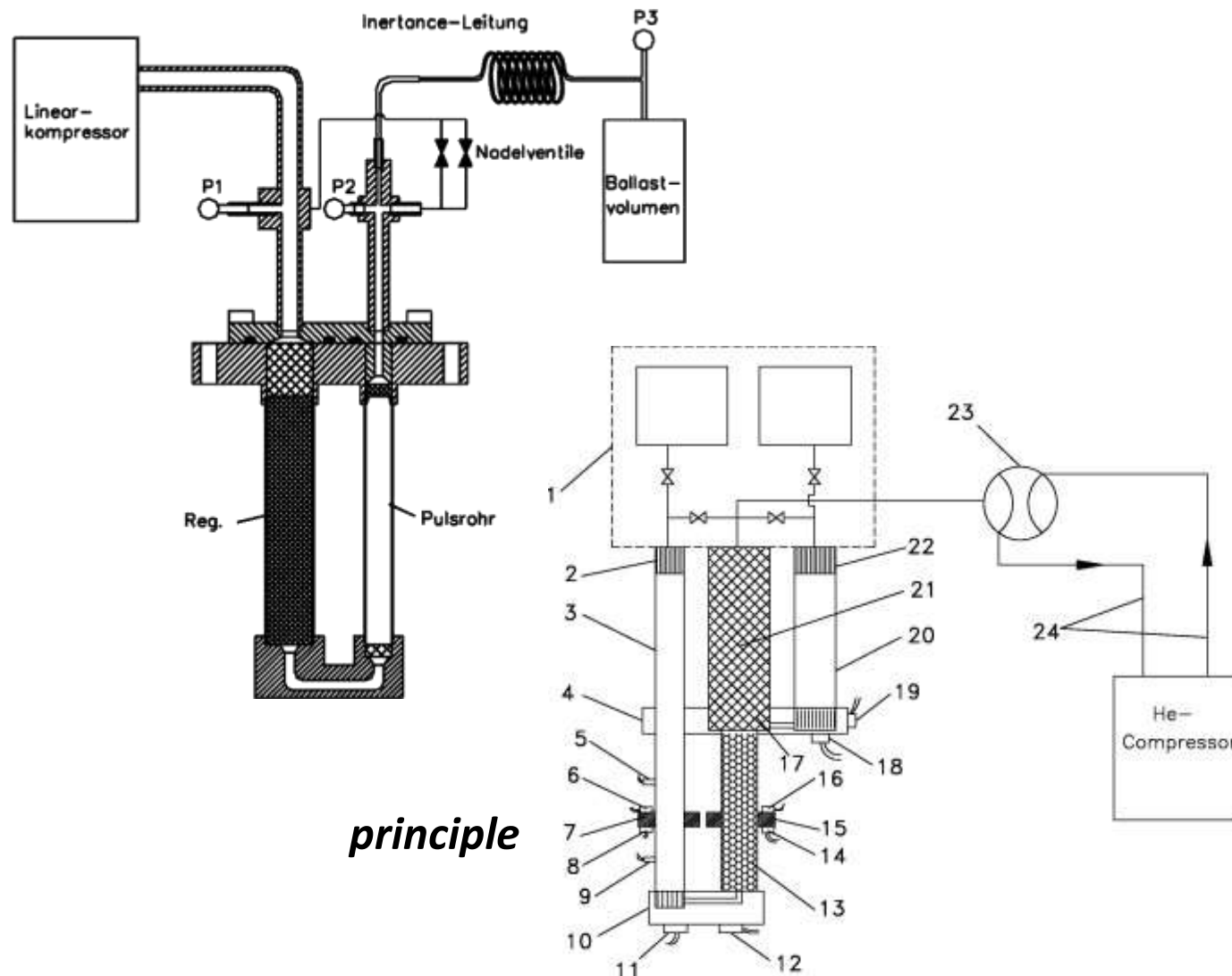
motion of **gas volume element** equivalent to motion of „**displacer piston**“

90° phase shift between motion of „**displacer piston**“ and „**work piston**“ realized by buffer volume

90° phase shift required for finite heat transport

# III.1.3 Regenerative Machines

- Pulse – Tube – Refrigerator (PTR) (realizations)



*principle*



Cryomech  
PT410



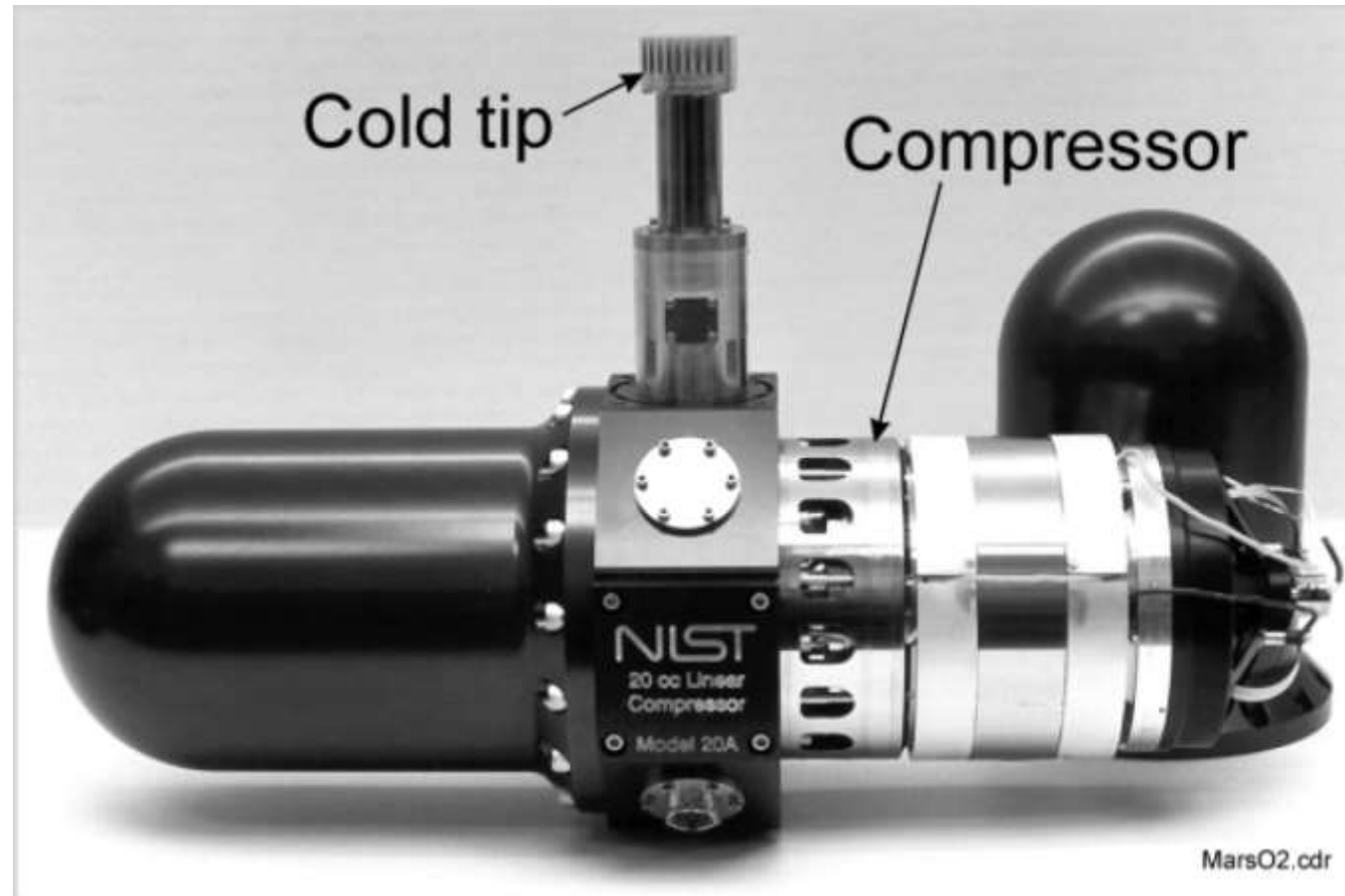
Cryomech  
PT425 RM

[www.cryomech.com](http://www.cryomech.com)

commercially available  
PTR with GM drive

1.0 W @ 4.2K, 35 W @ 45K,  $T_{\min} = 2.5 \text{ K}$   
(2-stage PTR)

# III.1.3 Regenerative Machines



*pulse tube refrigerator for studies of liquefying oxygen on Mars (580 mm total length)*

# III.1.3 Regenerative Machines

- Pulse – Tube – Refrigerator: summary

- presently very active development
- no moving parts at low temperatures

⇒ *long endurance*

*mobile base stations and satellite applications*

(e.g. for superconductive microwave filters)

- almost no vibrations
- efficiency lower than for displacer system
- only one simpler method:

⇒ *Joule-Thomson* cooling

# III.1.4 Joule-Thomson Cooling



**William Thomson (Lord Kelvin)**

Born: 26 June 1824, Belfast, Northern Ireland

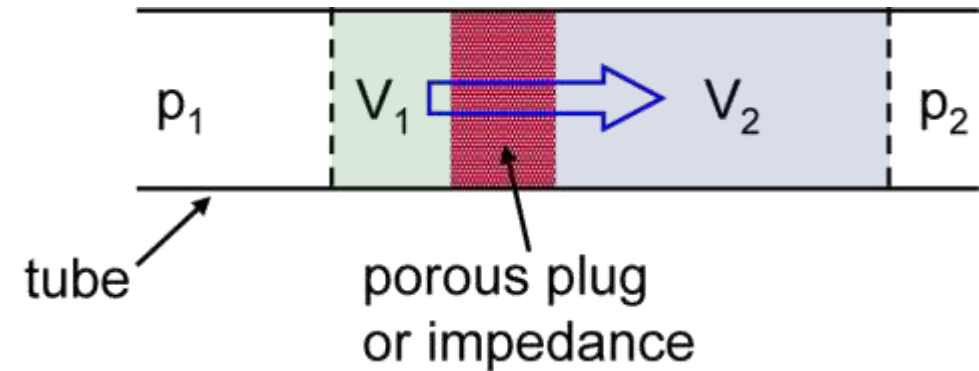
Died: 17 December 1907, Netherhall, Largs Ayrshire, Scotland



# III.1.4 Joule-Thomson Cooling

- Joule-Thomson process

- gas performs work against its own internal attractive forces
- working medium/gas ( $V_1$ ) flows through impedance and expands to  $V_2$



1<sup>st</sup> law of thermodynamics:  $\Delta U = \Delta Q + \Delta W$

$U_2 - U_1 = 0$  (adiabatic)

$-\int_0^{V_2} p_2 dV_2 - \int_{V_1}^0 p_1 dV_1 = -p_2 V_2 + p_1 V_1$

$$\Rightarrow U_2 + p_2 V_2 = U_1 + p_1 V_1$$

this means: process with **constant enthalpy**:  $H \equiv U + pV = \text{const.}$

- for **ideal gas**:  $p_1 V_1 = p_2 V_2$  and hence  $U_1 = U_2$ , respectively  $T_1 = T_2 \rightarrow$  **no cooling !!**

# III.1.4 Joule-Thomson Cooling

- Joule-Thomson process: **real gas**

transformation of gas into liquid on decreasing  $T$  and (or) increasing  $p$  due to **work against attractive interaction between the molecules**

- weak long-range attraction:**  
tends to keep molecules closer together, same effect as additional compression of the gas

$$p_{\text{eff}} = p + \frac{a}{V^2}$$

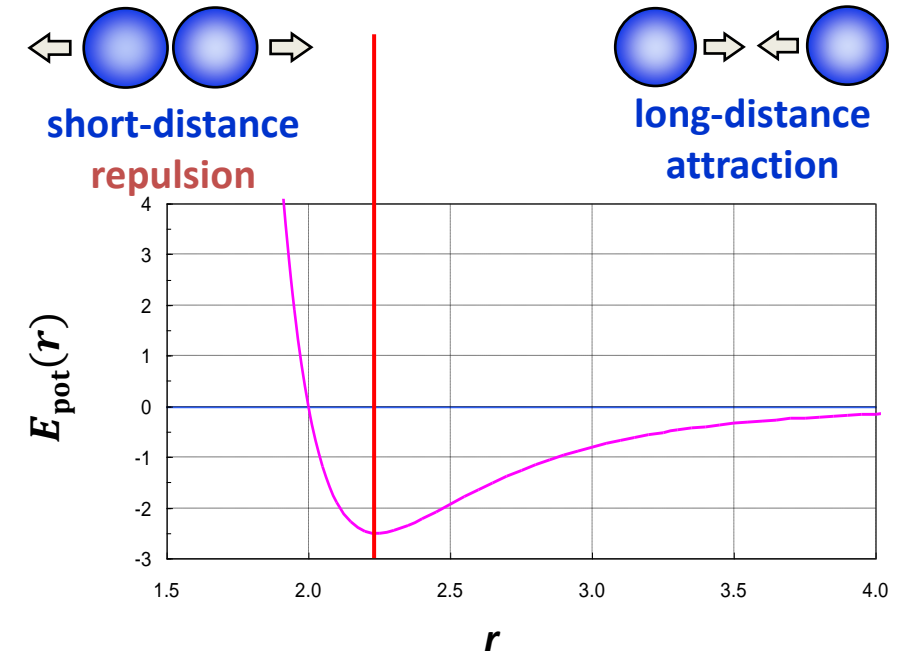
$a$  is a measure of the long-range attraction

- strong short-range repulsion:**  
molecules are rigid:  
 $p \rightarrow \infty$  as soon as the molecules “touch” each other.

$$V_{\text{eff}} = V - b$$

$b$  ( $\approx 4\pi\sigma^3/3$ ): “excluded volume” per particle

- Van der Waals equation:**  $\left(p + \frac{a}{V_m^2}\right)(V_m - b) = RT$



**expansion (decrease of pressure):**

*low pressure:*

attraction costs work  
 $\Rightarrow$  cooling of gas

*high pressure:*

repulsion provides work  
 $\Rightarrow$  heating of gas

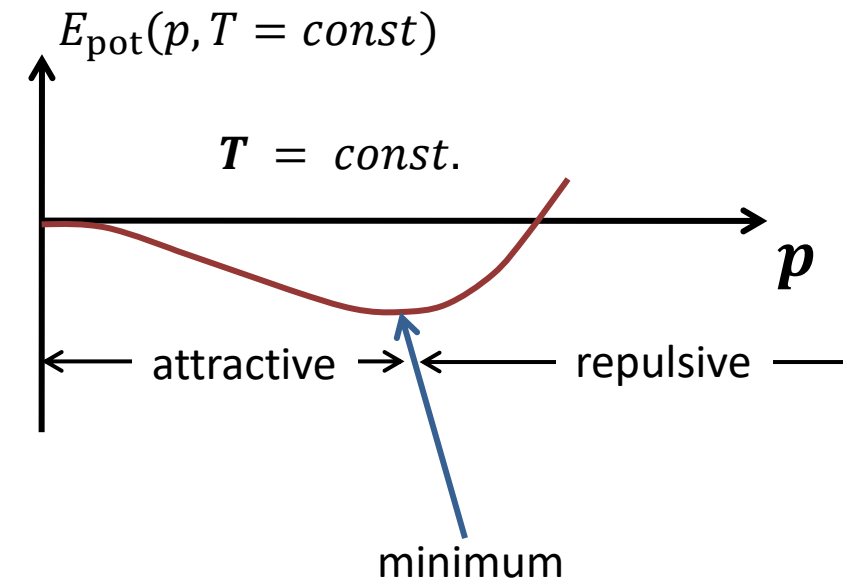
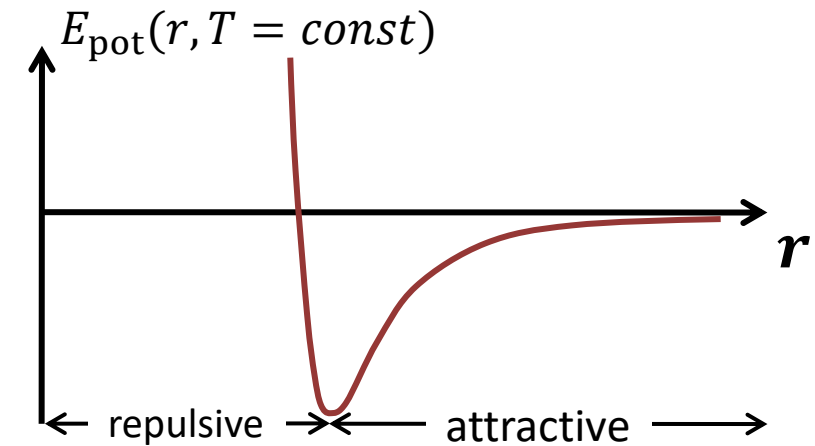


# III.1.4 Joule-Thomson Cooling

- Joule-Thomson process: **real gas**

interaction potential:

with  $p \propto n \equiv \frac{N}{V} = \frac{1}{\langle r^3 \rangle}$



# III.1.4 Joule-Thomson Cooling

- Joule-Thomson process: more detailed analysis of isenthalpic expansion

$$\Delta H = \left( \frac{\partial H}{\partial T} \right)_p \Delta T + \left( \frac{\partial H}{\partial p} \right)_T \Delta p = 0$$

with  $\left( \frac{\partial H}{\partial T} \right)_p = C_p \Rightarrow C_p \Delta T = - \left( \frac{\partial H}{\partial p} \right)_T \Delta p \Rightarrow$

$$-\frac{1}{C_p} \left( \frac{\partial H}{\partial p} \right)_T = \left( \frac{\Delta T}{\Delta p} \right)_H \equiv \mu_{JT}$$

**Joule-Thomson  
coefficient**

$\mu_{JT} > 0$ : cooling on expansion

$\mu_{JT} < 0$ : heating on expansion

with  $\Delta H = T\Delta S + V\Delta p \Rightarrow \left( \frac{\partial H}{\partial p} \right)_T = T \left( \frac{\partial S}{\partial p} \right)_T + V$

and  $\left( \frac{\partial S}{\partial p} \right)_T = - \left( \frac{\partial V}{\partial T} \right)_p$

$$\Rightarrow \mu_{JT} = \left( \frac{\Delta T}{\Delta p} \right)_H = -\frac{1}{C_p} \left( \frac{\partial H}{\partial p} \right)_T = \frac{1}{C_p} \left( T \left( \frac{\partial V}{\partial T} \right)_p - V \right)$$

# III.1.4 Joule-Thomson Cooling

- Joule-Thomson process: more detailed analysis of isenthalpic expansion – **ideal gas**

– for ideal gas:  $pV = RT \Rightarrow \left(\frac{\partial V}{\partial T}\right)_p = \frac{R}{p} = \frac{V}{T} \Rightarrow \mu_{JT} = 0$

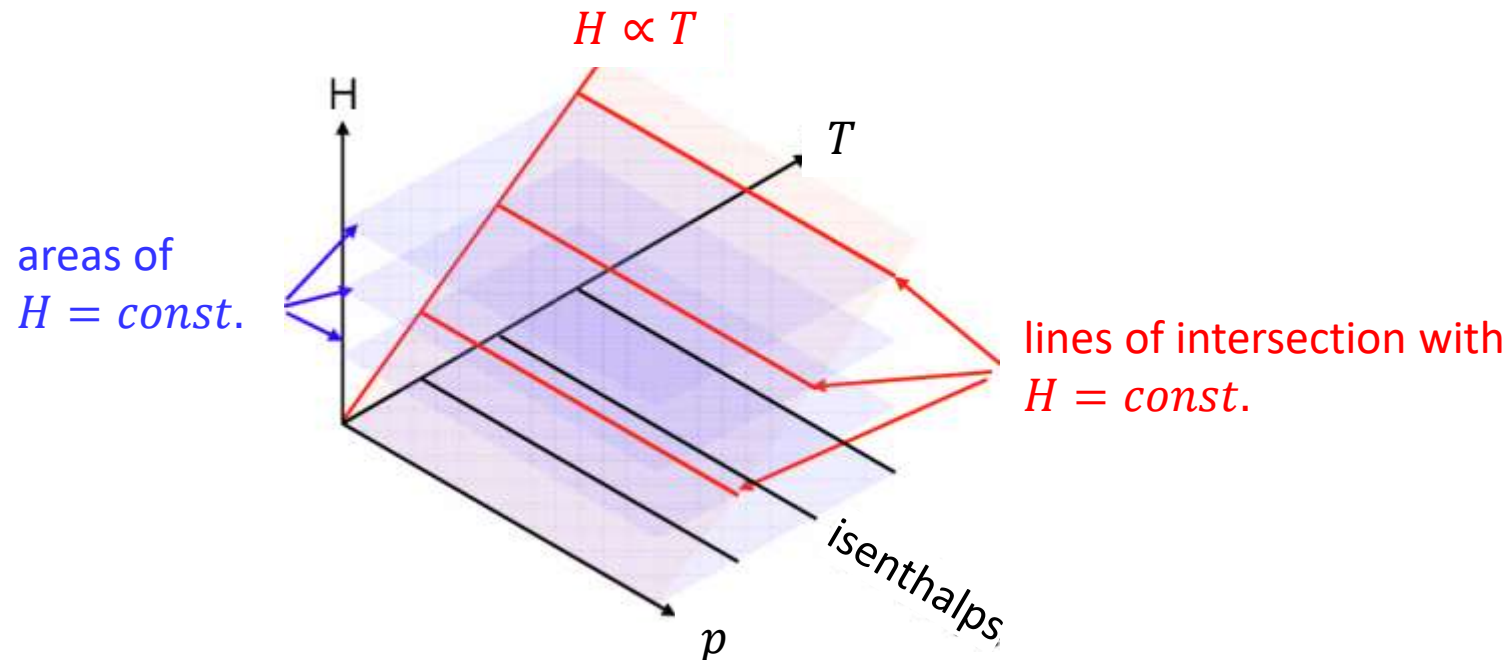
$$\mu_{JT} = \left(\frac{\Delta T}{\Delta p}\right)_H = -\frac{1}{C_p} \left(\frac{\partial H}{\partial p}\right)_T = \frac{1}{C_p} \left( T \left(\frac{\partial V}{\partial T}\right)_p - V \right)$$

$$H(T, p) = U + pV = \frac{3}{2} Nk_B T + Nk_B T = \frac{5}{2} Nk_B T = \text{const.} \Rightarrow \mu_{JT} \propto \left(\frac{\partial H}{\partial p}\right)_T = 0$$

equipartition theorem  
for monoatomic gas

ideal gas law

@  $T = \text{const}$



# III.1.4 Joule-Thomson Cooling

- Joule-Thomson process: more detailed analysis of isenthalpic expansion – **real gas**

– for real gas:  $\left(p + \frac{a}{V_m^2}\right)(V_m - b) = RT \implies H(T, p) = U + pV = \frac{5}{2}Nk_B T + \Delta U(p, T) \neq \text{const.}$

– **at low densities:** we can use **approximation**  $p \gg \frac{a}{V^2}, V \gg b$  and obtain

$$pV + \frac{a}{V} - pb = RT, \quad \left| \frac{\partial}{\partial T}(\dots)_p \right| \implies p \left( \frac{\partial V}{\partial T} \right)_p - \frac{a}{V^2} \left( \frac{\partial V}{\partial T} \right)_p = R \Rightarrow \left( \frac{\partial V}{\partial T} \right)_p = \frac{R}{p - \frac{a}{V^2}}$$

insert into

$$\mu_{JT} = \left( \frac{\Delta T}{\Delta p} \right)_H = -\frac{1}{C_p} \left( \frac{\partial H}{\partial p} \right)_T = \frac{1}{C_p} \left( T \left( \frac{\partial V}{\partial T} \right)_p - V \right)$$

$$\implies \mu_{JT} = \left( \frac{\Delta T}{\Delta p} \right)_H \approx \frac{1}{C_p} \left( 2 \frac{a}{RT} - b \right)$$

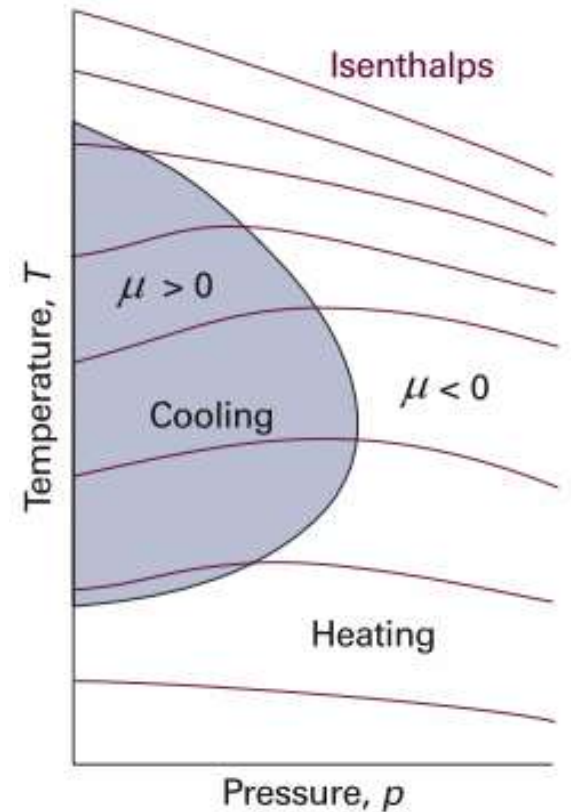
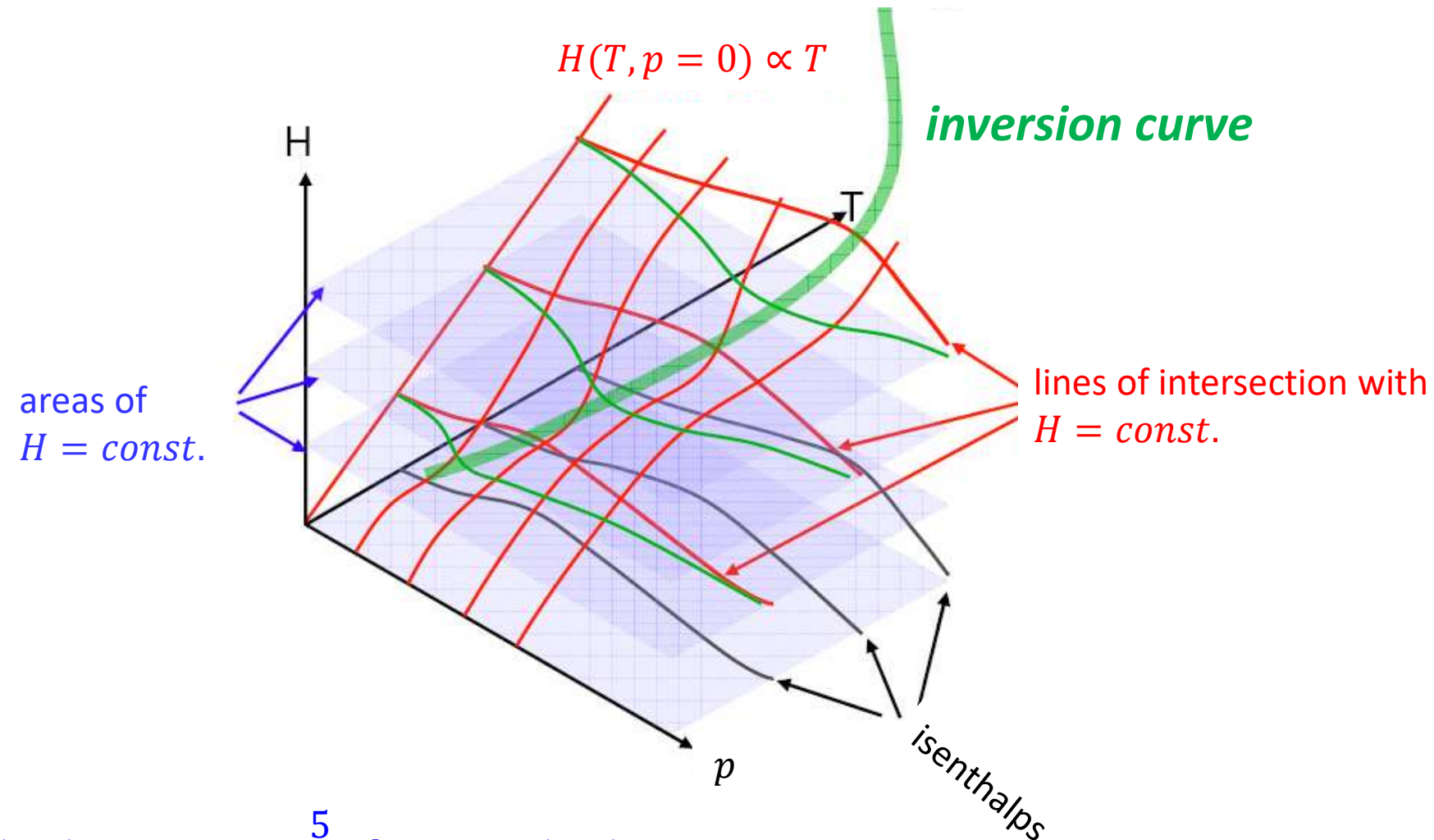
$\mu_{JT} > 0$  for  $T < 2a/bR \Rightarrow$  cooling on expansion  
 $\mu_{JT} < 0$  for  $T > 2a/bR \Rightarrow$  heating on expansion

**inversion temperature:**

$$T_{\text{inv}} = \frac{2a}{bR}$$

# III.1.4 Joule-Thomson Cooling

- Joule-Thomson process: more detailed analysis of isenthalpic expansion – **real gas**



$$H(T, p) = U + pV = \frac{5}{2} Nk_B T + \Delta U(p, T) \neq \text{const.}$$

# III.1.4 Joule-Thomson Cooling

- Joule-Thomson process: more detailed analysis of isenthalpic expansion – **real gas**

- without “low density” approximation

$$\mu_{JT} = \frac{(2a/RT)(1 - b/V^2) - b}{C_p[1 - (2a/VRT)(1 - b/V)^2]}$$

low density **approximation**  $p \gg \frac{a}{V^2}, V \gg b$ :

$$\mu_{JT} \approx \frac{1}{C_p} \left( 2 \frac{a}{RT} - b \right)$$

**inversion curve:** points where  $\mu_{JT} = 0$ :

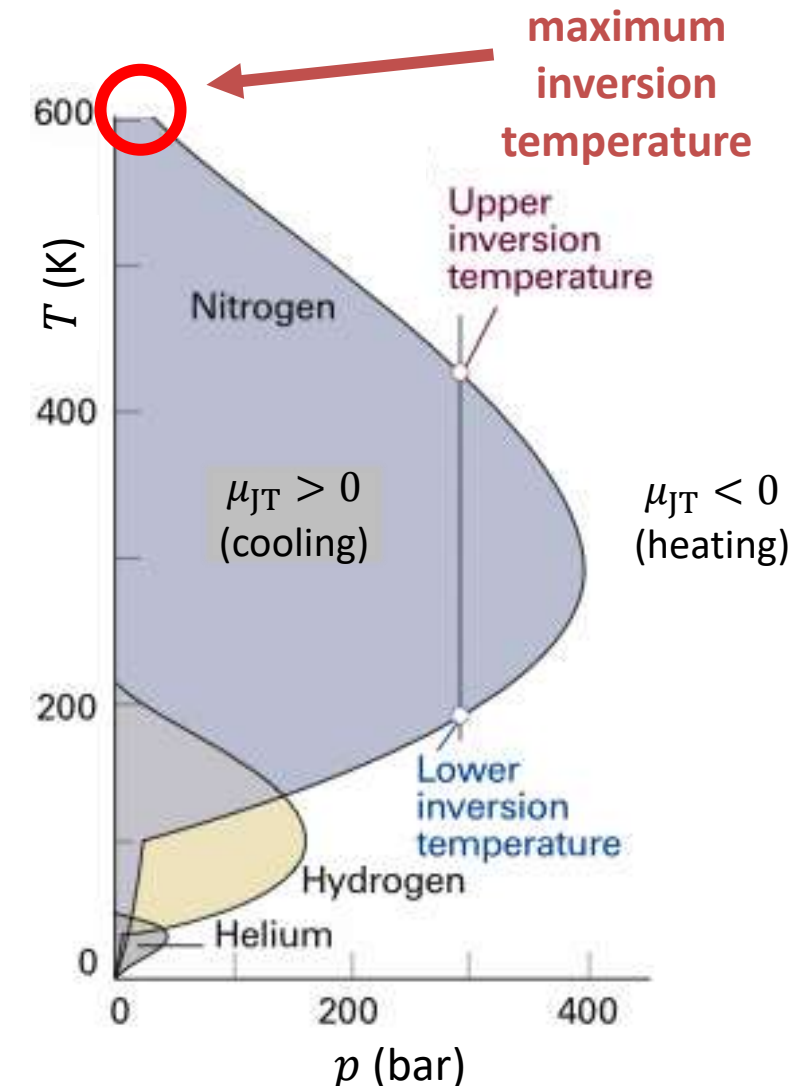
for vdW gas:  $(2a/RT)(1 - b/V)^2 = b$

**inversion temperature:**

$$T_{inv} = \frac{2a}{bR} \left( 1 - \frac{b}{V} \right)^2$$

equation of state gives  $T_{inv}(p, T)$

maximum inversion temperature:  $T_{inv} = \frac{2a}{bR}$

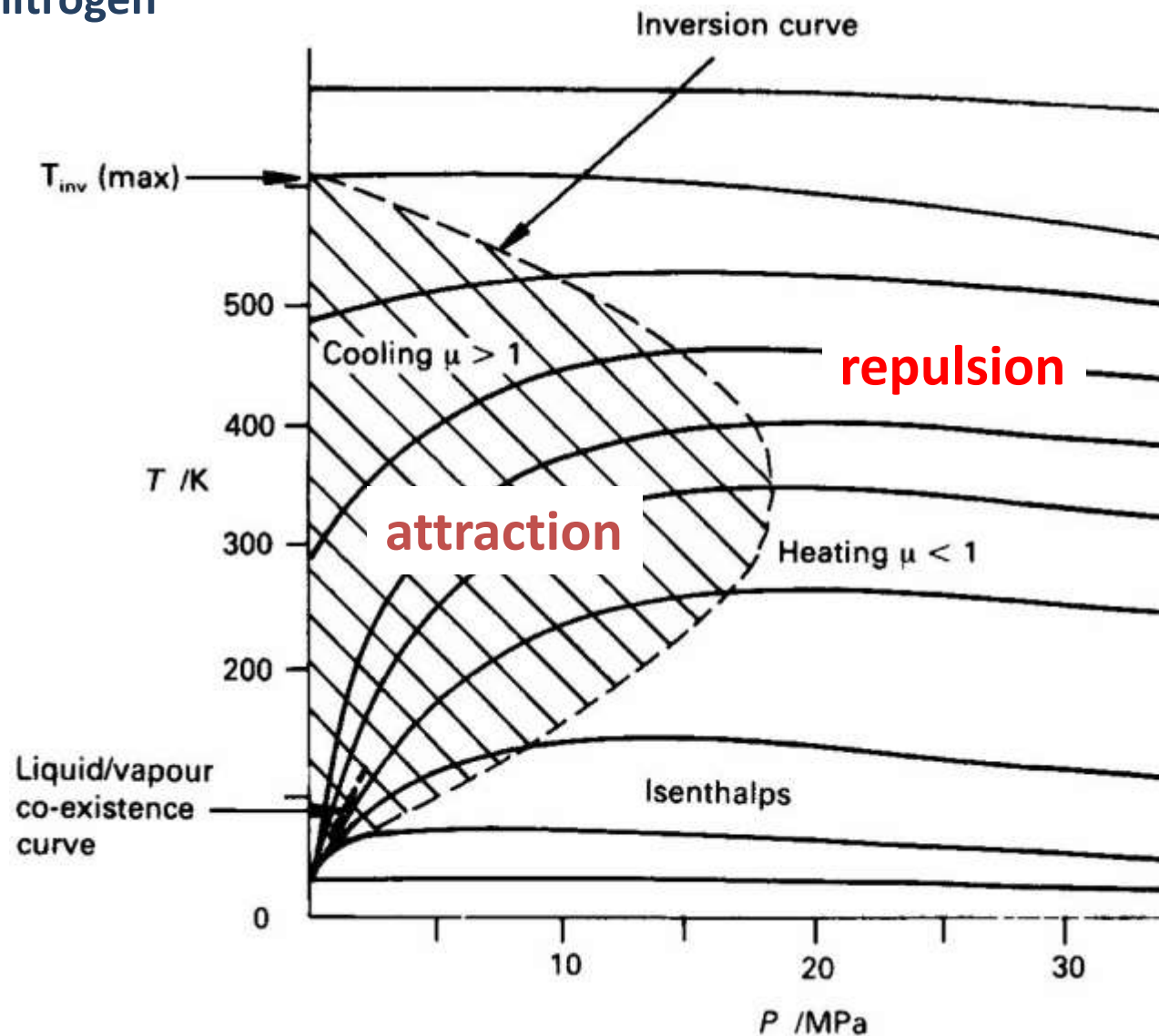


# III.1.4 Joule-Thomson Cooling

- Joule-Thomson coefficient for nitrogen

$$\mu_{JT} \propto \left( \frac{\partial T}{\partial p} \right)_H$$

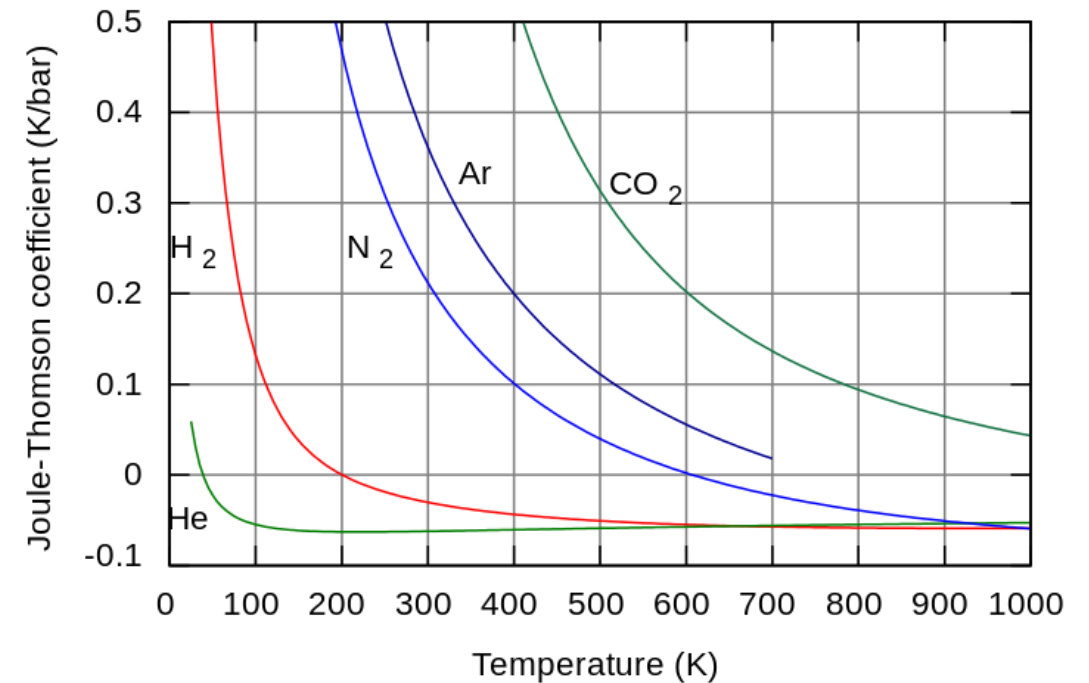
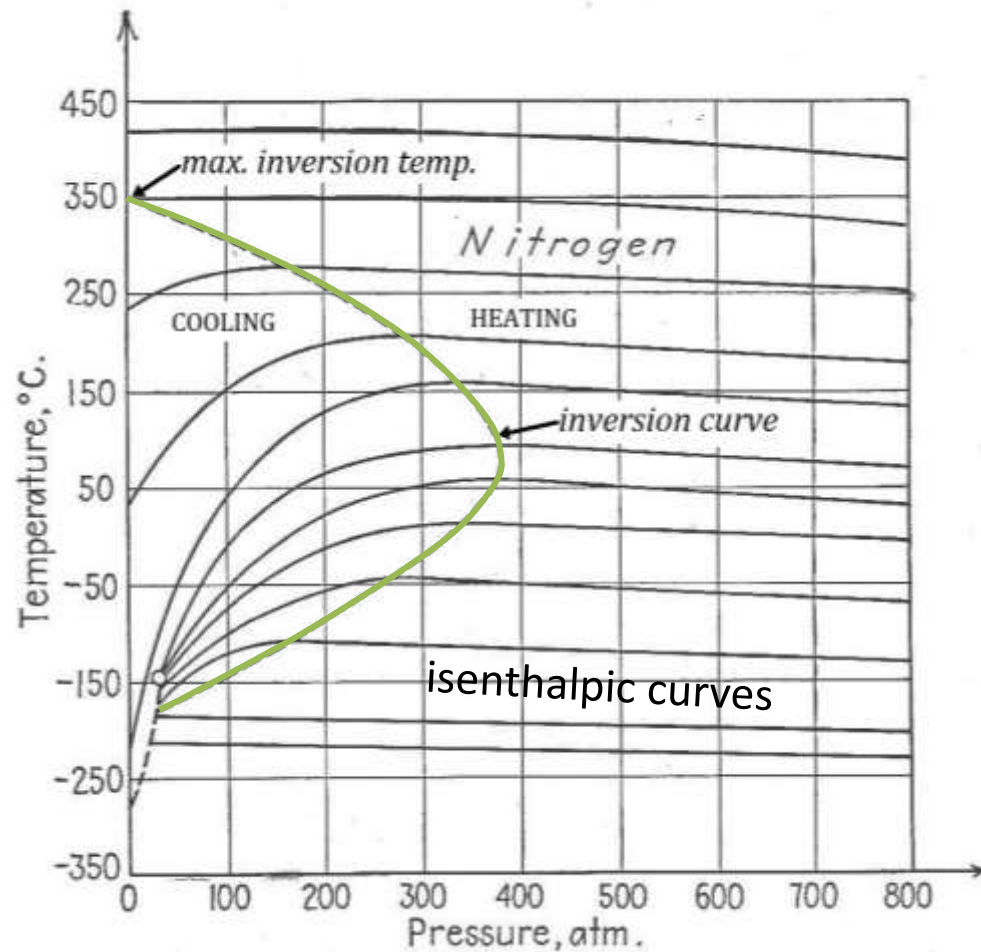
slope of isenthalps





# III.1.4 Joule-Thomson Cooling

- Joule-Thomson coefficient for nitrogen



Joule-Thomson coefficients for various gases at atmospheric pressure

# III.1.4 Joule-Thomson Cooling

- Joule-Thomson cooling: inversion temperature

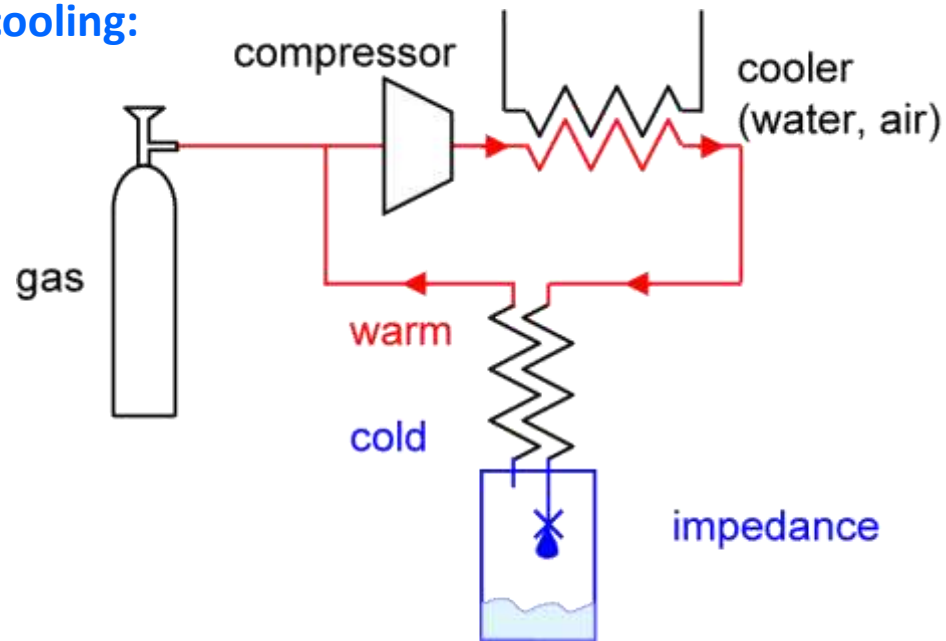
gas	maximum inversion temperature [K]
Helium-3	(23)
Helium-4	45
Hydrogen	205
Neon	250
Nitrogen	621
Air	603
Carbon monoxide	652
Argon	794
Oxygen	761
Methane	939
Carbon dioxide	1500
Ammonia	1994

*vdW gas can be  
liquefied only for  
 $T < T_{\text{inv}}$  !!!*

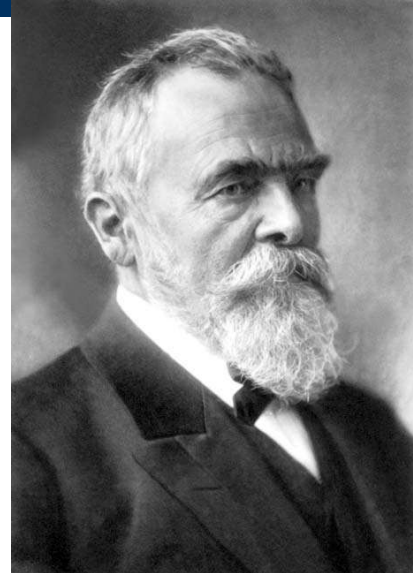
# III.1.4 Joule-Thomson Cooling

- “Linde process”

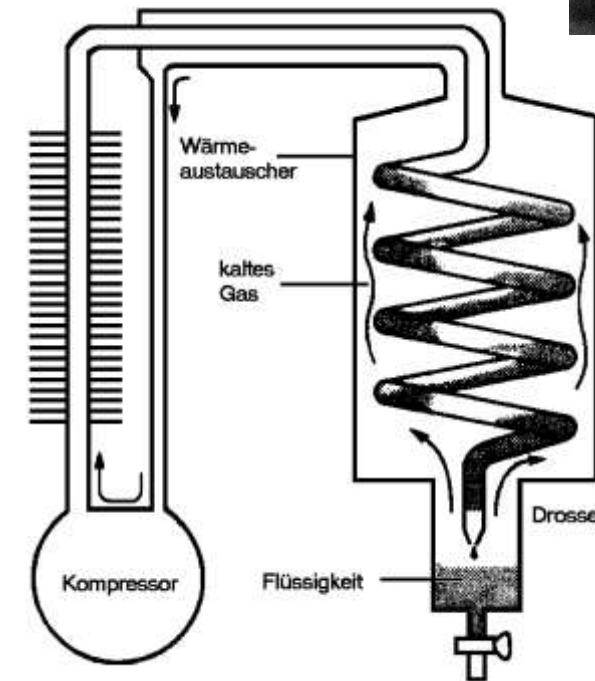
closed cycle cooling:



- gas is cooled by JT-expansion until liquid drops out the impedance
- patent application by Carl von Linde on May 12, 1903 (liquefaction of oxygen)



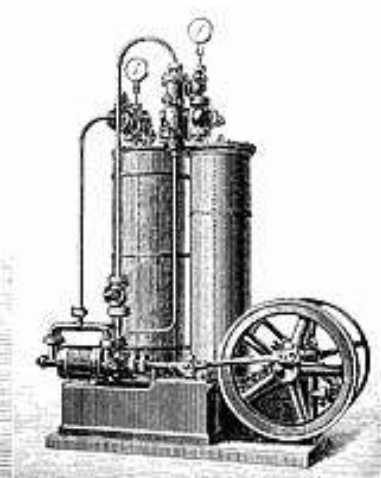
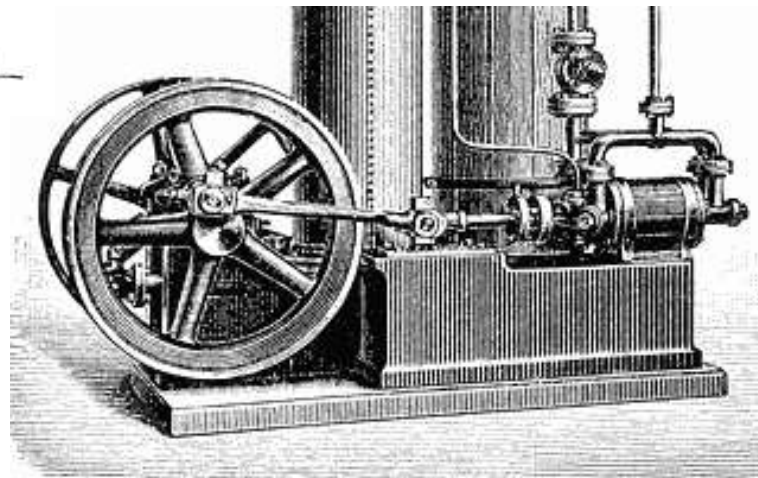
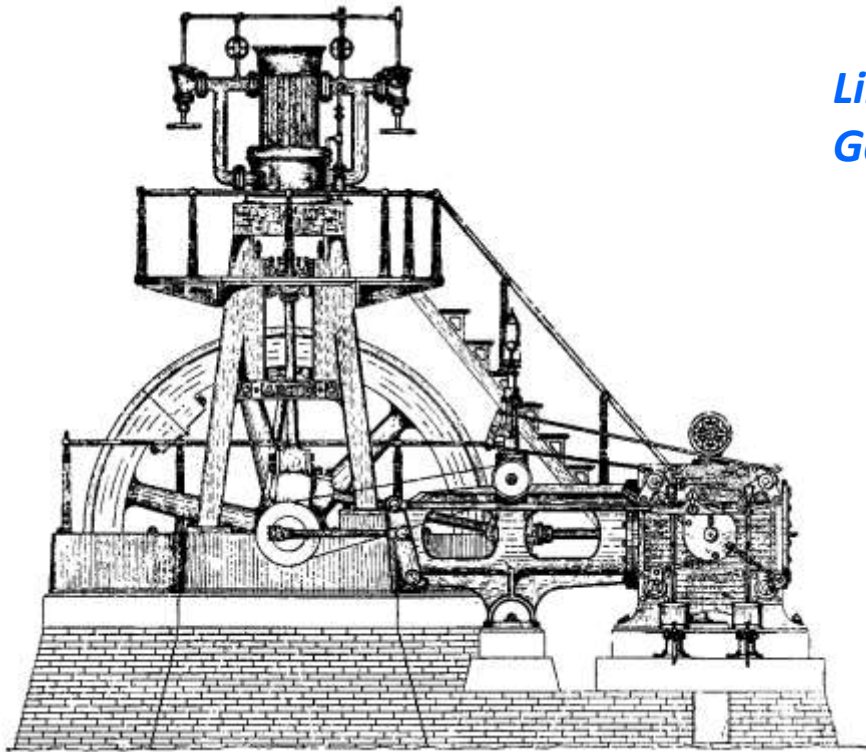
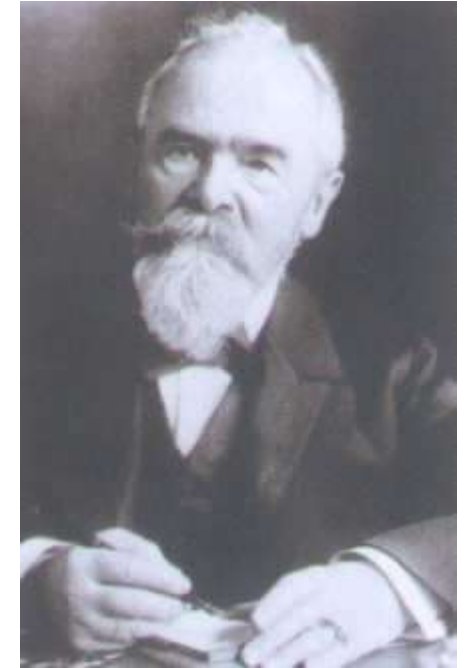
Carl von Linde  
(1842 – 1934)



(Source: PTB Braunschweig)

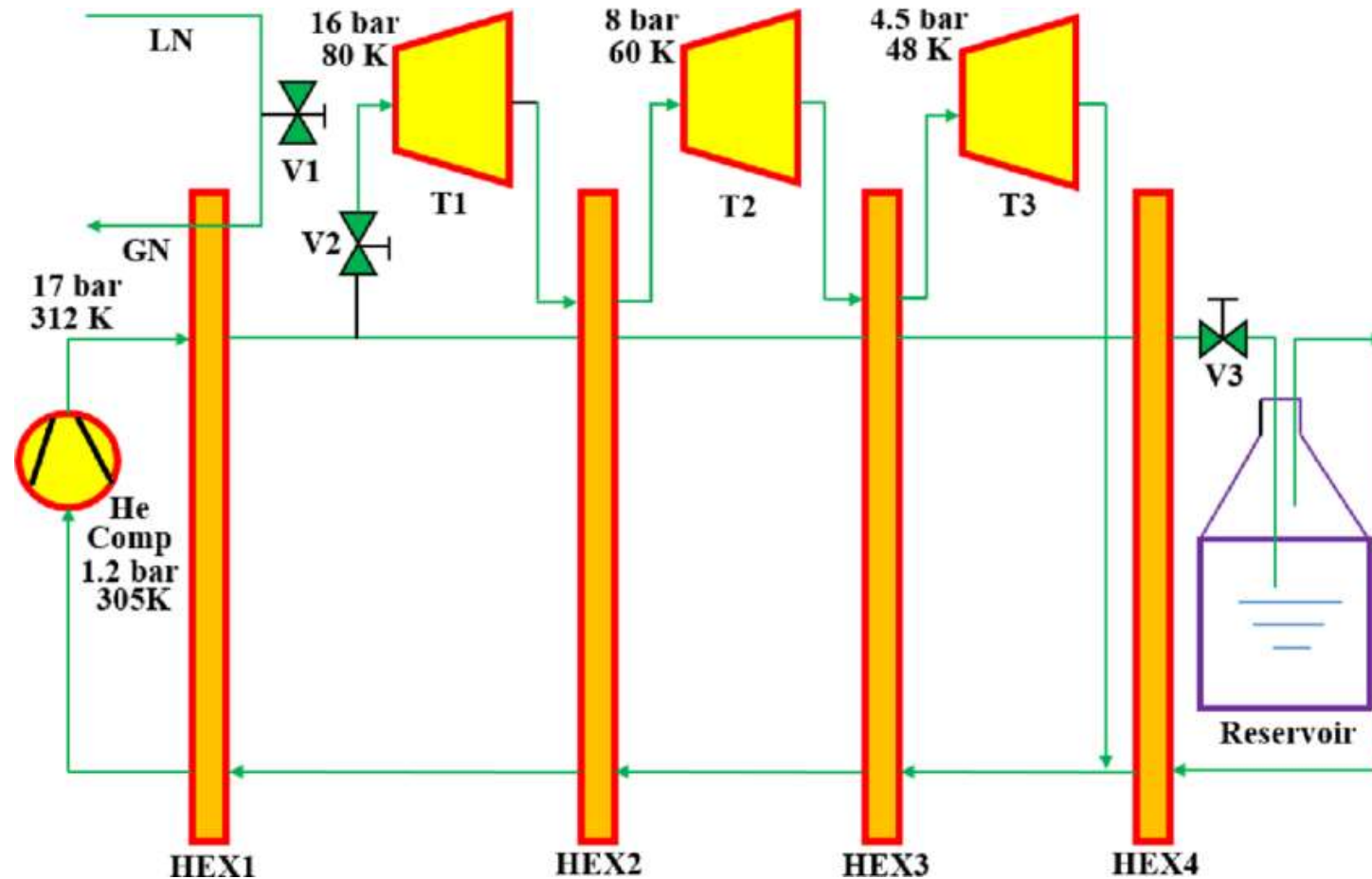
# III.1.4 Joule-Thomson Cooling

*Lindesche  
Gasverflüssigungsanlage (1895)*



# III.1.4 Joule-Thomson Cooling

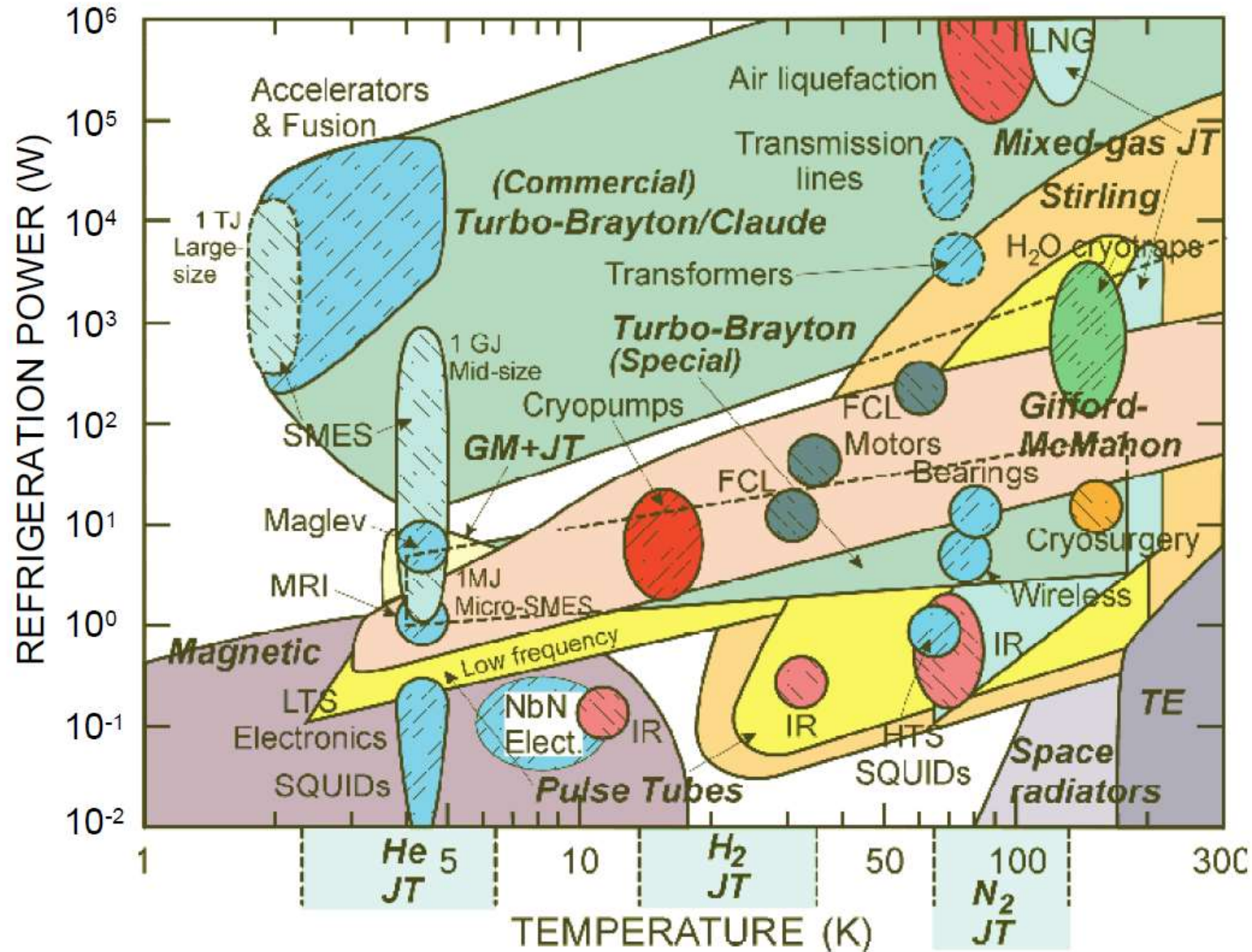
- Helium liquefaction by Joule-Thomson cooling



schematics of a Helium liquefier

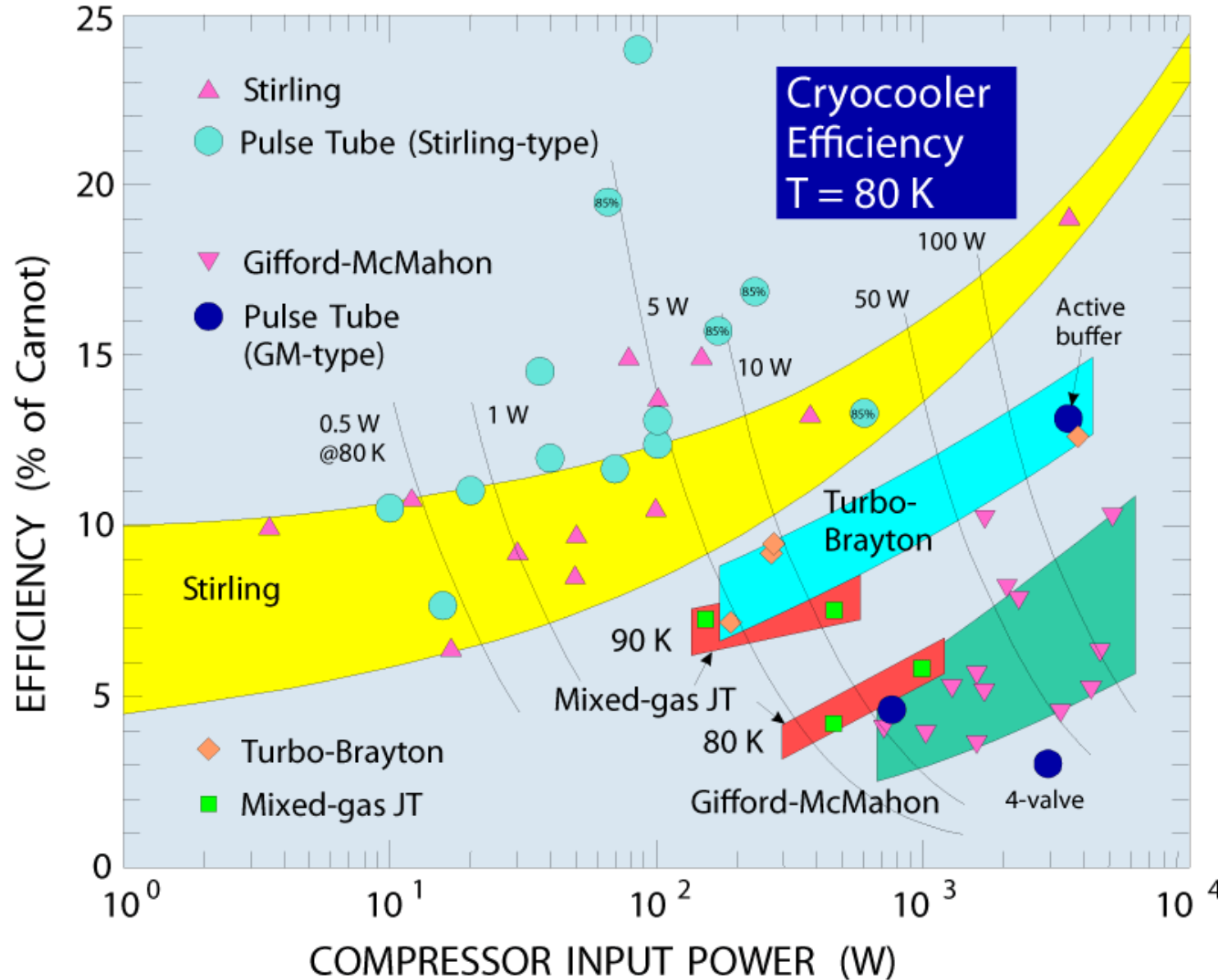


# III.1.5 Summary



application of cooling techniques  
in plane of refrigeration power  
versus temperature

# III.1.5 Summary

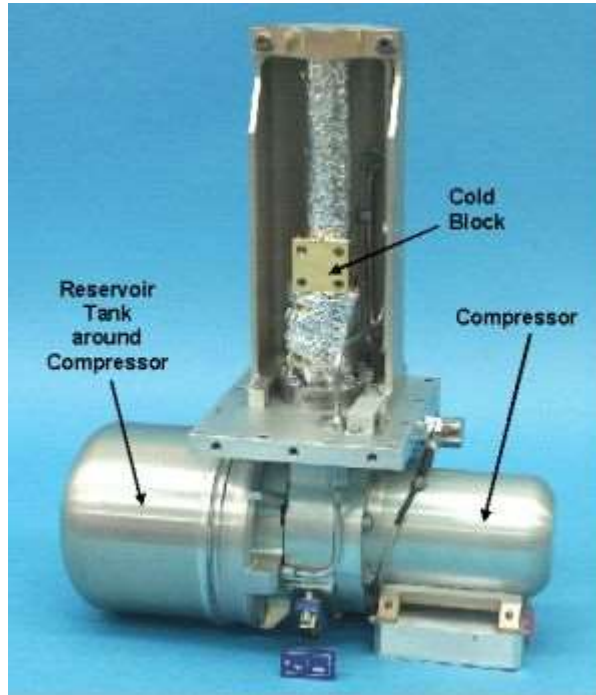


efficiency of cryocoolers



# III.1.5 Summary

*Northrop Grumman's HEC cryocooler*



## specification for cryocooler:

- 1 Watt of cooling @ 80 K, rejecting heat at 300 K
- 10 year life
- 230 K to 340 K survival temperature
- survival of launch vibration (non-operating)
- low exported vibration
- high efficiency
- no maintenance possible → oil-free

*Sumitomo Heavy Industries*



## Stirling cycle miniature cryocooler:

- lightweight cooler, ideal for cooling of sensors and other electronics when low power consumption is important
- mean time before failure of 24,000 hours
- cooling capacity of 1 W @ 80 K
- power consumption of only 55 W.



Walther  
Meißner  
Institut

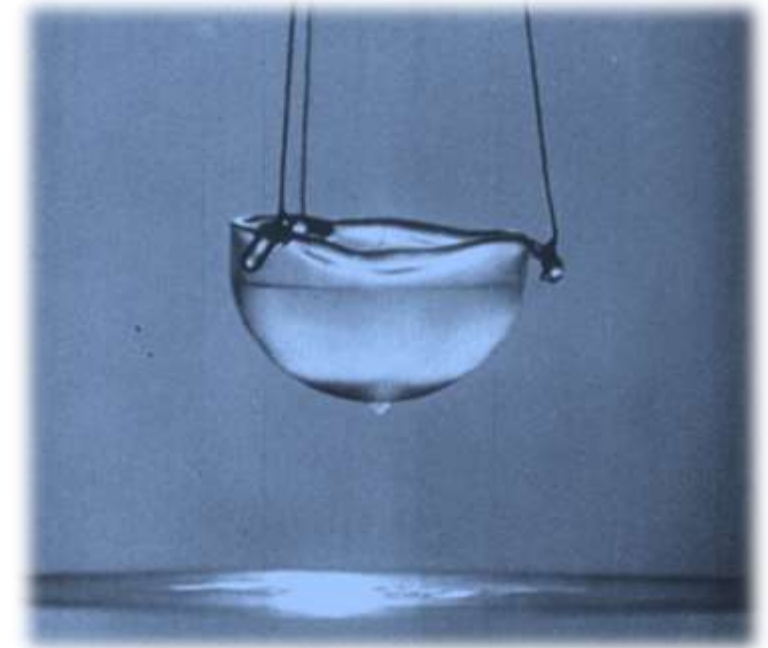
BAaW

BAYERISCHE  
AKADEMIE  
DER  
WISSENSCHAFTEN

Technische  
Universität  
München



# Superconductivity and Low Temperature Physics II



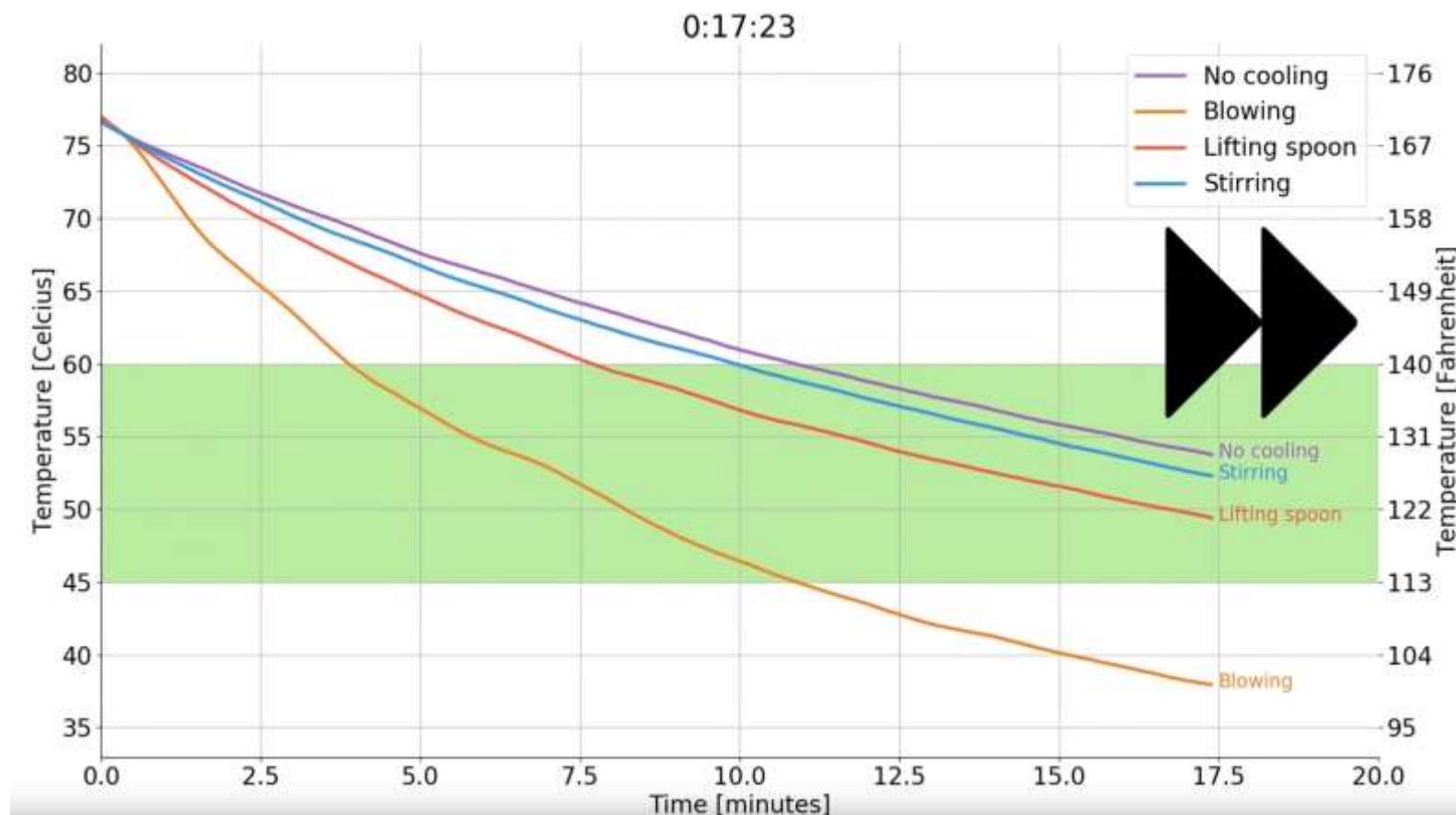
**Lecture No. 12**

**R. Gross**

**© Walther-Meißner-Institut**

# III.1.6 Evaporation Cooling

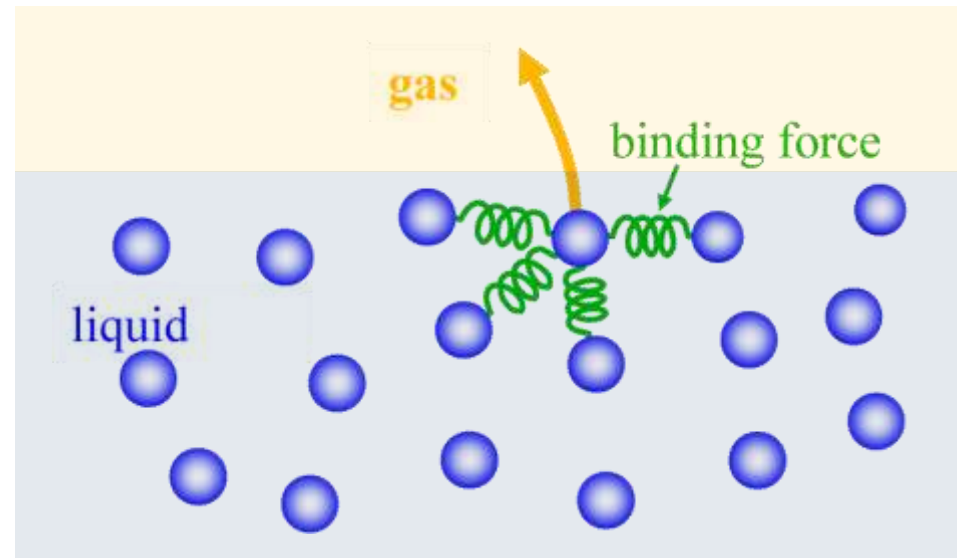
R. Gross © Walther-Meißner-Institut (2004 - 2023)



# III.1.6 Evaporation Cooling

- physics of evaporation cooling
  - everyday experience: sweating, wind direction, cooling of coffee, ...  
 ⇒ **moisten finger, evaporation cooling**

- microscopically:



- evaporation: ***work required to overcome binding forces***
  - ⇒ **only the fastest molecules will do it**
  - ⇒ high-energy particles are lost
  - ⇒ liquid cools down

# III.1.6 Evaporation Cooling

- physics of evaporation cooling
  - limit of evaporation cooling:  $k_B T$  becomes too small compared to  $\Delta H_{\text{vap}}$  when lowering  $T$  (heat of evaporation)
  - $\Delta H_{\text{vap}}$  should be small to reach large cooling power at low temperatures
  - numbers: about 1 K can be reached with liquid  $^4\text{He}$ , about 0.3 K with  $^3\text{He}$
  - boiling point can be calculated by using the Clausius-Clapeyron equation, if heat of vaporization and the vapor pressure of the liquid at a certain temperature are known

# III.1.6 Evaporation Cooling

- physics of evaporation cooling: Clausius-Clapeyron equation

$$\frac{dp}{dT} = \frac{S_{\text{gas}} - S_{\text{liquid}}}{V_{\text{gas}} - V_{\text{liquid}}} = \frac{\Delta H_{\text{vap}}}{(V_{\text{gas}} - V_{\text{liquid}})T} \simeq \frac{\Delta H_{\text{vap}}}{V_{\text{gas}} T}$$

$\Delta H_{\text{vap}}$ : molar latent heat [J/mole]  
 $\approx 90$  J/mole for  $^4\text{He}$

- approximate expression using  $pV = RT$  (ideal gas):

$$\frac{dp}{p} = \frac{\Delta H_{\text{vap}}}{RT^2} dT$$

- integration yields (assuming that  $\Delta H_{\text{vap}}$  is constant over the considered  $T$  range):

$$\ln\left(\frac{p}{p_0}\right) = \frac{\Delta H_{\text{vap}}}{R} \left(\frac{1}{T_0} - \frac{1}{T}\right)$$

- normal boiling temperature:

$$T_B(p) = \left( \frac{1}{T_0} - \frac{R}{\Delta H_{\text{vap}}} \ln\left(\frac{p}{p_0}\right) \right)^{-1}$$

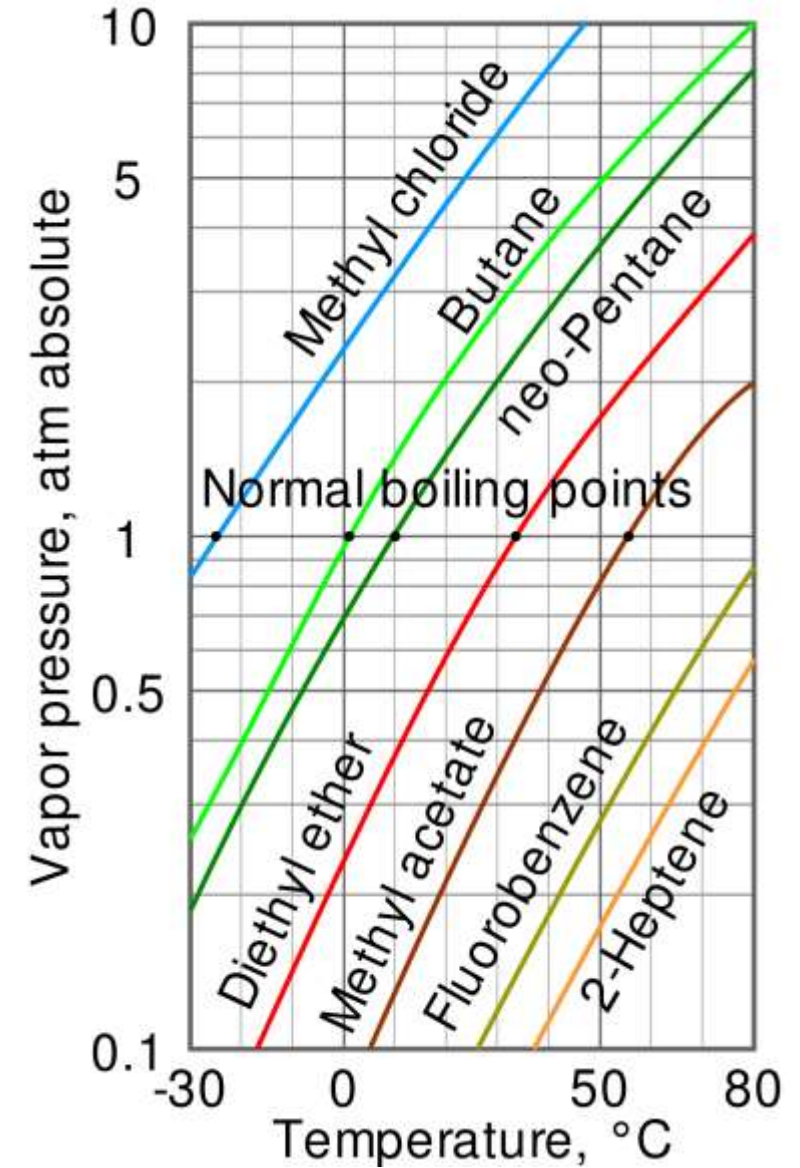
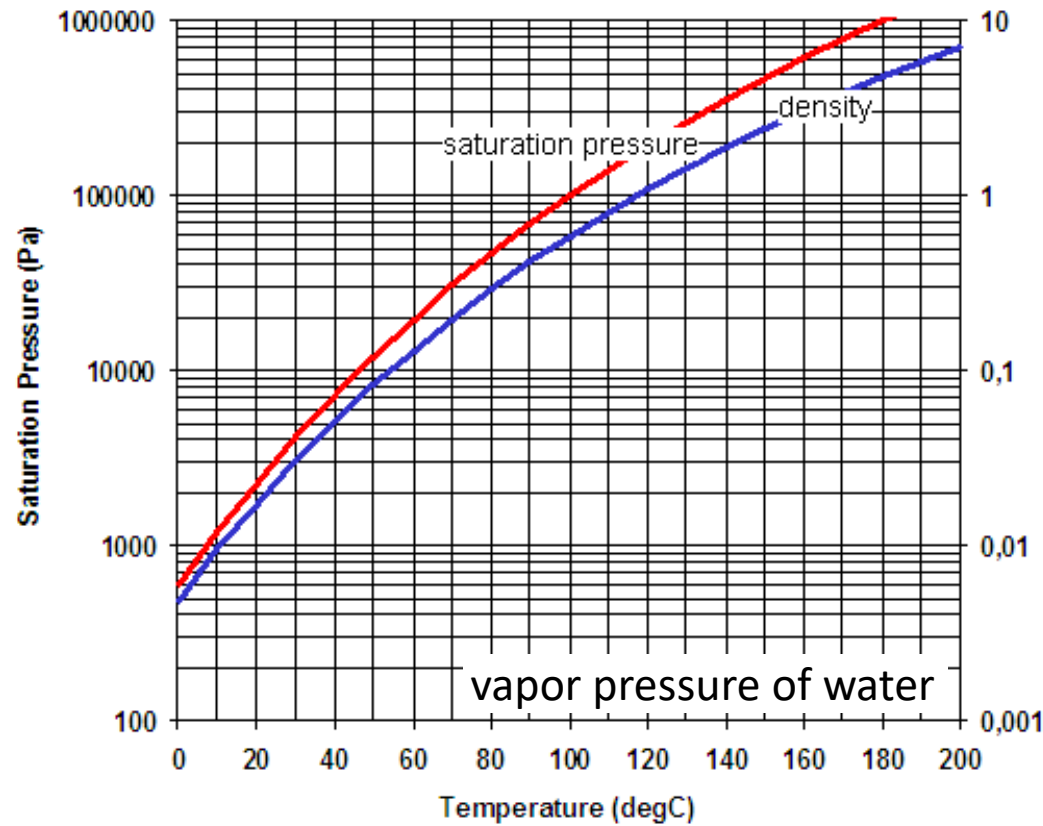
pressure above liquid

boiling temperature at  $p_0$

boiling point corresponds to the temperature at which the vapor pressure of the liquid equals the surrounding environmental pressure

# III.1.6 Evaporation Cooling

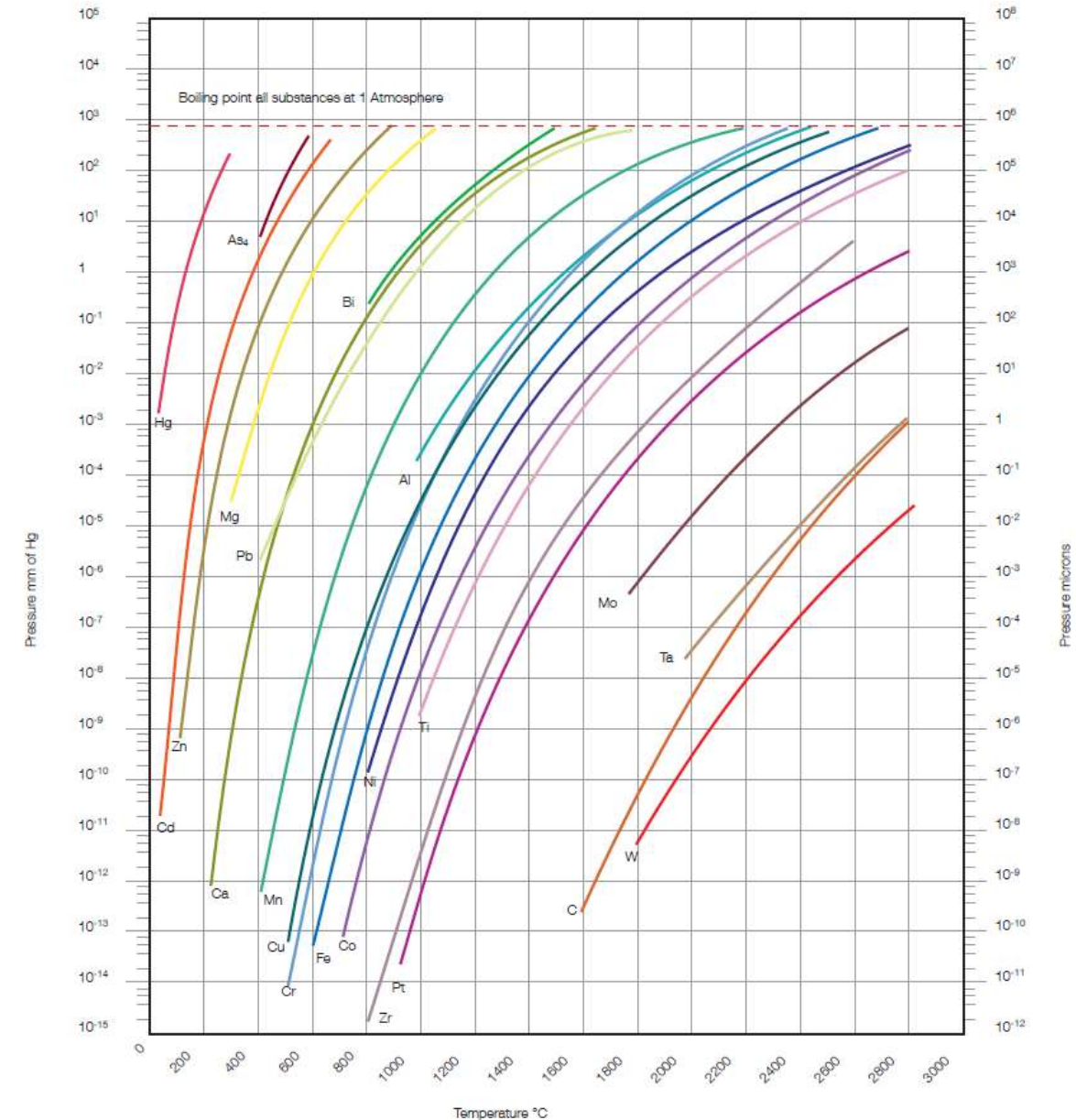
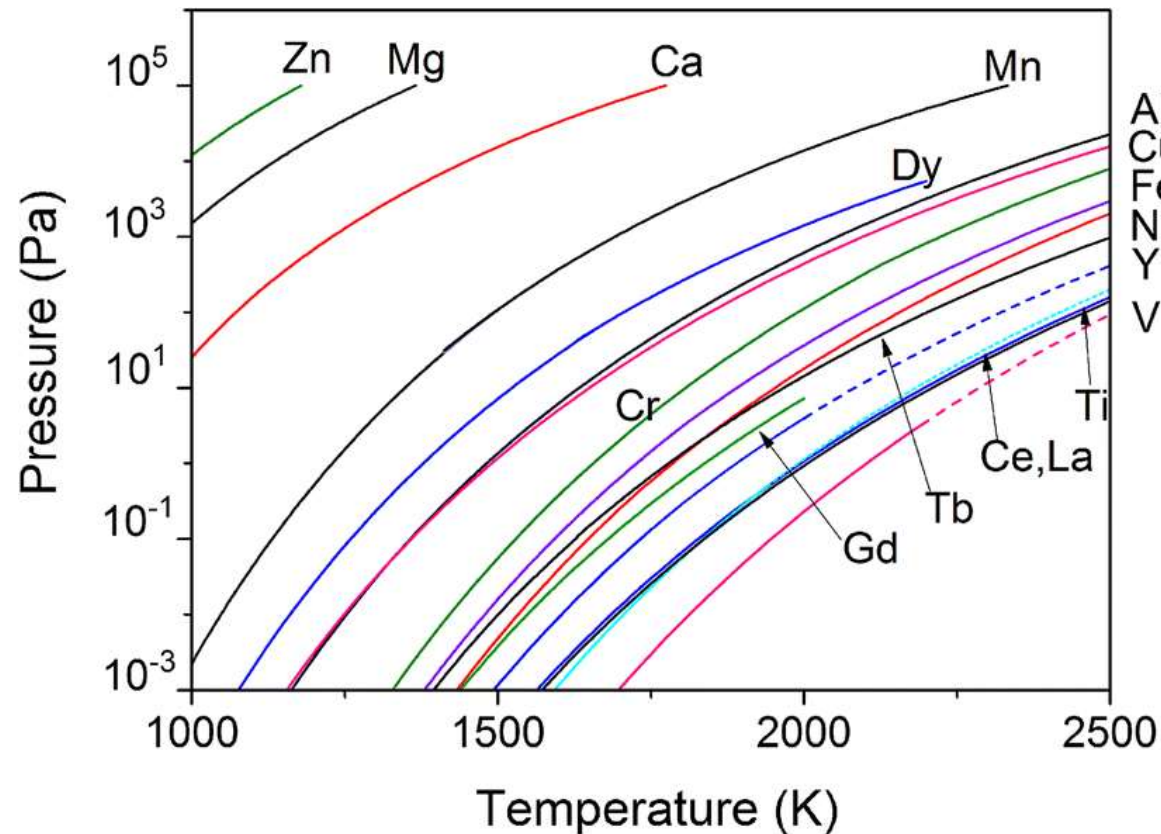
- physics of evaporation cooling: vapor pressure





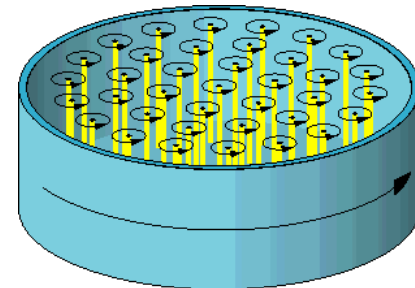
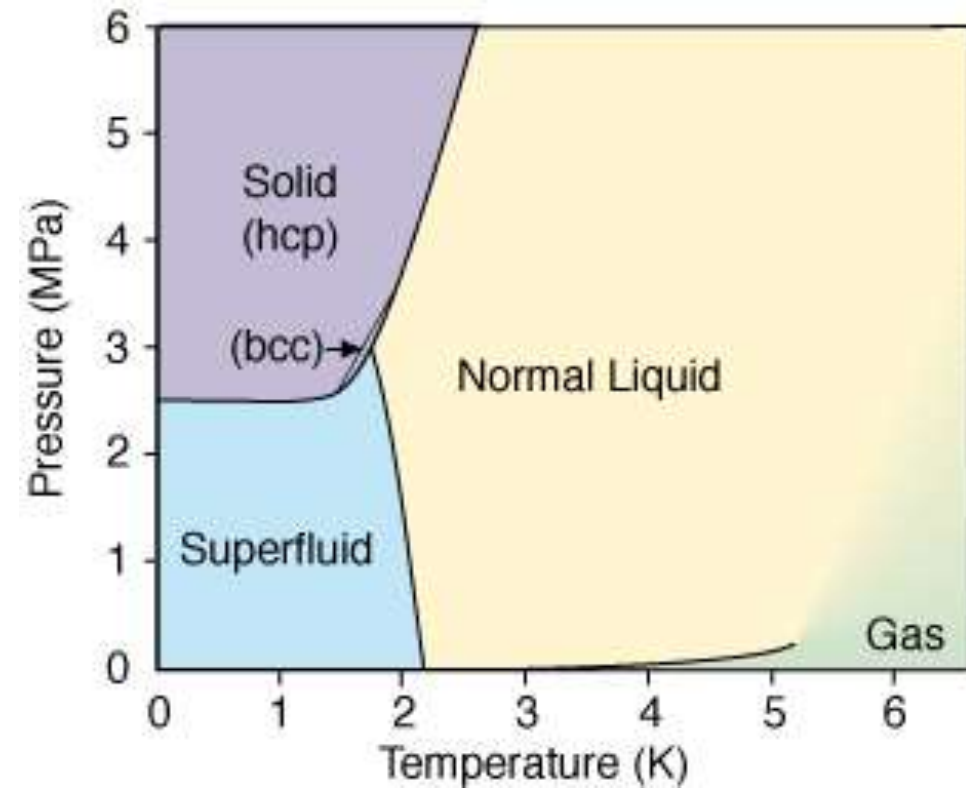
# III.1.6 Evaporation Cooling

- physics of evaporation cooling:  
vapor pressure of some elements



# III.1.6 Evaporation Cooling

- liquid  $^4\text{He}$  (cf. chapter I)
  - boson
  - liquid down to 0 K (@ 1 atm)
  - superfluid  $^4\text{Helium}$  at 2.17 K
    - Bose condensation: macroscopic number of atoms in ground state
    - very low viscosity
    - very high heat conduction
    - strange thermomechanical effects
    - creeping on vertical surfaces
    - vortex core with radius  $0.8 \text{ \AA}$  @ 0.6K
    - explained by a two-fluid model
  - density  $125 \text{ kg/m}^3$



# III.1.6 Evaporation Cooling

- liquid helium cryostats
  - LHe has small latent heat
    - ➔ good thermal insulation by vacuum
    - ➔ LHe container of poor thermal conductivity, glass or stainless steel
    - ➔ thermal radiation shield at liquid Nitrogen temperature to reduce black-body radiation
  - bath cryostat      - sample is immersed in the LHe
  - gas flow cryostat      - sample is located in cold He gas

# III.1.6 Evaporation Cooling

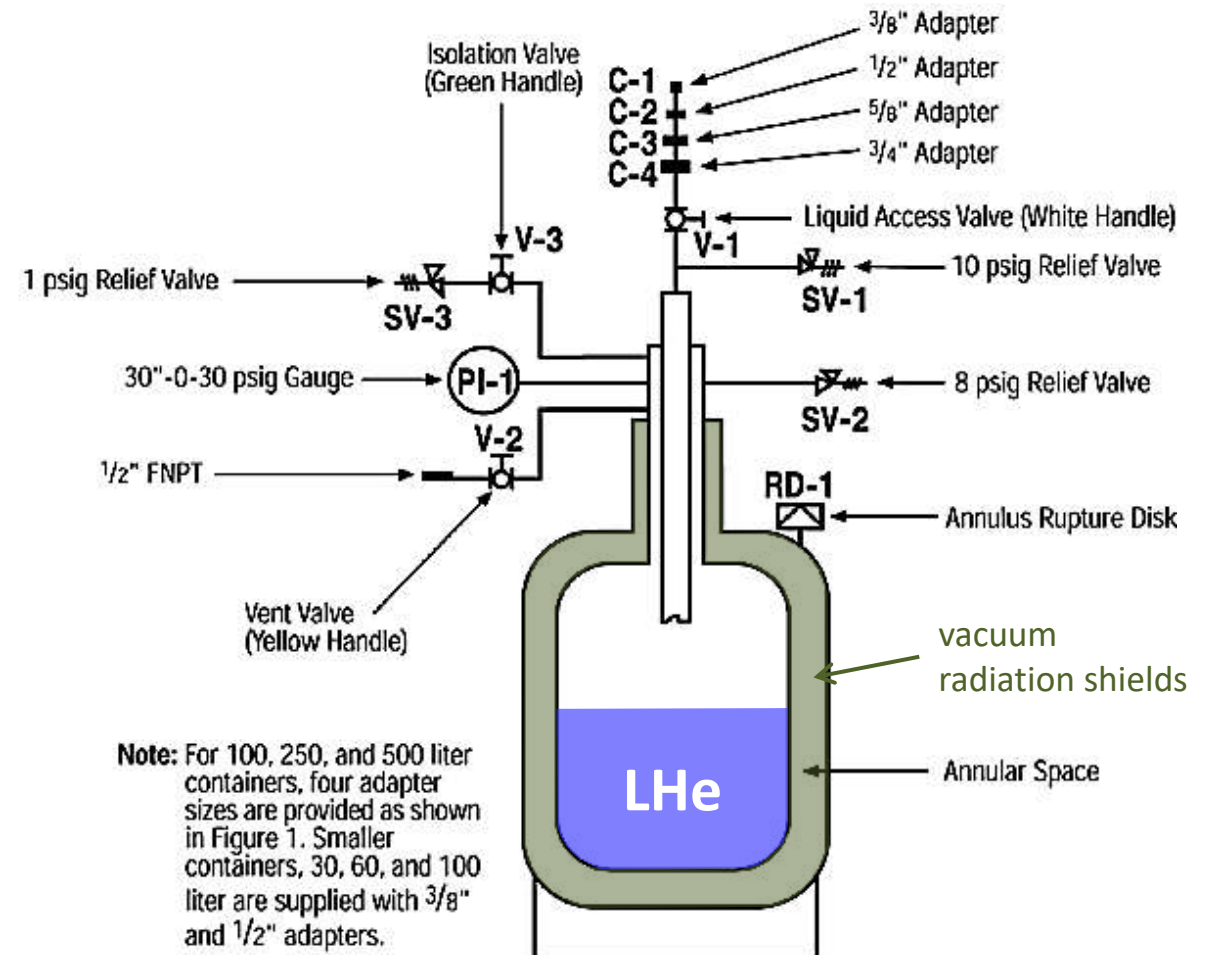
- liquid helium container

narrow neck to minimize

- heating by radiation
- heating by thermal conduction

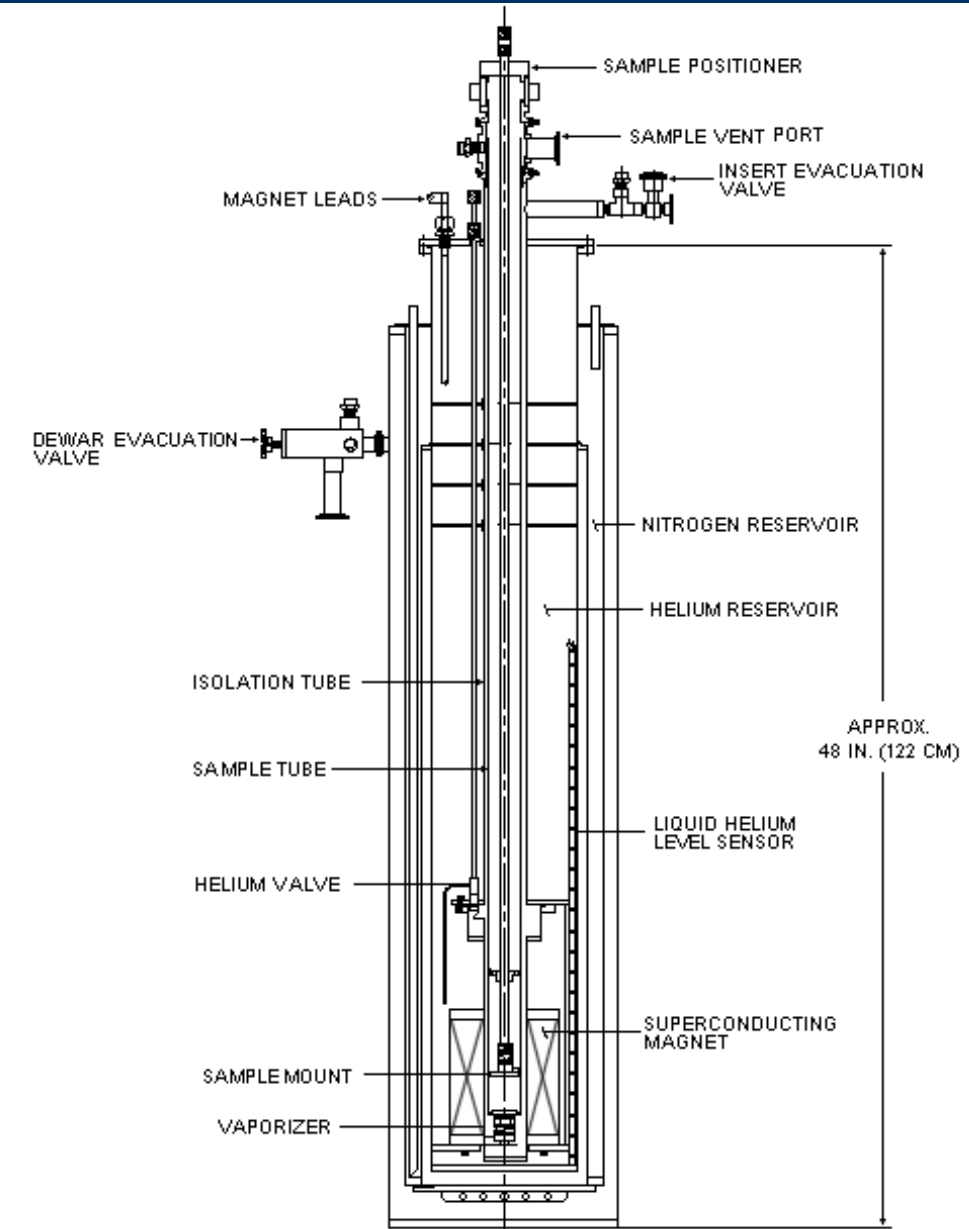
typical losses

- 1 liter of LHe / day



# III.1.6 Evaporation Cooling

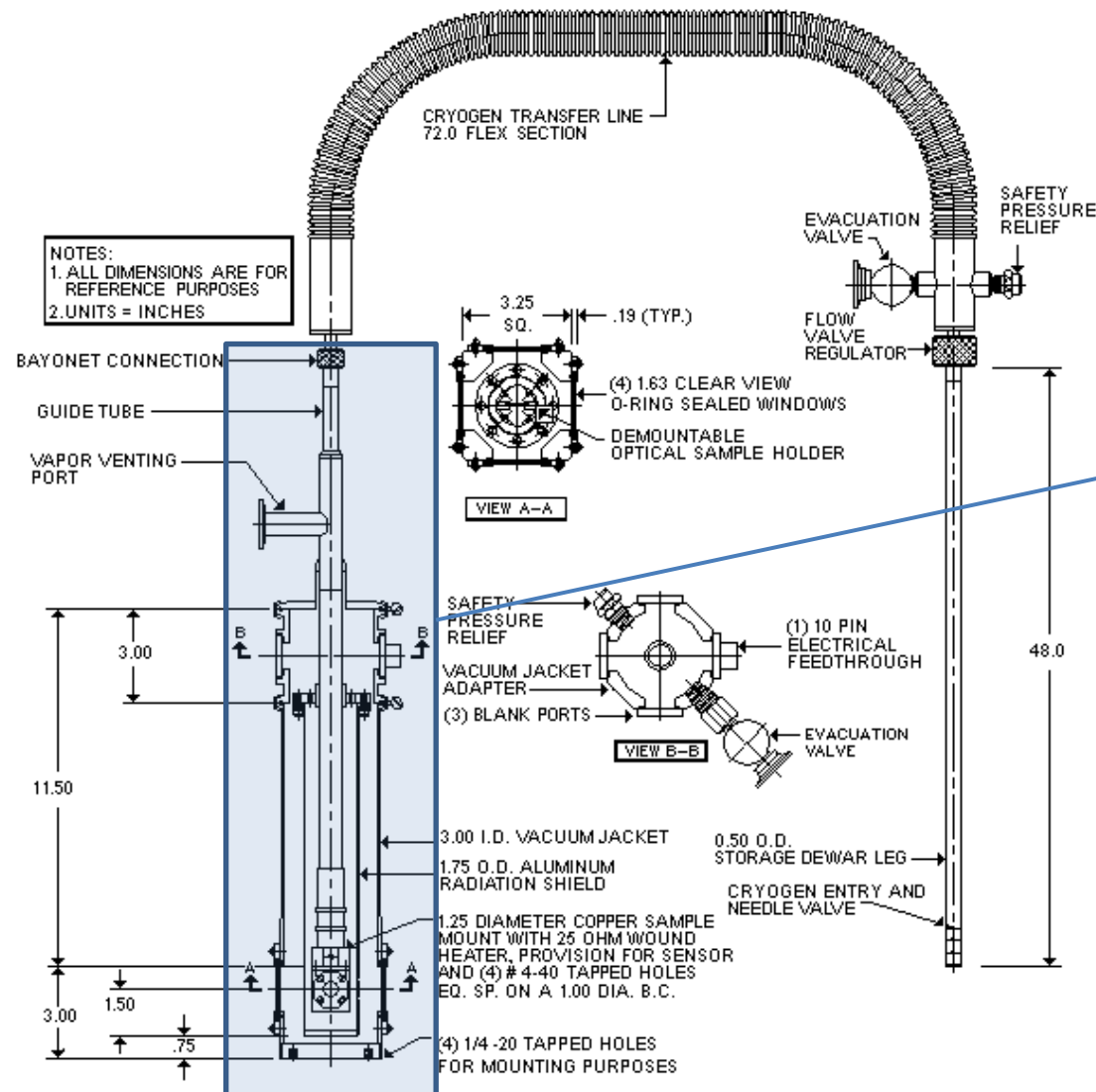
- liquid helium bath cryostat





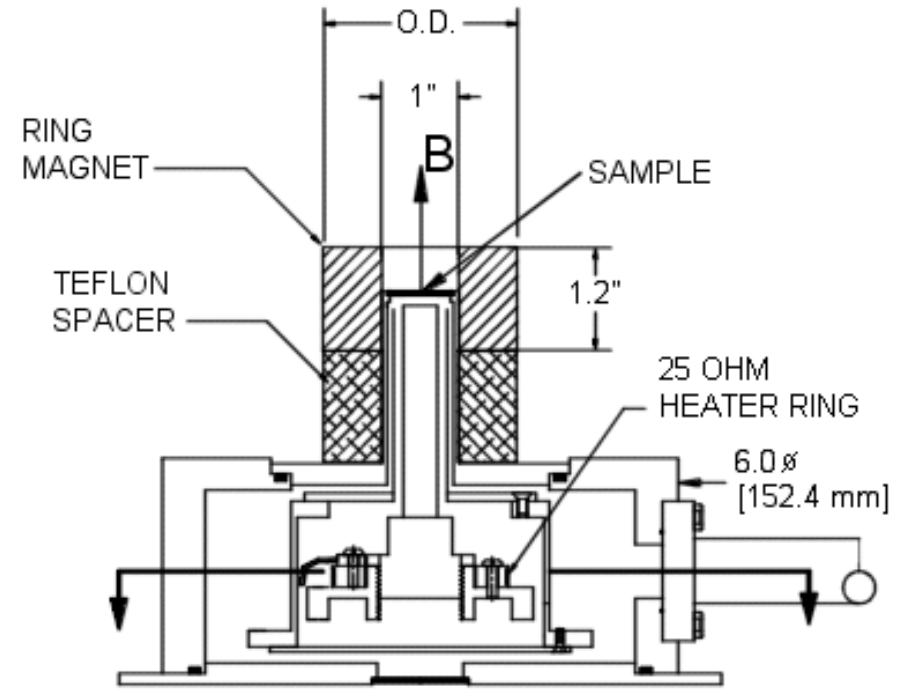
# III.1.6 Evaporation Cooling

- liquid helium gas flow cryostat (1)



# III.1.6 Evaporation Cooling

- liquid helium gas flow cryostat (2)



Possible Permanent Magnet Layout with  
ST-500 Microscopy Cryostat

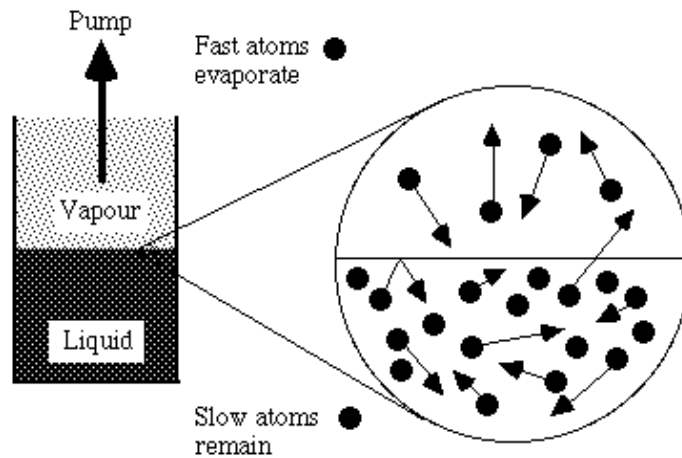
- no liquid Nitrogen required
- radiation shield cooled by cold helium return gas



# III.1.6 Evaporation Cooling

- liquid helium temperatures below 4.2 K

- reduce the vapour pressure over bath of  $^4\text{He}$
- temperature down to 1.2 K at pumping power of  $10 \text{ m}^3/\text{h}$



cooling power:

$$\dot{Q} = \dot{n}_{\text{gas}} \Delta H_{\text{vap}} \propto p \propto \exp\left(-\frac{\Delta H_{\text{vap}}}{RT}\right)$$

rate of atoms going to gas phase

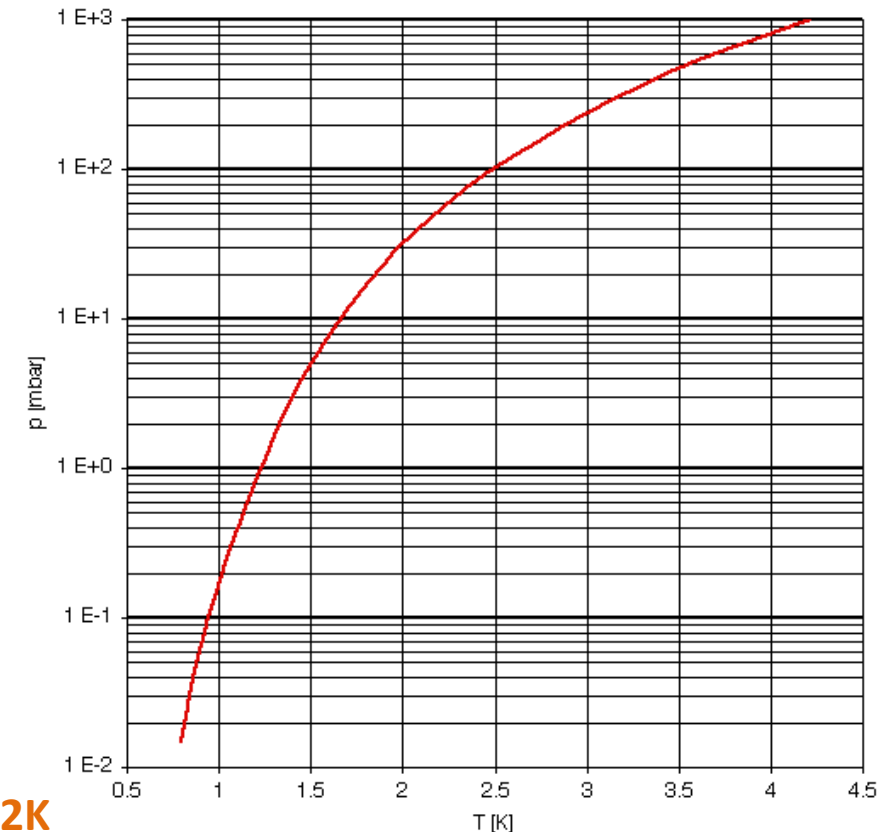
→ up to 10 mW cooling power @ 1.2K

Clausius-Clapeyron equation:

$$\frac{dp}{dT} = \frac{\Delta H_{\text{vap}}}{(V_{\text{gas}} - V_{\text{liquid}})T} \simeq \frac{\Delta H_{\text{vap}}}{V_{\text{gas}}T} \Rightarrow T_B(p) = \left(\frac{1}{T_0} - \frac{R \ln(p/p_0)}{\Delta H_{\text{vap}}}\right)^{-1}$$

$$\ln \frac{p}{p_0} = \frac{\Delta H_{\text{vap}}}{R} \left(\frac{1}{T_0} - \frac{1}{T}\right)$$

$\Delta H_{\text{vap}}$ : molar latent heat [J/mole]  
 $\simeq N_A \cdot E_{\text{binding}} \simeq 90 \text{ J/mole}$  for  $^4\text{He}$



# III.1.6 Evaporation Cooling

- liquid  $^3\text{He}$  (cf. chapter I)
  - fermion
  - superfluid at 2.5mK
    - formation of weakly bound fermions: Cooper pairs
  - density 59 kg/m<sup>3</sup>
  - higher vapour pressure than  $^4\text{He}$  due to smaller latent heat:
    - $\Delta H_{\text{vap}} = 40 \text{ J/mole} \rightarrow \text{cooling power} \approx 80\text{mW @ } 1.2\text{K and } 10 \text{ m}^3/\text{h}$
    - pumping speed
  - 0.3 K by pumping  $^3\text{He}$  vapour
    - some cm<sup>3</sup>
    - 0.1 mW cooling power @ 0.3K
  - $^3\text{He}$  obtained by nuclear reactions
    - extremely expensive
    - 1 liter of  $^3\text{He}$  gas costs more than US \$5.000 (2022)

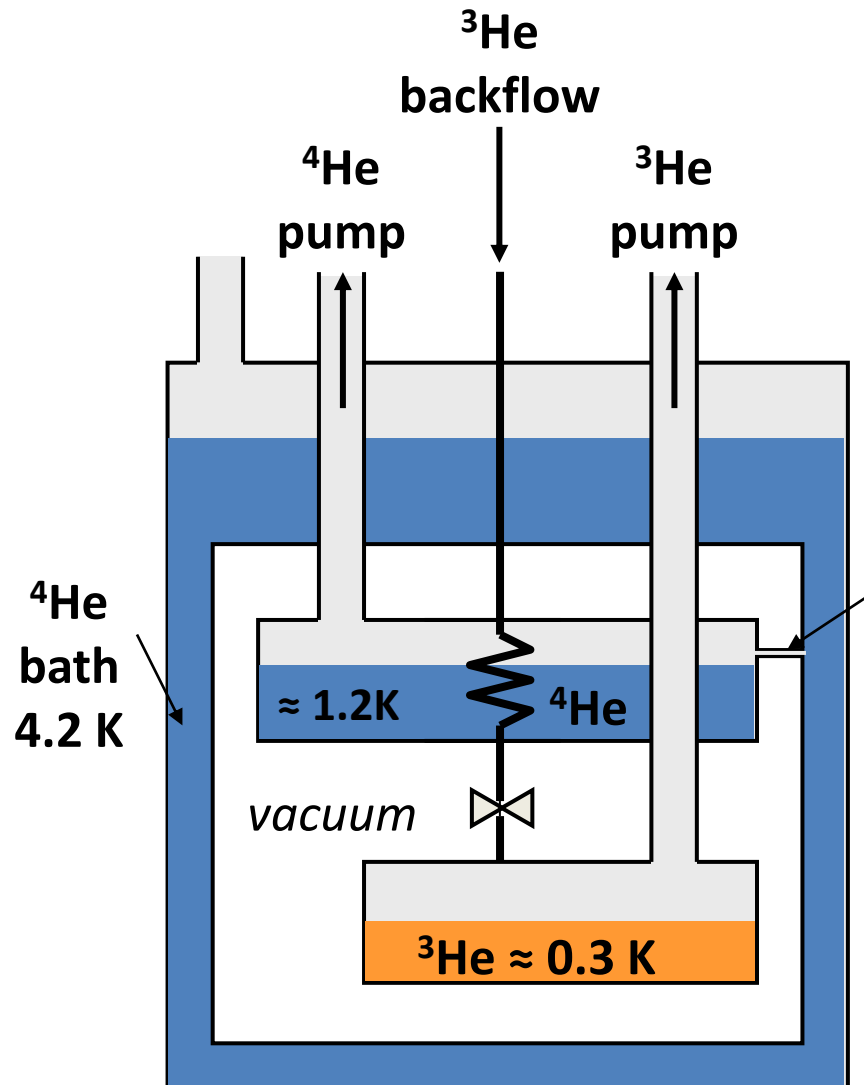
# III.1.6 Evaporation Cooling

- liquid  $^3\text{He}$  cryostat

latent heat of  $^3\text{He}$ :  $\Delta H_{\text{vap}} = 40 \text{ J/mole}$  as compared to  $90 \text{ J/mole}$  for  $^4\text{He}$   
 $\rightarrow$  larger cooling power

$$\dot{Q} = \dot{n}_{\text{gas}} \Delta H_{\text{vap}} \propto p \propto \exp\left(-\frac{\Delta H_{\text{vap}}}{RT}\right)$$

$\approx 80 \text{ mW}$  @  $1.2 \text{ K}$  for  $^3\text{He}$  as compared to  
 $\approx 10 \text{ mW}$  @  $1.2 \text{ K}$  for  $^4\text{He}$



$\leftarrow$  condensation of backflowing  $^3\text{He}$  gas

$\leftarrow$  flow restriction for condensed  $^3\text{He}$

*minimum temperature  $\approx 300 \text{ mK}$   
 cooling power  $\approx 0.1 \text{ mW}$*

# III.1.7 $^3\text{He}/^4\text{He}$ Dilution Cooling

- $^3\text{He}/^4\text{He}$  dilution refrigeration (cf chapter I.6)

- operation principle:

- remove  $^3\text{He}$  atoms from the dilute phase below  $T_{\text{tri}} = 0.87 \text{ K}$
- transport of  $^3\text{He}$  atoms across phase boundary to maintain equilibrium concentration
- corresponds to evaporation of  $^3\text{He}$  from concentrated phase
- **cooling effect as the latent heat of evaporation is removed**

- for Fermi liquid:

$$\rightarrow \mathbf{C_{V,concentrated}} < \mathbf{C_{V,diluted}} \quad (x_3 = \mathbf{0.065}) \quad (C_V \propto T/T_F, \quad T_F \propto n_{^3\text{He}}^{2/3})$$

- with  $U = Q = \int_0^T C_V(T') dT' = \frac{\gamma}{2} T^2$  we therefore obtain ( $C_V = \gamma T$ ,  $\gamma$  = Sommerfeld coefficient):

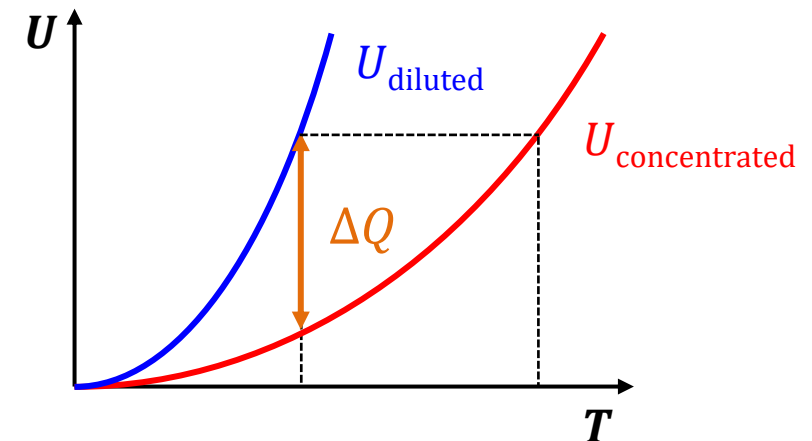
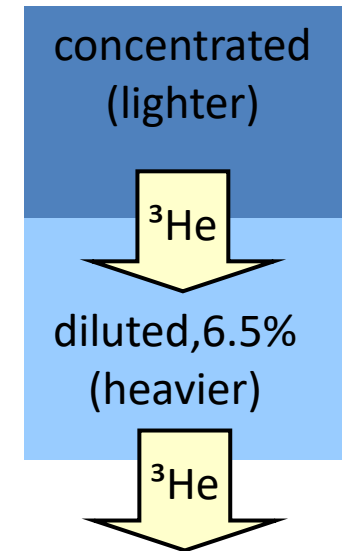
$$\mathbf{U_{concentrated}(T)} < \mathbf{U_{diluted}(T)}$$

- on transition across phase boundary:

$$dG = 0 = dU - TdS \Rightarrow dU = TdS = dQ$$

- removal of heat  $\Delta Q = T\Delta S = T [S_{\text{dil}}(T) - S_{\text{con}}(T)]$
- **cooling effect**

- $^3\text{He}/^4\text{He}$  dilution refrigerator



# III.1.7 $^3\text{He}/^4\text{He}$ Dilution Cooling

- $^3\text{He}/^4\text{He}$  dilution refrigeration: cooling power (cf chapter I.6)

- assumption: *one mole of  $^3\text{He}$  crosses boundary between diluted and concentrated phase*

- removed heat:

$$\Delta Q = T \Delta S = T [S_{\text{dil}}(T) - S_{\text{con}}(T)]$$

- cooling power:  $\Delta \dot{Q} = \left( \frac{\Delta Q}{n_{^3\text{He}}} \right) \cdot \dot{n}_{^3\text{He}} \Rightarrow$

$$\Delta \dot{Q} = \dot{n}_{^3\text{He}} T [s_{\text{dil}}(T) - s_{\text{con}}(T)]$$

$$S_{\text{dil}} = \frac{S_{\text{dil}}}{n_{^3\text{He}}}, S_{\text{con}} = \frac{S_{\text{con}}}{n_{^3\text{He}}}$$

- since there is no volume change:

$$dQ = dU = T dS = C_V dT, \quad U(T) = \int_0^T C_V dT', \quad S(T) = \int_0^T \frac{C_V}{T'} dT'$$

➤ with  $C_{\text{mol}} = \frac{\pi^2 N_A k_B^2 T}{2 \varepsilon_F}$  and  $\varepsilon_F = \frac{\hbar^2}{2m_{^3\text{He}}^*} \left( \frac{3\pi^2 N_{^3\text{He}}}{V} \right)^{2/3} = \frac{\hbar^2}{2m_{^3\text{He}}^*} \left( \frac{3\pi^2 N_A}{V_{\text{mol}}} \right)^{2/3}$  (standard expressions for Fermi liquid)

we obtain the entropy

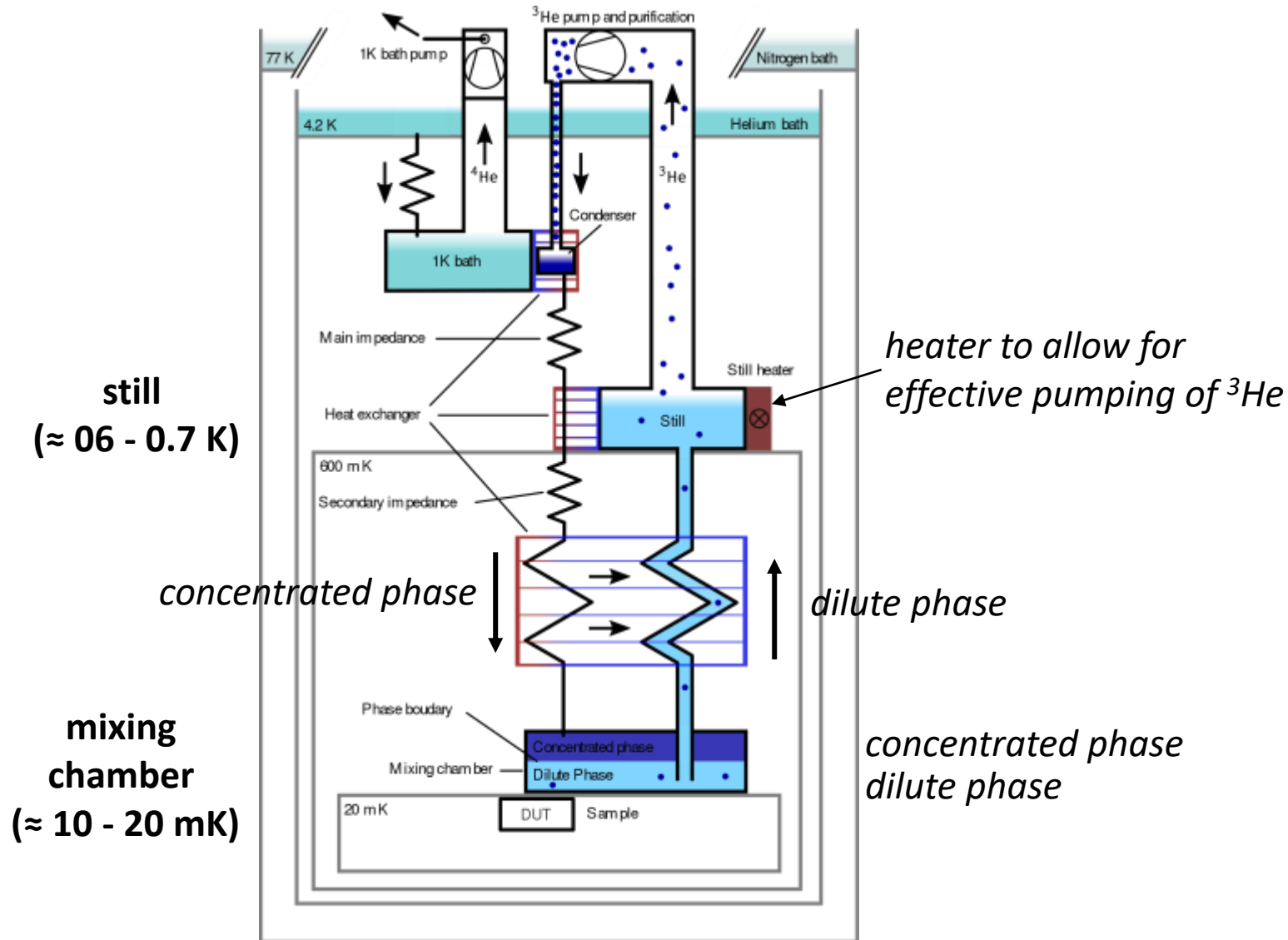
$$S_{\text{mol}} = \frac{\pi^2 N_A k_B^2 m_{^3\text{He}}^*}{\hbar^2} \left( \frac{V}{3\pi^2 N_{^3\text{He}}} \right)^{2/3} T \propto \frac{T}{n_{^3\text{He}}}$$

$$\Rightarrow S_{\text{con}} < S_{\text{dil}} \quad (\text{as } n_{^3\text{He,con}} > n_{^3\text{He,dil}})$$

$$\Rightarrow \dot{Q}(T) = 84 \dot{n}_{^3\text{He}} T^2 [\text{W}] \quad (\text{cooling power} \propto ^3\text{He throughput } \dot{n}_3 [\text{mol/s}])$$

# III.1.7 $^3\text{He}/^4\text{He}$ Dilution Cooling

- $^3\text{He}/^4\text{He}$  dilution refrigeration: large cooling power  $\dot{Q}(T) = 84 \dot{n}_{^3\text{He}} T^2 [\text{W}]$  requires large  $^3\text{He}$  throughput



*pumping of  $^3\text{He}$  generates osmotic pressure*

*$\rightarrow ^3\text{He}$  flows from mixing chamber to still*

*$\rightarrow$  only possible if  $^3\text{He}$  atoms cross phase boundary*

*$\rightarrow$  cooling*

**minimum temperature  $\approx 1.5 \text{ mK}$**

# III.1.7 $^3\text{He}/^4\text{He}$ Dilution Cooling

- $^3\text{He}/^4\text{He}$  dilution refrigeration

- example:
  - desired cooling power:  $10^{-5} \text{ W}$
  - still temperature:  $0.7 \text{ K}$
  - mixing chamber temperature:  $10 \text{ mK}$

- what is the required pumping speed ??

$$\dot{n}_3 = \frac{10^{-5}}{84 (10^{-2})^2} = 0.0012 \text{ mole / s}$$

$$\dot{Q} = 84 \dot{n}_3 T^2$$

we assume that  $^3\text{He}$  is an ideal gas ( $R = 8.31 \text{ J / mole K}$ )

$$\dot{V} = \dot{n}_3 RT / p$$

$$R = 8.31 \text{ J/mole K}$$

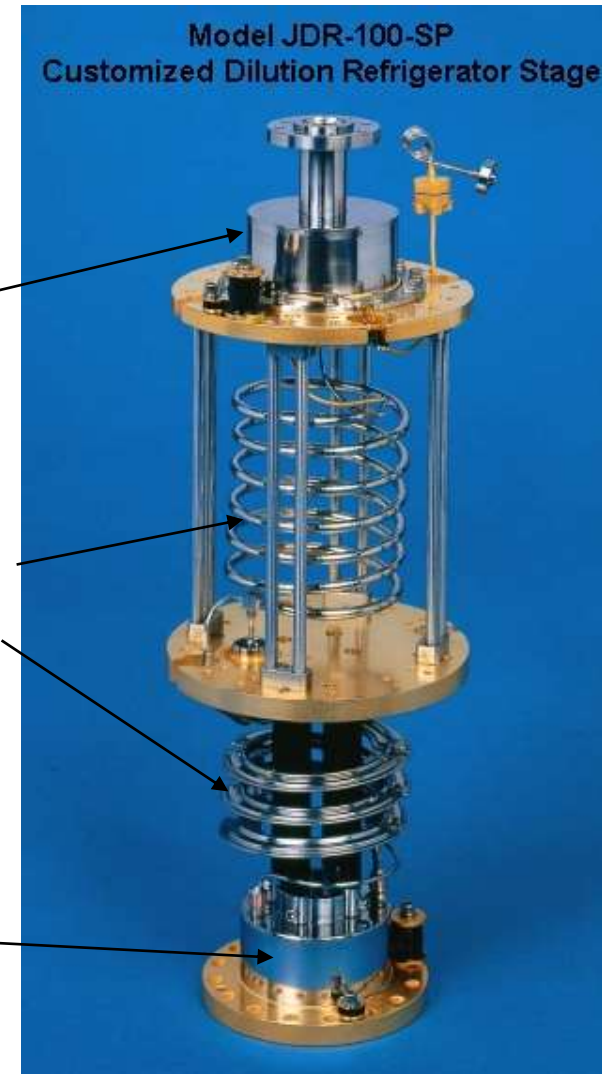
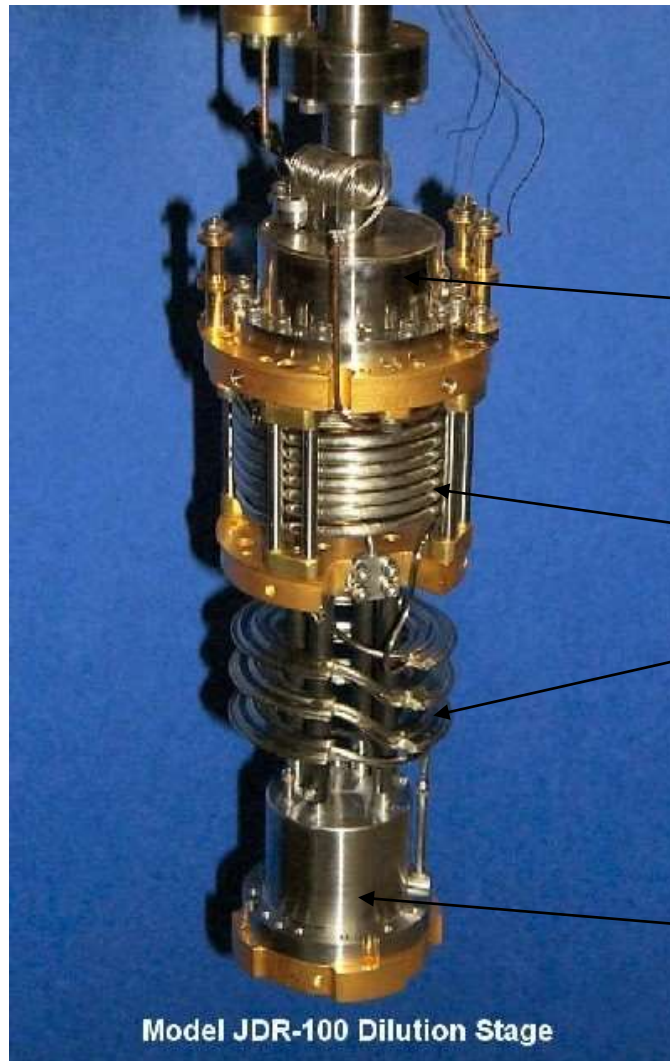
- numbers:
  - vapor pressure of  $^3\text{He}$  at  $0.7 \text{ K}$ :  $0.0828 \text{ mbar} = 8.28 \text{ Pa}$
  - @  $300 \text{ K}$  we obtain:

$$\dot{V} = 0.0012 \cdot 8.31 \cdot 300 / 8.28 = 0.363 \text{ m}^3/\text{s} \approx 360 \text{ l/s}$$

→ **large  $^3\text{He}$  pump is required**



# III.1.7 $^3\text{He}/^4\text{He}$ Dilution Cooling

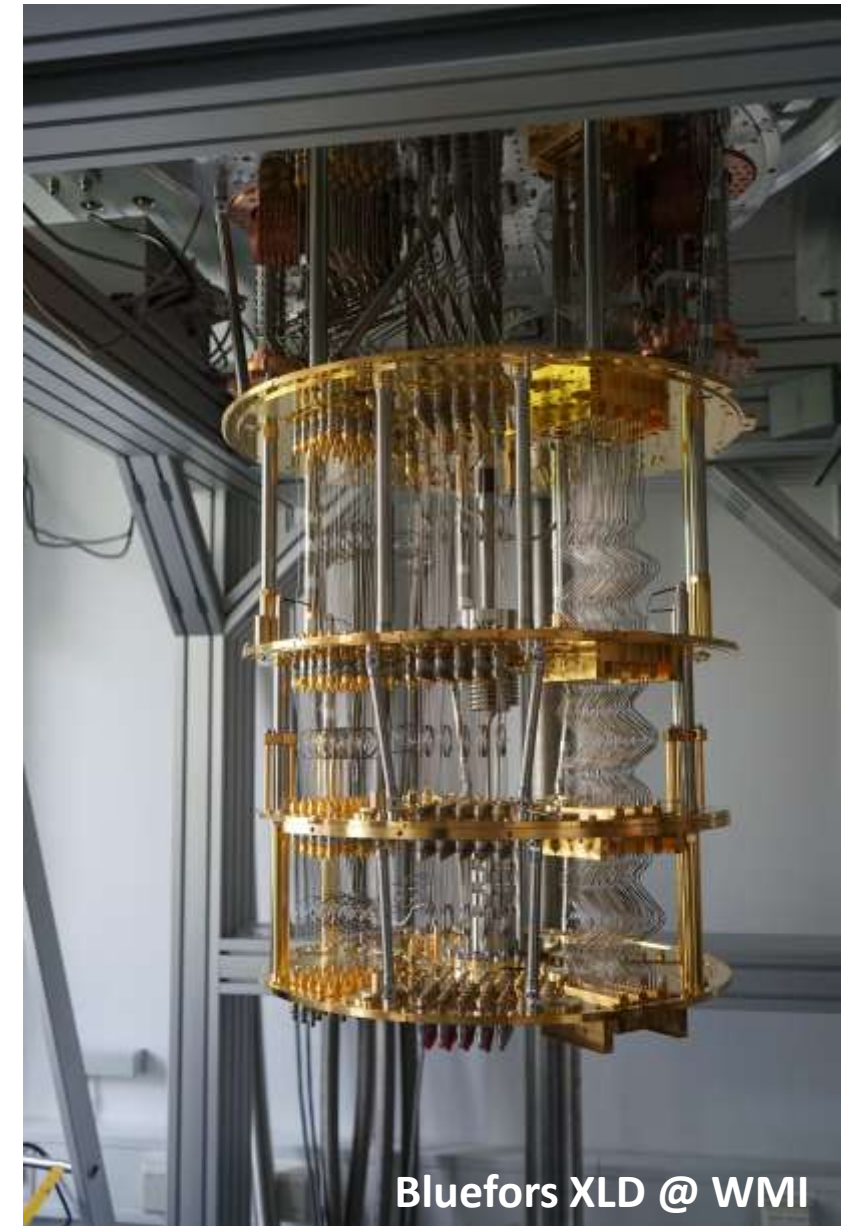
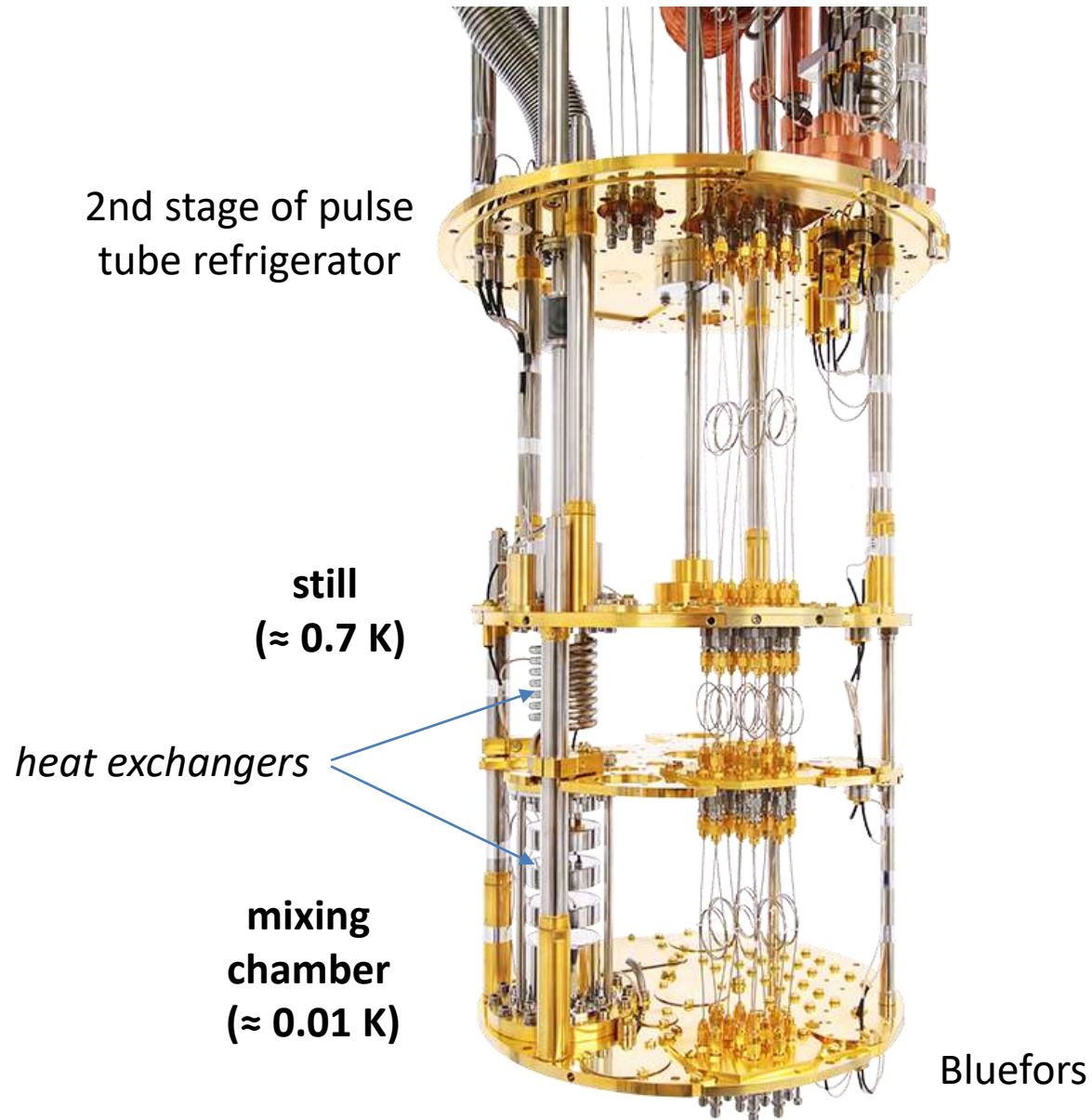


still

heat  
exchangers

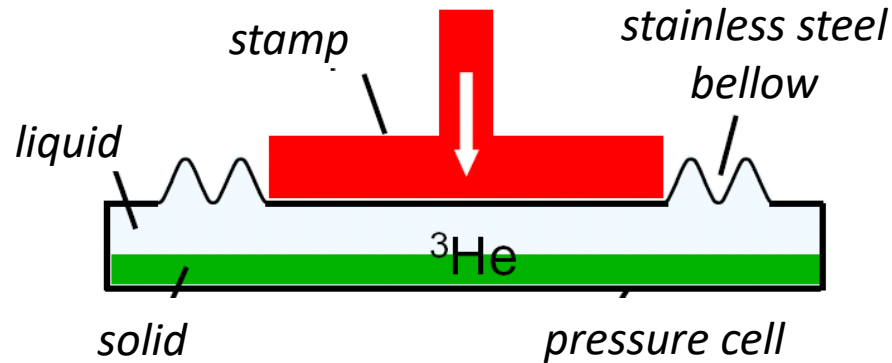
mixing  
chamber

# III.1.7 $^3\text{He}/^4\text{He}$ Dilution Cooling

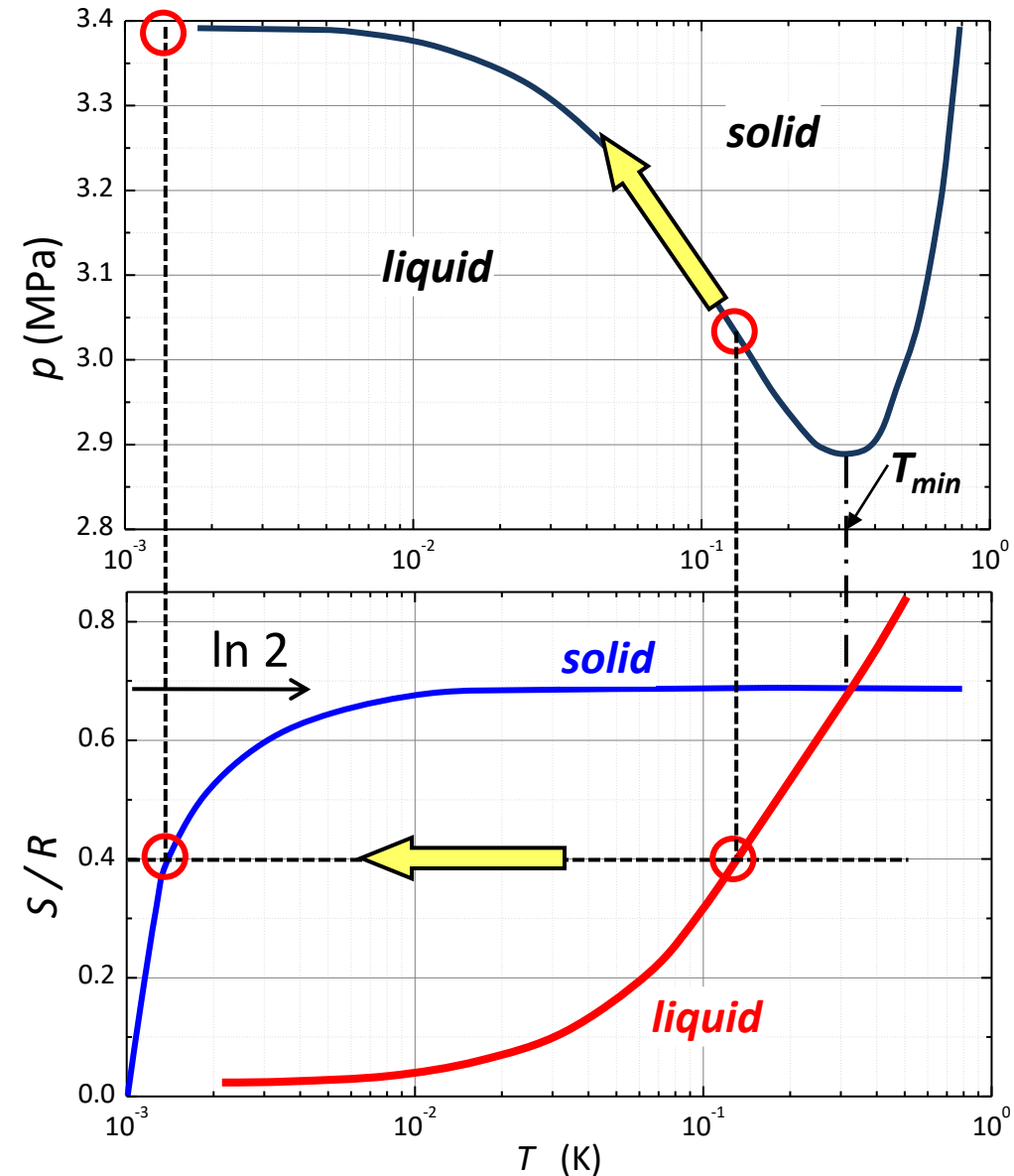


# III.1.8 Pomeranchuk Cooling

- Pomeranchuk cooling (cf. chapter I.5.2)



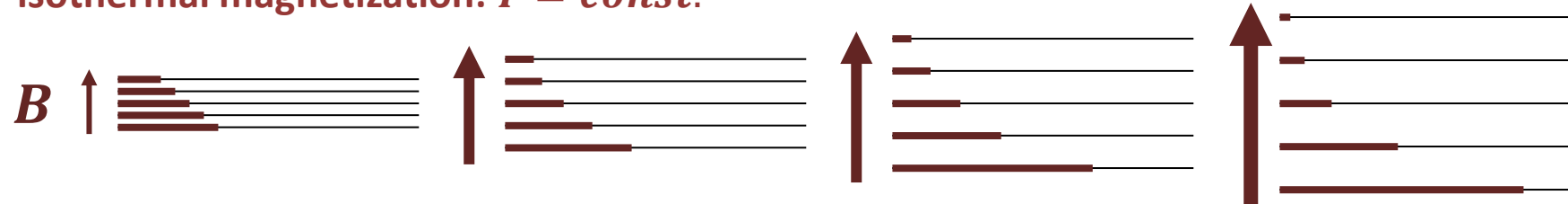
- precooling to  $T < T_{\min}$
- adiabatic compression  
→ *solidification and cooling*
- lowest  $T$ :  $\approx 1.5$  mK
- *limitation due to antiparallel spin ordering in solid  $^3\text{He}$*



# III.1.9 Adiabatic Demagnetization Cooling

- principle: combination of isothermal magnetization and adiabatic demagnetization

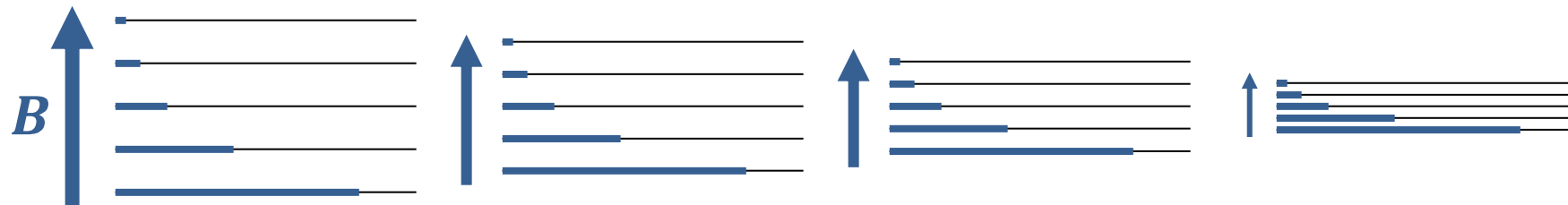
- isothermal magnetization:  $T = \text{const.}$



$$\frac{\Delta Q}{\Delta S} = T = \text{const}$$

- switch on  $B_{\text{ext}}$  at  $T = \text{const.}$ , magnetic work is done, generated heat  $\Delta Q$  is dumped into heat sink
- reduction of entropy if  $T = \text{const.}$

- isenthalpic or adiabatic demagnetization:  $S = \text{const.}$



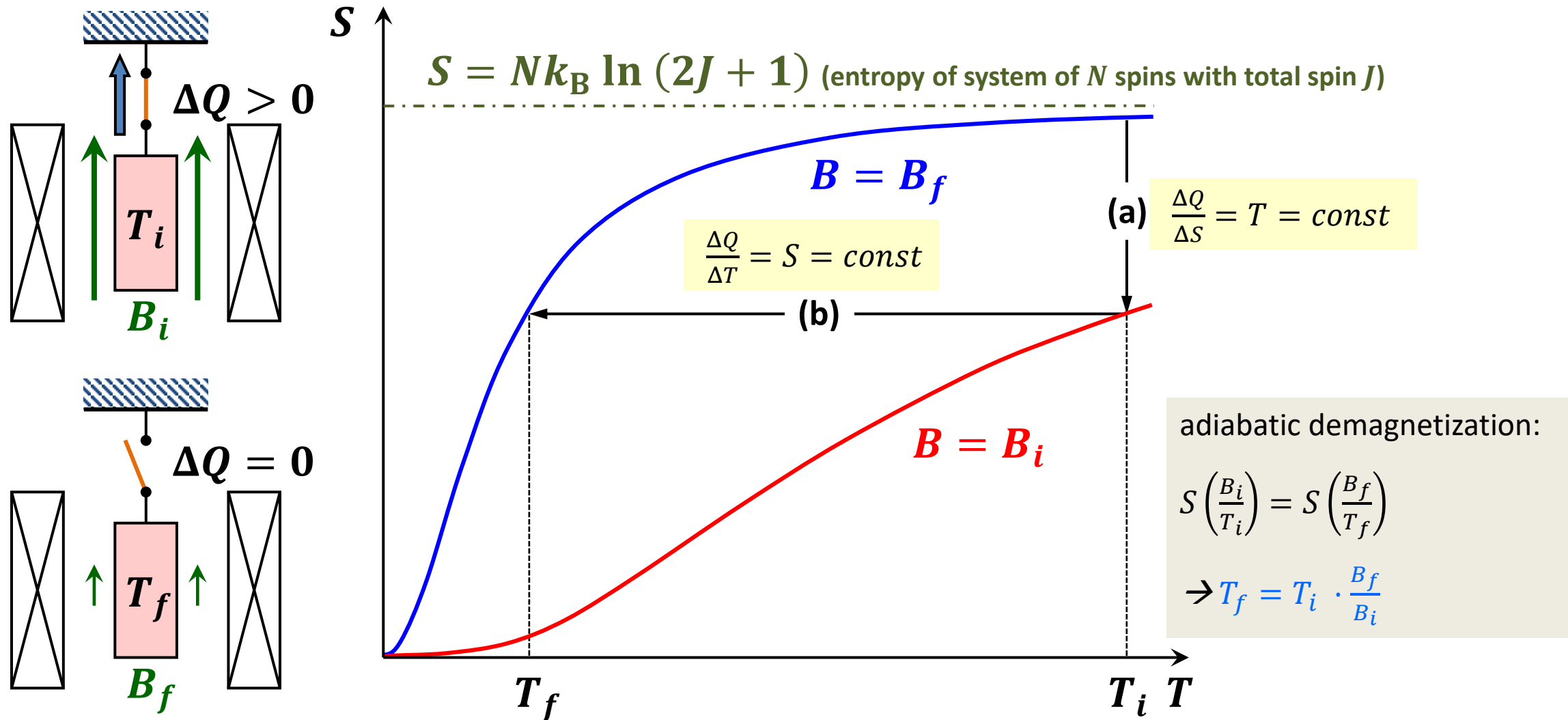
$$\frac{\Delta Q}{\Delta T} = S = \text{const}$$

- switching off  $B_{\text{ext}}$  is increasing entropy  $S$  of the magnetic system
- to keep  $S = \text{const.}$ ,  $T$  has to be reduced and, as  $\frac{\Delta Q}{\Delta T} = S = \text{const.}$ , heat  $\Delta Q$  is removed from spin system



# III.1.9 Adiabatic Demagnetization Cooling

- adiabatic demagnetization



# III.1.9 Adiabatic Demagnetization Cooling

- thermodynamics of adiabatic demagnetization

- which amount of heat  $\Delta Q_{\text{spin}}$  can be absorbed by the spin systems ?

$$\Delta Q_{\text{spin}}(B = 0) = \int_{T_f}^{T_i} C_{\text{spin}} dT = \int_{T_f}^{T_i} T \left( \frac{\partial S_{\text{spin}}}{\partial T} \right)_B dT \quad (\text{cooling capacity})$$

- entropy of spin system with spin quantum number  $J$  for  $g\mu_B B \ll k_B T$ :

$$S = Nk_B \left\{ \ln(2J + 1) - \frac{g^2 J(J + 1) \mu_B^2}{6k_B^2} \frac{B^2 + B_{\text{int}}^2}{T^2} \right\} \quad \text{finite } B_{\text{int}} \text{ due to finite spin-spin interaction}$$

- final temperature

$$T_f = T_i \sqrt{\frac{B_f^2 + B_{\text{int}}^2}{B_i^2 + B_{\text{int}}^2}}$$

remaining internal field due to finite magnetic interactions  
(should be as small as possible)

# III.1.9 Adiabatic Demagnetization Cooling

- adiabatic demagnetization

## paramagnetic salts:

e.g MAS =  $\text{MnSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$

- cooling of electron spins
- material with large entropy  $S/R$  but large  $B_{\text{int}}$
- lowest temperatures  $T_f \approx 100 \text{ mK}$
- large cooling capacity

e.g CMN =  $2\text{Ce}(\text{NO}_3)_3 \cdot 2\text{Mg}(\text{NO}_3)_2 \cdot 24\text{H}_2\text{O}$

- cooling of electron spins
- material with small entropy  $S/R$  but small  $B_{\text{int}}$
- lowest temperatures  $T_f \approx 2 \text{ mK}$
- small cooling capacity

## nuclear demagnetization:

e.g  $^{63}\text{Cu}$  ( $L = \frac{3}{2}$ ) or  $^{65}\text{Cu}$  ( $L = \frac{3}{2}$ )

( $B_{\text{int}} \approx 0.3 \text{ mT}$ ,  $T_i \approx 10 \text{ mK}$ ,  $B_i \approx 3 \text{ T}$ )

- cooling of nuclear spins
- $T_f (B_f = 0) \approx 1 \mu\text{K}$
- problem: transfer of spin temperature to lattice

**long spin-lattice relaxation time**

other materials:  $^{141}\text{PrNi}_5$  ( $L = \frac{5}{2}$ ),  $^{195}\text{Pt}$  ( $L = \frac{1}{2}$ )



# III.1.9 Adiabatic Demagnetization Cooling



Cryogen-free Two Stage Adiabatic Demagnetization Refrigerator from Janis

A cryogen-free two stage adiabatic demagnetization refrigerator using a 4 K pulse tube cryocooler. Gallium Gadolinium Garnet (GGG) and Ferric Ammonium Alum (FAA) paramagnetic pills were used for the first and second stage of the ADR, with Kevlar string supports for each stage. The FAA stage reaches a base temperature below 50 mK, and remains below 100 mK for more than two days.

# III.1.9 Adiabatic Demagnetization Cooling



TEMPERATURE RANGE  
100 mK - 300 K



SAMPLE COOLDOWN TIME  
< 3 hours



CONTINUOUS OPERATION ABOVE  
300 mK



## Key Benefits

Built for an improved sub-Kelvin experience

### Fast sample characterization ^

The L-Type Rapid features kiutra's proprietary puck-based top-loading sample exchange mechanism. This option allows the user to prepare their sample on a sample puck and load it into the cryostat in just a couple of minutes – there is no need to warm up the cryostat. In this way, a sample can be installed and cooled to the base temperature in less than three hours. After completion of the low-temperature tests, the sample can be removed just as quickly, and within a few minutes the next device, prepared on a spare puck, can be loaded. Thus, the L-Type Rapid enables the highest sample turnover for sub-Kelvin studies.

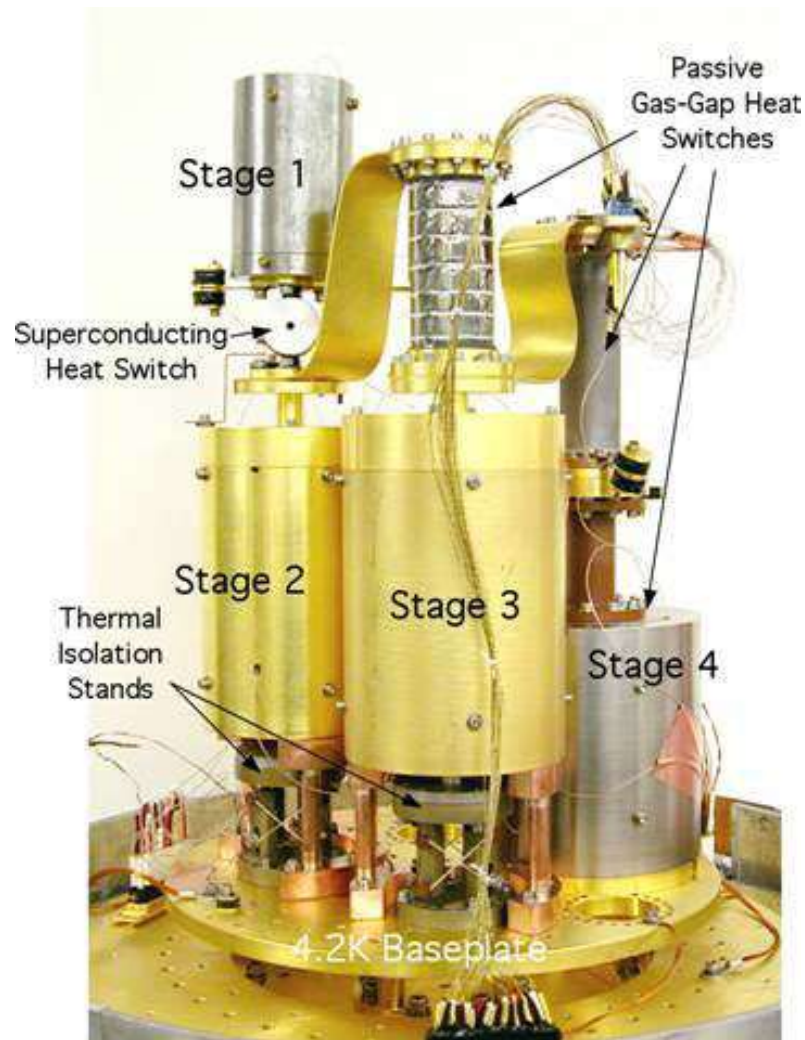
### Wide temperature range v

### Highly modular cooling platform v



kiutra GmbH  
Flößergasse 2  
81369 Munich, Germany

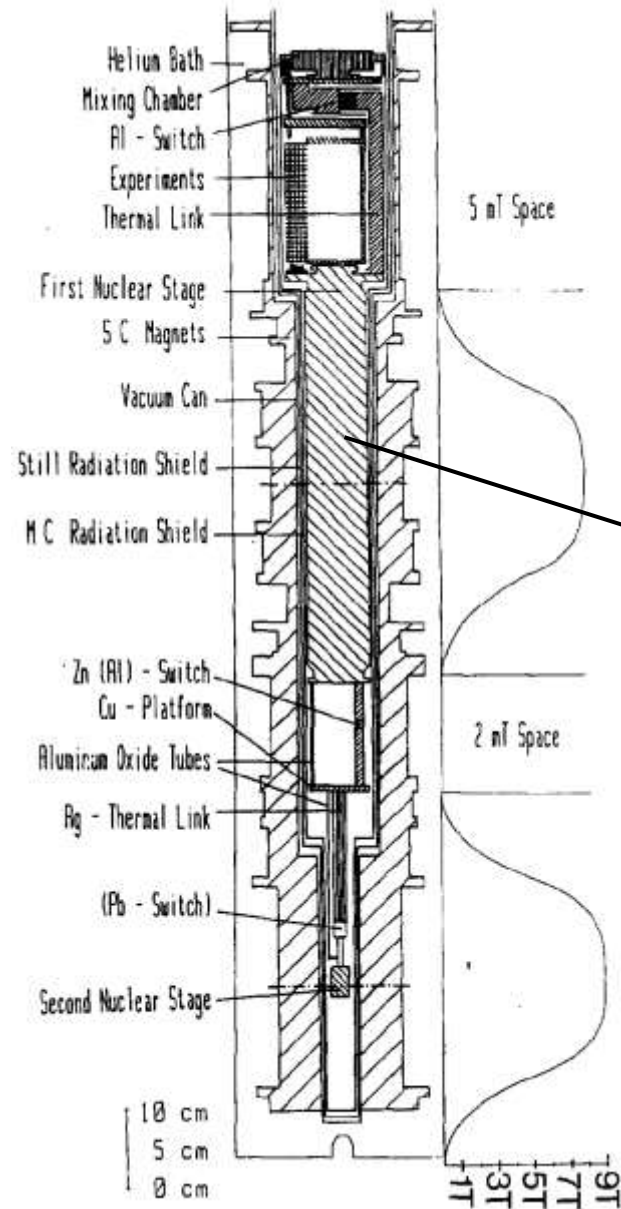
# III.1.9 Adiabatic Demagnetization Cooling



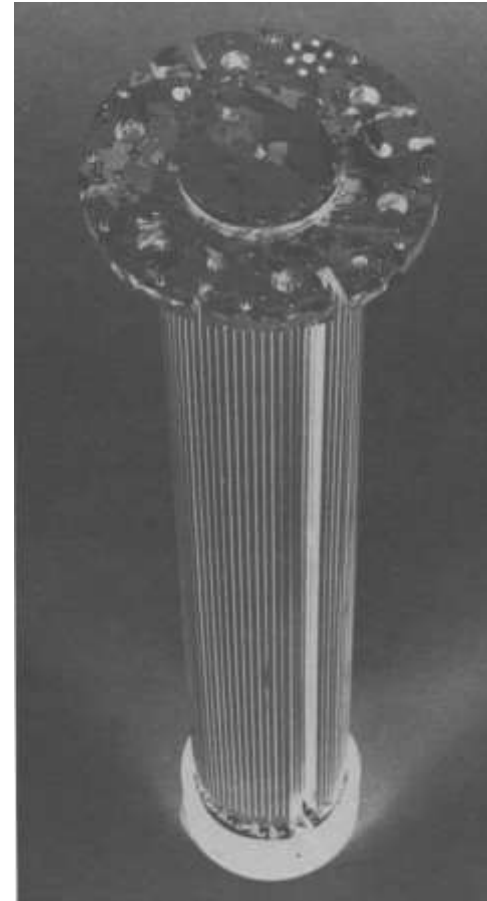
Continuous Adiabatic Demagnetization Refrigerator (CADR) under development at NASA's Goddard Space Flight Center

- CADR to cool from below 5K to  $\approx 35$  mK
- advantage: no stored cryogenics
  - maximizing the lifetime/mass ratio for the instrument

# III.1.9 Adiabatic Demagnetization Cooling

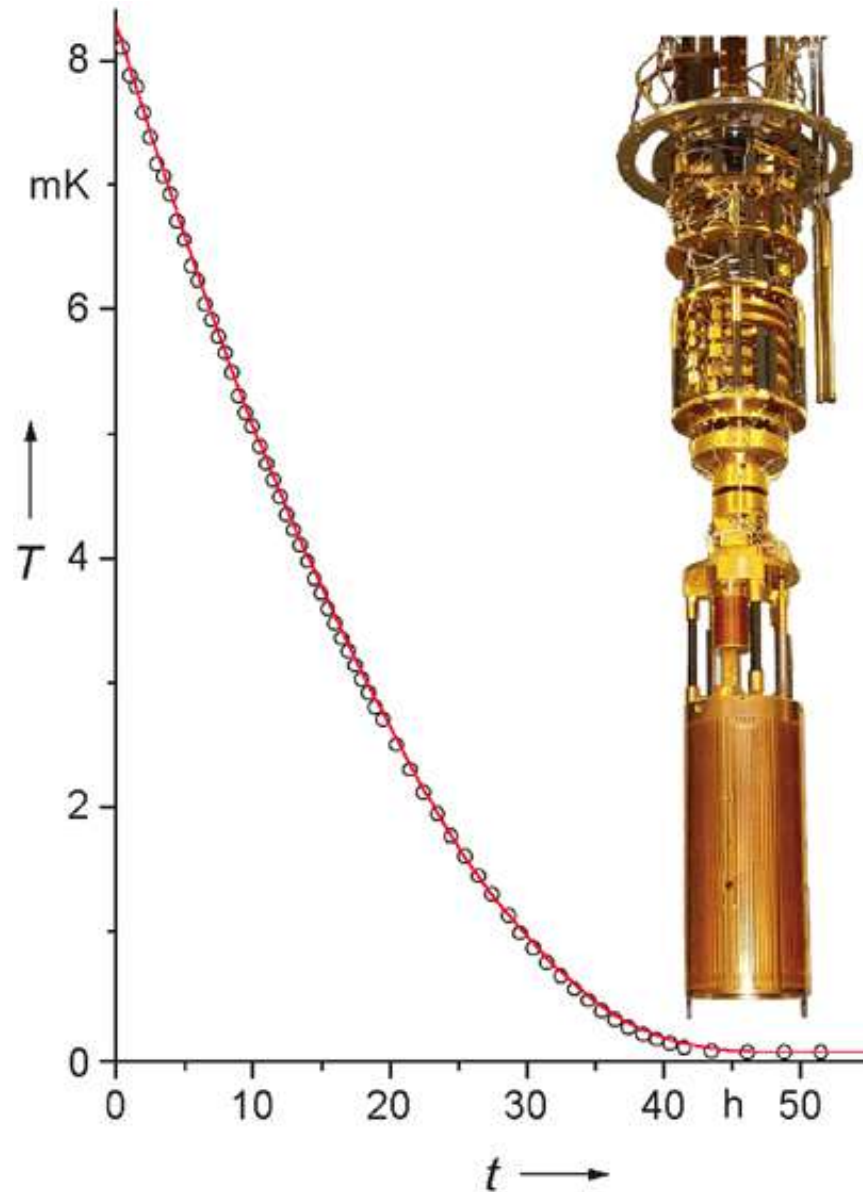


**Cu demagnetization stage**  
(length: 525 mm, diameter: 78 mm)



*R. Gloss et al.,  
J. Low Temp. Phys. 73, 101 (1988)*

# III.1.9 Adiabatic Demagnetization Cooling



## $\mu$ K facility of PTB Berlin:

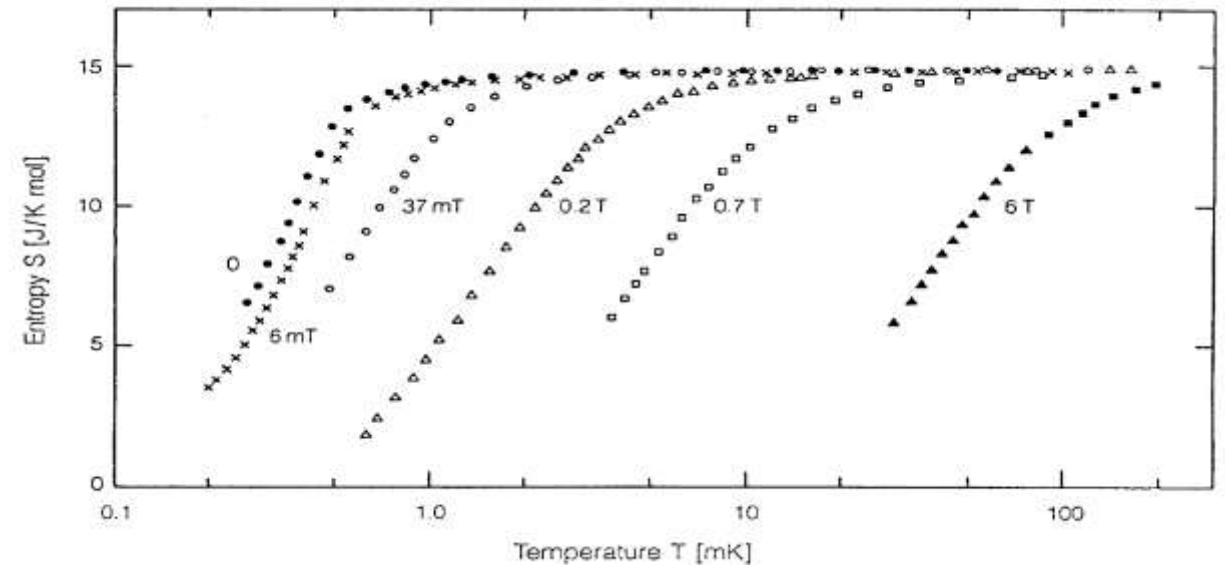
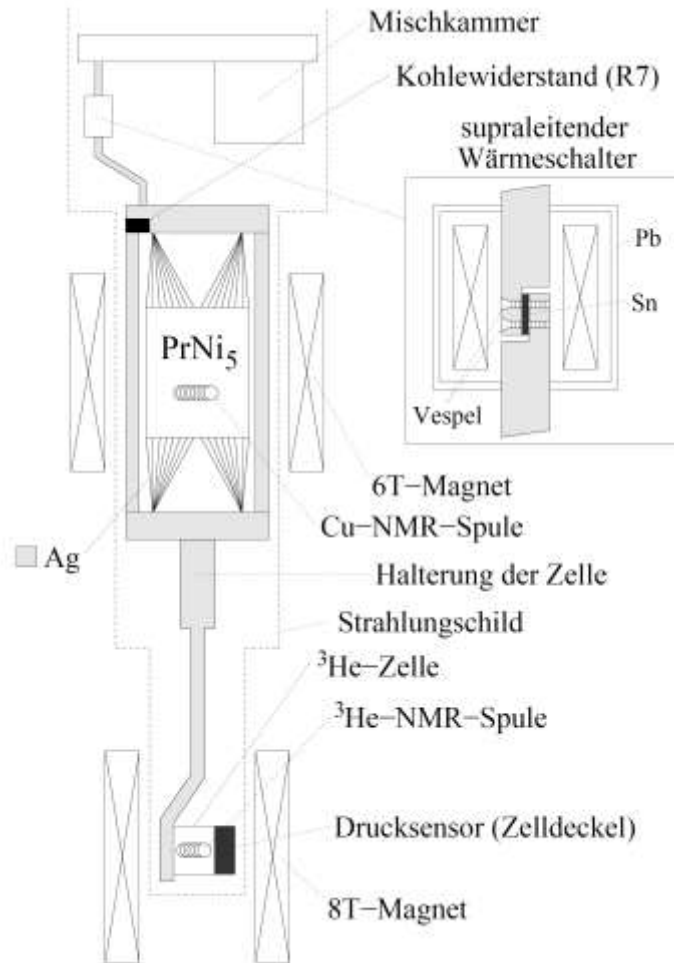
lattice temperatures measured on the 105-mol-copper stage of the Berlin microkelvin facility with Pt-NMR. The achieved minimal temperature was 23.3  $\mu$ K. The red line depicts the calculated course of temperature for the thermodynamically optimized demagnetization function.

heat leak: below 1.5 nW.



# III.1.9 Adiabatic Demagnetization Cooling

## „Bayerische Millimühle 2“ @ WMI





## ***Contents:***

### **III.1 Generation of Low Temperatures**

- III.1.1 Introduction
- III.1.2 Expansion Machine
- III.1.3 Regenerative Machine
- III.1.4 Joule-Thomson Cooling
- III.1.5 Summary
- III.1.6 Evaporation Cooling
- III.1.7 Dilution Cooling
- III.1.8 Pomeranchuk Cooling
- III.1.9 Adiabatic Demagnetization



### **III.2 Thermometry**

- III.2.1 Introduction
- III.2.2 Primary Thermometers
- III.2.3 Secondary Thermometers

# III.2 Thermometry

## III.2.1 Thermometry - Introduction

- temperature and temperature scales

- *temperature of a system in thermodynamic equilibrium:*

defined as the relation between the amount of heat  $\delta Q$  incident on the system during an infinitesimal quasi-static transformation, and the variation  $\delta S$  of its entropy during this transformation:

$$T = \frac{\delta Q}{\delta S}$$

→ for reversible Carnot process ( $dS = 0$ ):

$$0 = \oint \frac{\delta Q}{T}$$

- **Lord Kelvin** (1854): there is an absolute zero of temperature scale

$$\rightarrow T_0 = 0 \text{ K} = -273.15^\circ\text{C}$$

$$1 \text{ K} = 1^\circ\text{C}$$

see <http://www.its-90.com>

*Kelvin scale*

*Celsius scale (1742)*

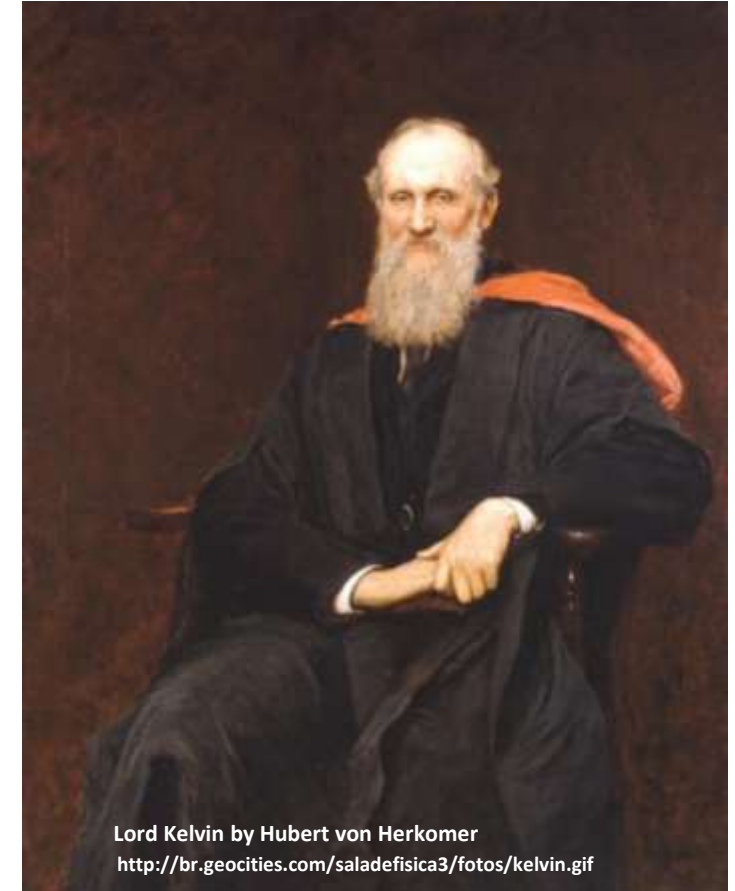
# III.2.1 Thermometry – Introduction

- William Thomson (Lord Kelvin)

Born	<a href="#">26 June 1824</a> , <a href="#">Belfast</a> , <a href="#">Co. Antrim</a> , <a href="#">Ireland</a>
Died	<a href="#">17 December 1907</a> (aged 83) <sup>[1]</sup> , <a href="#">Largs</a> , <a href="#">Ayrshire</a> , <a href="#">Scotland</a> <sup>[1]</sup>
Residence	<a href="#">Cambridge</a> , <a href="#">England</a> <a href="#">Glasgow</a> , <a href="#">Scotland</a>
Nationality	<a href="#">United Kingdom of Great Britain and Ireland</a>
Institutions	<a href="#">University of Glasgow</a>
<i>Alma mater</i>	<a href="#">Glasgow University</a> , <a href="#">Peterhouse</a> , <a href="#">Cambridge</a>

a variety of physical phenomena and concepts with which Thomson is associated are named Kelvin:

- *Kelvin material*
- *Kelvin water dropper*
- *Kelvin wave*
- *Kelvin-Helmholtz instability*
- *Kelvin-Helmholtz mechanism*
- *Kelvin-Helmholtz luminosity*
- *The SI unit of temperature, Kelvin*
- *Kelvin transform in potential theory*
- *Kelvin's circulation theorem*
- *Kelvin-bridge (also known as Thomson-bridge)*



William Thomson (Lord Kelvin)

# III.2.1 Thermometry – Introduction

- temperature scales

- SI temperature scale

the SI temperature scale is the Kelvin scale. It defines the triple point of water as the numerical value of 273.16, i.e., 273.16 K. The unit of temperature in this scale is the Kelvin (K).

- Celsius scale:

the Celsius scale has units of °C (degrees Celsius) with the size of the unit equal to 1 Kelvin.

$$T(^{\circ}\text{C}) = T(\text{K}) - 273.15$$

- agreement of bureaus of standards:

→ **ITS-90 temperature scale** for  $T > 0.65 \text{ K}$  (Comité International des Poids et Mesures 1990)

*the ITS-90 is defined by 17 fixed points and 4 defining instruments. It spans a temperature range from 0.65 K to 10 000 K. For cryogenic purposes the three defining instruments are helium vapor pressure thermometry, gas thermometry, and platinum resistance thermometry.*

→ **PLTS-2000 temperature scale** for lower  $T$  (Provisional Low Temperature Scale, melting curve of  $^3\text{He}$ )

*the PLTS-2000 is defined by a polynomial, relating the melting pressure of  $^3\text{He}$  to temperature from the range 0.9 mK to 1 K. The pressure to temperature relationship is based on primary thermometers such as Johnson noise and nuclear orientation.*

# III.2.1 Thermometry – Introduction

- **temperature scales**
  - **new definition of the Kelvin**

The Kelvin, symbol K, is a unit of measurement for temperature

The Kelvin scale is an absolute scale, which is defined such that 0 K is absolute zero and a change of thermodynamic temperature  $T$  by 1 Kelvin corresponds to a change of thermal energy  $k_B T$  by  $1.380649 \times 10^{-23}$  J.

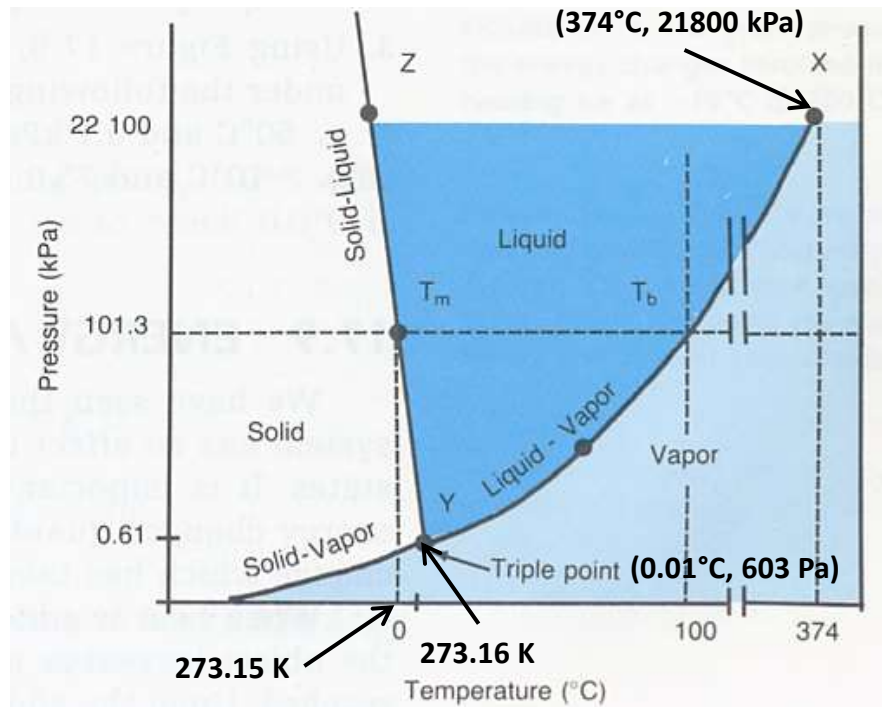
The Boltzmann constant  $k_B = 1.380649 \times 10^{-23} \text{ J} \cdot \text{K}^{-1}$  was exactly defined in the 2019 redefinition of the SI base units such that the triple point of water is  $T = 273.16 \pm 0.0001 \text{ K}$

# III.2.1 Thermometry – Introduction

- The Water Triple Point

The triple point of water is the most important defining thermometric fixed point used in the calibration of thermometers to the [International Temperature Scale of 1990 \(ITS-90\)](#).

It is the sole realizable defining fixed point common to the [Kelvin Thermodynamic Temperature Scale \(KTTS\)](#) and the ITS-90; the assigned value on these scales is **273.16 K (0.01°C)**





# III.2.1 Thermometry – Introduction

- defining Fixed Points of the ITS-90

Number	Temperature		Substance <sup>a</sup>	State <sup>b</sup>	W <sub>r</sub> (T <sub>90</sub> )
	T <sub>90</sub> /K	T <sub>90</sub> /°C			
1	3 to 5	-270.15 to -268.15	He	V	
2	13.8033	-259.3467	e-H <sub>2</sub>	T	0.001 190 07
3	~17	~-256.15	e-H <sub>2</sub> (or He)	V (or G)	
4	~20.3	-252.85	e-H <sub>2</sub> (or He)	V (or G)	
5	24.5561	-248.5939	Ne	T	0.008 449 74
6	54.3584	-218.7916	O <sub>2</sub>	T	0.091 718 04
7	83.8058	-189.3442	Ar	T	0.215 859 75
8	234.3156	-38.8344	Hg	T	0.844 142 11
9	273.16	0.01	H <sub>2</sub> O	T	1.000 000 00
10	302.9146	29.7646	Ga	M	1.118 138 89
11	429.7485	156.5985	In	F	1.609 801 85
12	505.078	231.928	Sn	F	1.892 797 68
13	692.677	419.527	Zn	F	2.568 917 30
14	933.473	660.323	Al	F	3.376 008 60
15	1234.93	961.78	Ag	F	4.286 420 53
16	1337.33	1064.18	Au	F	
17	1357.77	1084.62	Cu	F	

<sup>a</sup> All substances except <sup>3</sup>He are of natural isotopic composition, e-H<sub>2</sub> is hydrogen at the equilibrium concentration of the ortho- and para-molecular forms.  
<sup>b</sup> V: vapour pressure point; T: Triple Point (temperature at which the solid, liquid and vapour phases are in equilibrium); G: gas thermometer point; M,F melting point, freezing point (temperature, at a pressure of 101 325 Pa, at which the solid and liquid phases are in equilibrium)

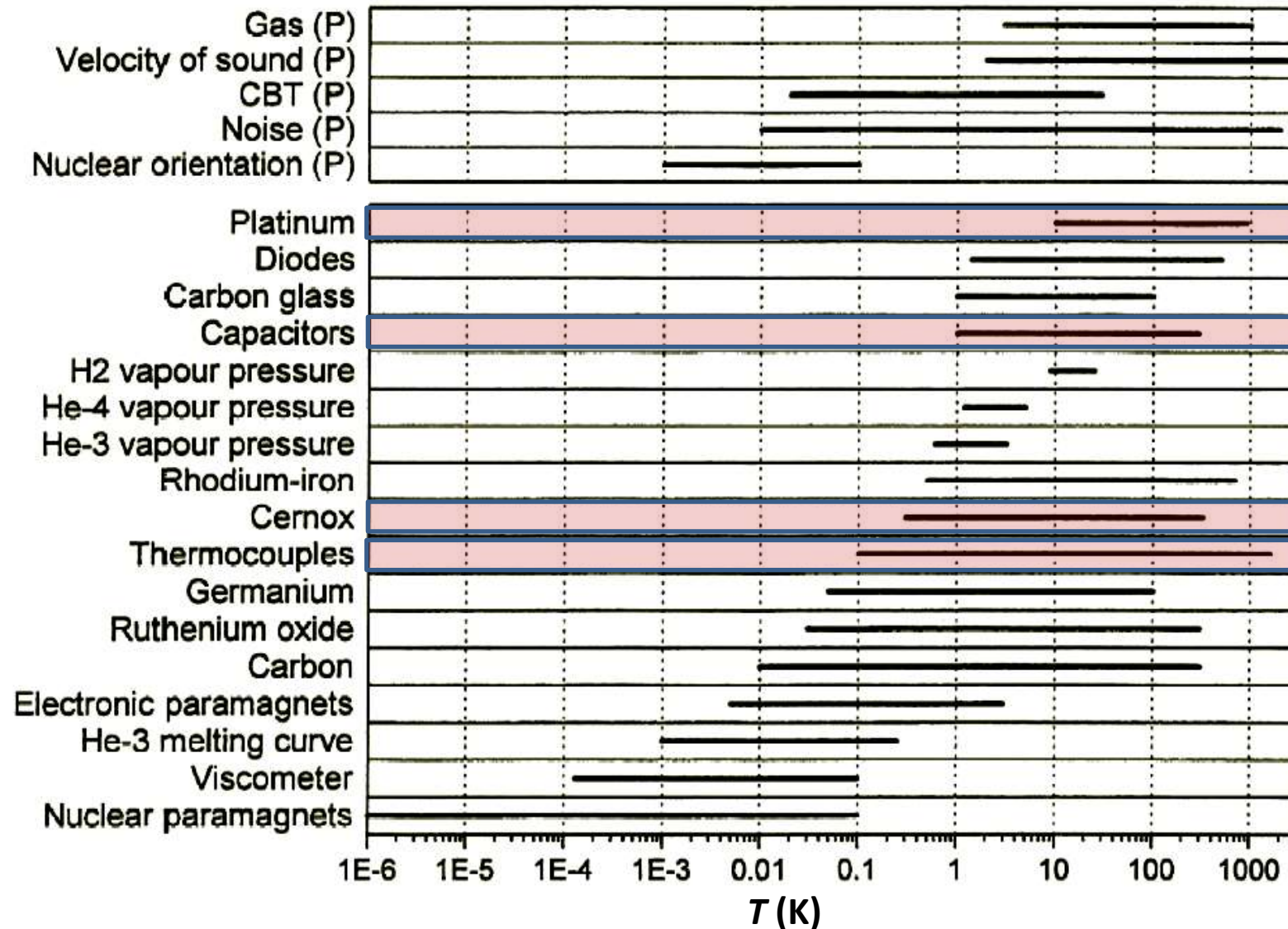
see <http://www.its-90.com>

# III.2.1 Thermometry – Introduction

- temperature measurement
  - definition of temperature via reversible Carnot process is not well suited for establishing useful measuring methods
  - in practice: use of *fixpoints* and *interpolation polynoms*
  - *primary thermometers*:
    - measured quantity is related directly to temperature (*in a theoretically predictably way*)
    - no calibration is required
  - *secondary thermometers*:
    - measured quantity varies with temperature in a reproducible way
    - must be calibrated using a primary thermometer
  - requirements for temperature measurement:
    - → good thermal contact between thermometer and sample
    - → low self-heating
    - fast response to temperature changes

# III.2.1 Thermometry – Introduction

- typical temperature range of some thermometers



# III.2.1 Thermometry – Introduction

- most common thermometers for  $1\text{K} < T < 300\text{ K}$ 
  - gas thermometer:  $p = p(T)$ 
    - Helium gas  $\approx$  ideal gas down to 10K:
  - vapour pressure thermometer:  $T_{\text{liquid}} = f(p_{\text{vapor}})$ 
    - pressure of 10 Pa corresponds to 0.4 K for  $^3\text{He}$
  - thermocouples:  $V_{\text{th}} = V_{\text{th}}(T)$
  - resistance thermometry:  $R = R(T)$ 
    - 1K - 300K
    - semiconductors (e.g. Ge doped with Arsenic has 100-500  $\Omega/\text{K}$  @ 4.2K, self-heating around 10  $\mu\text{A}$ )
    - p-n junction diode (problem with high bias current  $\rightarrow$  self heating)
  - capacitance thermometry:  $C = C(T)$ 
    - based on temperature change of dielectric properties
    - virtually no magnetic field-induced errors
  - noise thermometer:  $S = S(T)$ 
    - Johnson noise in resistor:  $S_V = 4k_B T R$
    - like gas thermometer, but with electrons
    - with SQUID measurements: 0.1% @ 1K



# III.2.1 Thermometry – Introduction

- most common thermometers for  $T < 1$  K

$1 \text{ mK} \leq T \leq 1 \text{ K}$ :

- magnetic susceptibility thermometer**

Curie's law:  $\chi = \mu_0 \frac{M}{B} = \frac{C}{T}$

$M$ : magnetization  
 $B$ : applied magnetic field  
 $C$ : Curie constant

mutual inductance between two coils:  $m = m_0 f \chi$

→ Cerium magnesium nitrate (CMN) useful from 1 K – 10 mK

→ low temperature limit set by magnetic ordering at  $\approx 1$  mK



- resistance thermometers**

$T < 1 \text{ mK}$ :

- nuclear Magnetic Resonance (NMR) thermometer**

→ temperature dependence of spin relaxation


→ platinum ideal choice for NMR thermometry

## ***Contents:***

### **III.1 Generation of Low Temperatures**

- III.1.1 Introduction
- III.1.2 Expansion Machine
- III.1.3 Regenerative Machine
- III.1.4 Joule-Thomson Cooling
- III.1.5 Summary
- III.1.6 Evaporation Cooling
- III.1.7 Dilution Cooling
- III.1.8 Pomeranchuk Cooling
- III.1.9 Adiabatic Demagnetization

### **III.2 Thermometry**

- III.2.1 Introduction
-  III.2.2 Primary Thermometers
- III.2.3 Secondary Thermometers



# III.2.2 Primary Thermometers


- gas thermometers

- ideal gas would be a perfect thermometer:

$$pV = nRT \quad \rightarrow \text{measure pressure at constant volume}$$

- for real gases life is more complicated  $\rightarrow$  deviations from ideal behavior

$$pV = n\{RT + b(T)p + c(T)T^2 + d(T)T^3 + \dots\}$$

  
 virial coefficients (tabulated ITS-90 values)

- systematic errors:
  - $\rightarrow$  dead volumes
  - $\rightarrow$  thermal expansion of cell, elastic deformation of cell
  - $\rightarrow$  adsorption and desorption from walls

$\rightarrow$  *mainly used in calibration laboratories !*

# III.2.2 Primary Thermometers

- vapour pressure thermometry

- Clausius-Clapeyron equation: 
$$\frac{dp}{dT} = \frac{L(T)}{(V_{\text{gas}} - V_{\text{liquid}})T} \approx \frac{\Delta H_{\text{vap}}(T)}{V_{\text{gas}}T}$$

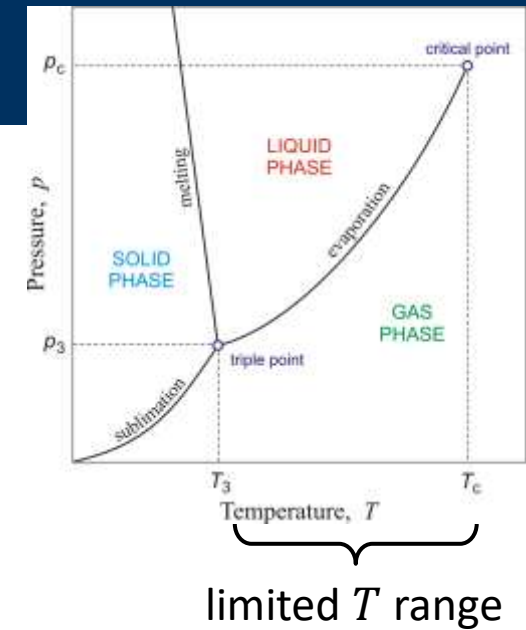
- for ideal gas ( $pV = RT$ ): 
$$R \ln p + \text{const.} = \int \frac{\Delta H_{\text{vap}}(T)}{T^2} dT$$

- if  $\Delta H_{\text{vap}}(T)$  is known  $\rightarrow$  **determine  $T$  of liquid (e.g. He) via measurement of He pressure above liquid**

- in practice, a set of secondary vapour pressure scales is used:

ITS-90: 
$$T = \sum_i A_i \left\{ \frac{(\ln p + B)}{C} \right\}^i$$
 (in principle no primary thermometer !!)

- with  $^3\text{He}$  and  $^4\text{He}$ : ITS defined down to 0.65 K



# III.2.2 Primary Thermometers

- helium vapor pressure thermometer

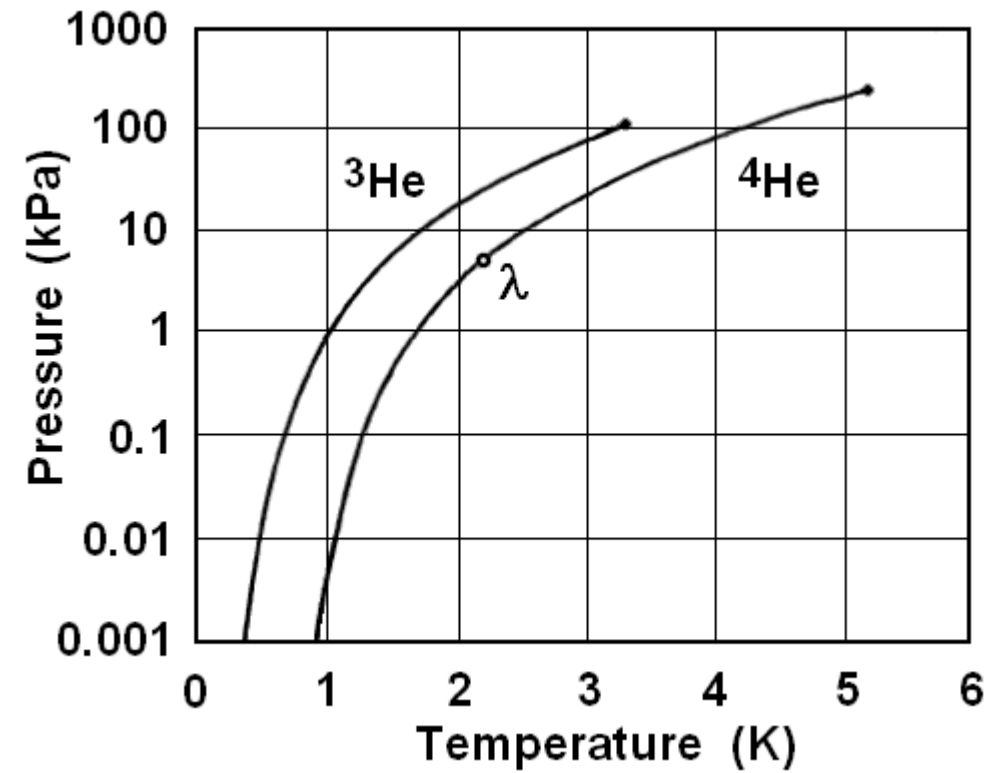
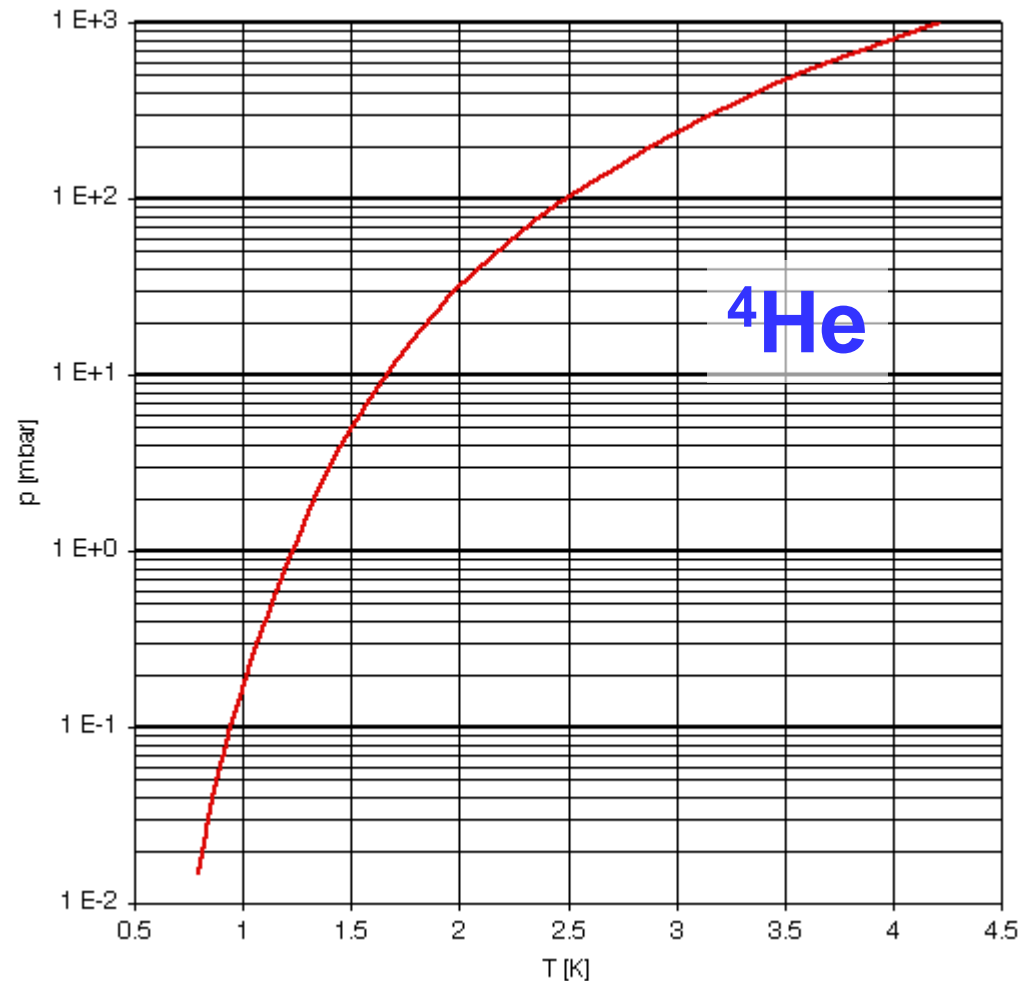
values of the constants for the helium vapour-pressure, and the temperature range for which each equation, identified by its set of constants, is valid (see <http://www.its-90.com>).

	<sup>3</sup> He 0.65 K to 3.2 K	<sup>4</sup> He 1.25 K to 2.1768 K	<sup>4</sup> He 2.1768 K to 5.0 K
A <sub>0</sub>	1.053 447	1.392 408	3.146 631
A <sub>1</sub>	0.980 106	0.527 153	1.357 655
A <sub>2</sub>	0.676 380	0.166 756	0.413 923
A <sub>3</sub>	0.372 692	0.050 988	0.091 159
A <sub>4</sub>	0.151 656	0.026 514	0.016 349
A <sub>5</sub>	-0.002 263	0.001 975	0.001 826
A <sub>6</sub>	0.006 596	-0.017 976	-0.004 325
A <sub>7</sub>	0.088 966	0.005 409	-0.004 973
A <sub>8</sub>	-0.004 770	0.013 259	0
A <sub>9</sub>	-0.054 943	0	0
B	7.3	5.6	10.3
C	4.3	2.9	1.9

$$T = \sum_i A_i \left\{ \frac{(\ln p + B)}{C} \right\}^i$$

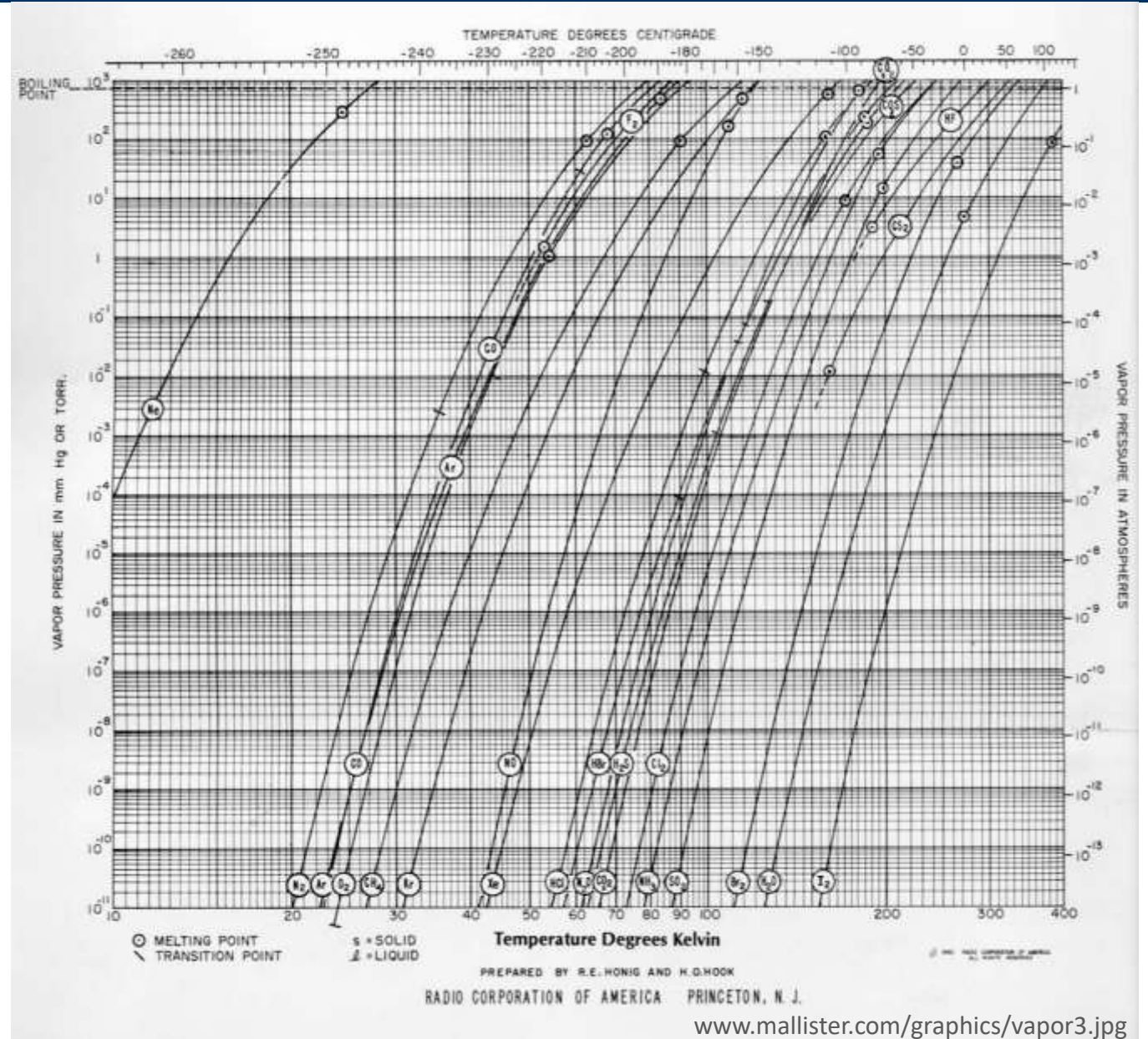
# III.2.2 Primary Thermometers

- helium vapor pressure thermometer



# III.2.2 Primary Thermometers

- vapor pressure thermometry



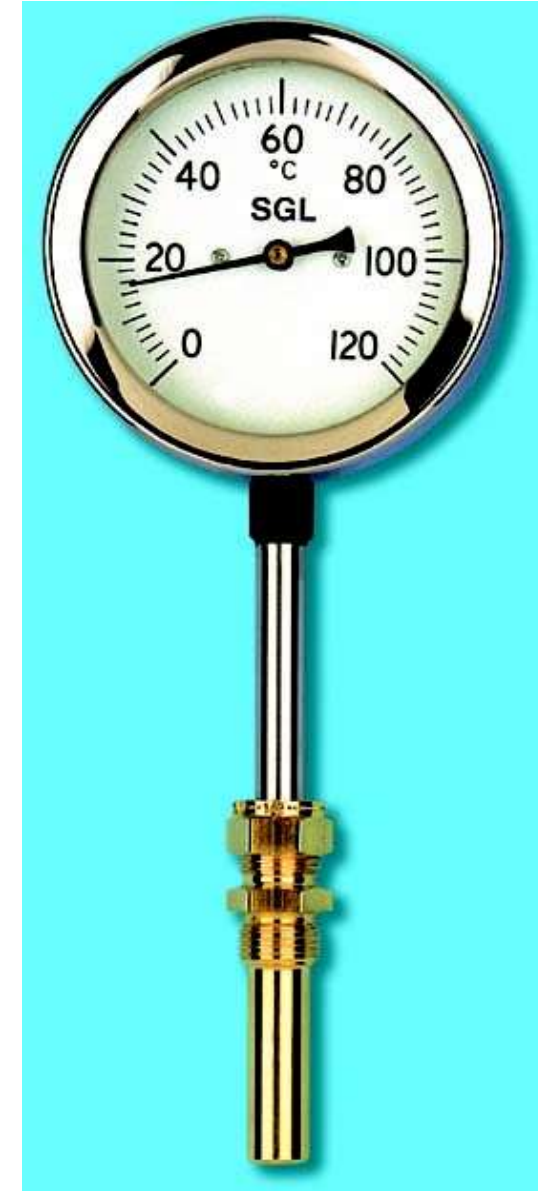
# III.2.2 Primary Thermometers

- vapor pressure thermometry



[www.bm-industries.com](http://www.bm-industries.com)

Scale in °C	Range of measure in °C
- 40+25	-13+15
- 25+40	+5+30
- 10+60	+25+50
0+100	+45+85
+25+125	+70+110
+40+160	+100+145
+50+200	+120+185
+60+250	+165+230
+100+320	+230+300
+160+400	+280+375





## III.2.2 Primary Thermometers

- $^3\text{He}$  melting curve thermometry

- use of melting curve of  $^3\text{He}$  to define PLTS-2000 temperature scale *down to 0.9 mK*

- polynom for melting curve:

$$p = \sum_{i=-3}^9 \alpha_i T^i$$

→ coefficients given by **PLTS-2000**

→ also use of 4 fix points

*minimum of melting curve,  
transition temperatures to A and B phase and  
afm order of nuclear spins in solid  $^3\text{He}$*

# III.2.2 Primary Thermometers

- $^3\text{He}$  melting curve thermometry

$$p/\text{MPa} = \sum_{i=-3}^{+9} a_i (T_{2000}/\text{K})^i$$

$$a_{-3} = -1.385\,544\,2 \cdot 10^{-12}$$

$$a_{-2} = 4.555\,702\,6 \cdot 10^{-9}$$

$$a_{-1} = -6.443\,086\,9 \cdot 10^{-6}$$

$$a_0 = 3.446\,743\,4 \cdot 10^0$$

$$a_1 = -4.417\,643\,8 \cdot 10^0$$

$$a_2 = 1.541\,743\,7 \cdot 10^1$$

$$a_3 = -3.578\,985\,3 \cdot 10^1$$

$$a_4 = 7.149\,912\,5 \cdot 10^1$$

$$a_5 = -1.041\,437\,9 \cdot 10^2$$

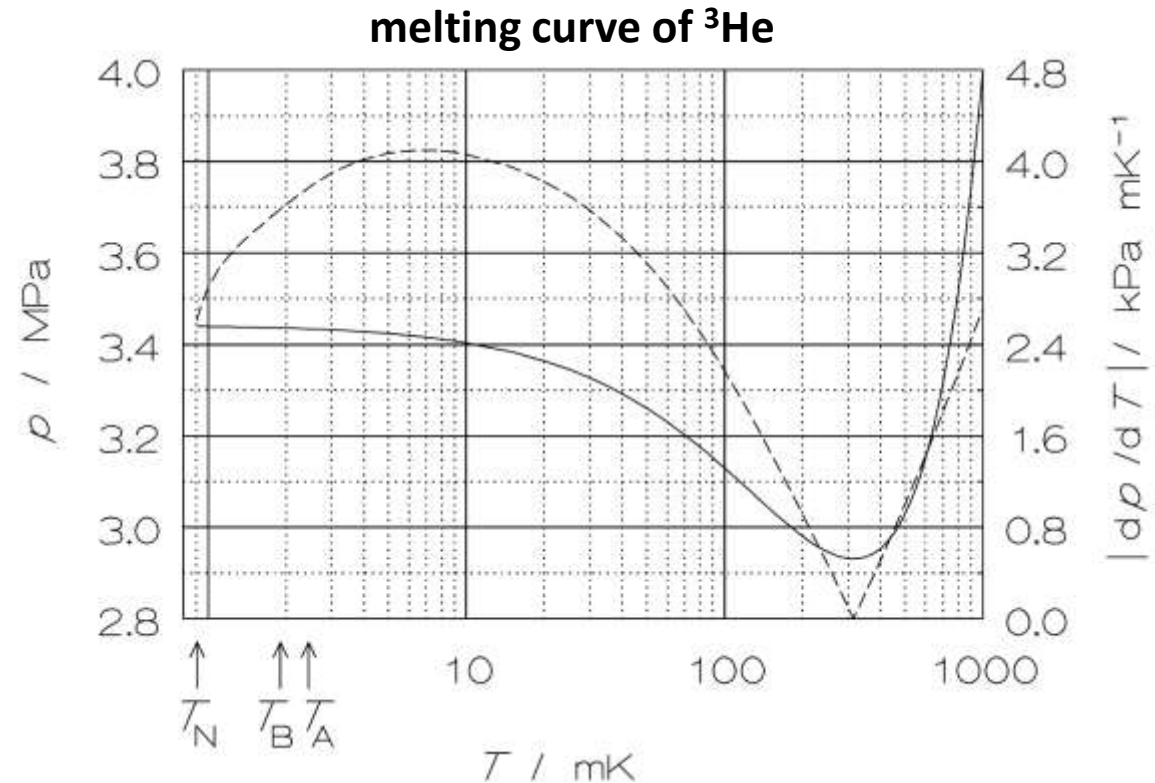
$$a_6 = 1.051\,853\,8 \cdot 10^2$$

$$a_7 = -6.944\,376\,7 \cdot 10^1$$

$$a_8 = 2.683\,308\,7 \cdot 10^1$$

$$a_9 = -4.587\,570\,9 \cdot 10^0$$

source: R.L. Rusby et al. (2001)



Point	$p/\text{MPa}$	$T_{2000}/\text{mK}$
minimum	2.93113	315.24
A	3.43407	2.444
A-B	3.43609	1.896
Néel	3.43934	0.902

## III.2.2 Primary Thermometers

- noise thermometry

- Nyquist theorem:

$$S_V = 4k_B T R$$

voltage noise  
 power spectral density  
 ( $V^2/\text{Hz}$ )

temperature

resistance

valid only in the low frequency limit  $f \ll k_B T / h$  ( $\approx 20 \text{ GHz @ } 1\text{K}$ )

- temperature determined by measurement of  $S_V$  and  $R$

- **example:**  $R = 10 \text{ k}\Omega$ ,  $T = 1 \text{ K}$ , band width  $\Delta f = 105 \text{ Hz}$

$\rightarrow (S_V \Delta f)^{1/2} \approx 2 \cdot 10^{-7} \text{ V} \rightarrow$  sensitive amplifier required  $\rightarrow$  SQUID preamplifier

$\rightarrow P \approx 10^{-18} \text{ W}$

# III.2.2 Primary Thermometers

- superconducting fix point thermometers
  - based on the precise measurement of the transition temperatures of superconductors
  - available from NIST at Boulder

## NIST fixpoint device

### ITS-90

Element	TC90/ K	<sup>+</sup> T <sup>C</sup> ( K)	Substance	Width (mK)	Reproducibility (1 standard deviation) (mK)
		0.0160	°W	0.7	± 0.2
Cd	0.5200 ± 0.0030	0.0230	°Be	0.2	0.1
Zn	0.8500 ± 0.0030	0.0990	°Ir	0.8	0.1
Al	1.1810 ± 0.0025	0.1605	°AuAl <sub>2</sub>	0.3	0.1
In	3.4145 ± 0.0025	0.2065	°AuIn <sub>2</sub>	0.4	0.15
Pb	7.1997 ± 0.0025	0.5190	+Cd	0.5-8.0	0.3
Nb	9.2880 ± 0.0025	0.8510	+Zn	2.5-10	0.4
		1.1796	+Al	1.5-4.0	0.4
		3.4145	+In	0.5-2.5	0.2
		7.1996	+Pb	0.6-2.0	0.2

°SRM 768 [Schooley and Soulen (1982)]


+SRM 767 [Schooley et al. (1980)] \* T<sub>90</sub> above 0.5 K.

## ***Contents:***

### **III.1 Generation of Low Temperatures**

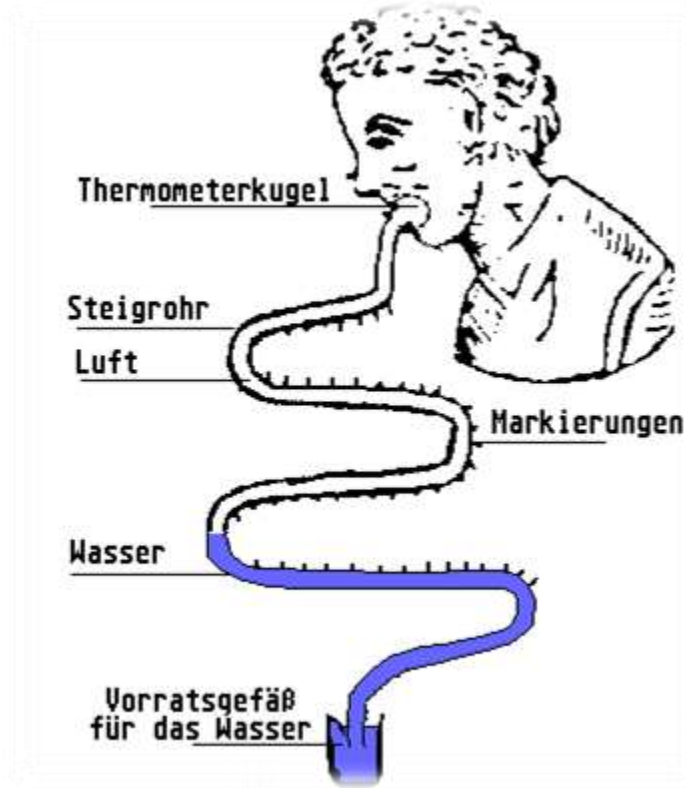
- III.1.1 Introduction
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### **III.2 Thermometry**

- III.2.1 Introduction
- III.2.2 Primary Thermometers
-  III.2.3 Secondary Thermometers

# III.2.3 Secondary Thermometers

- there are many ....





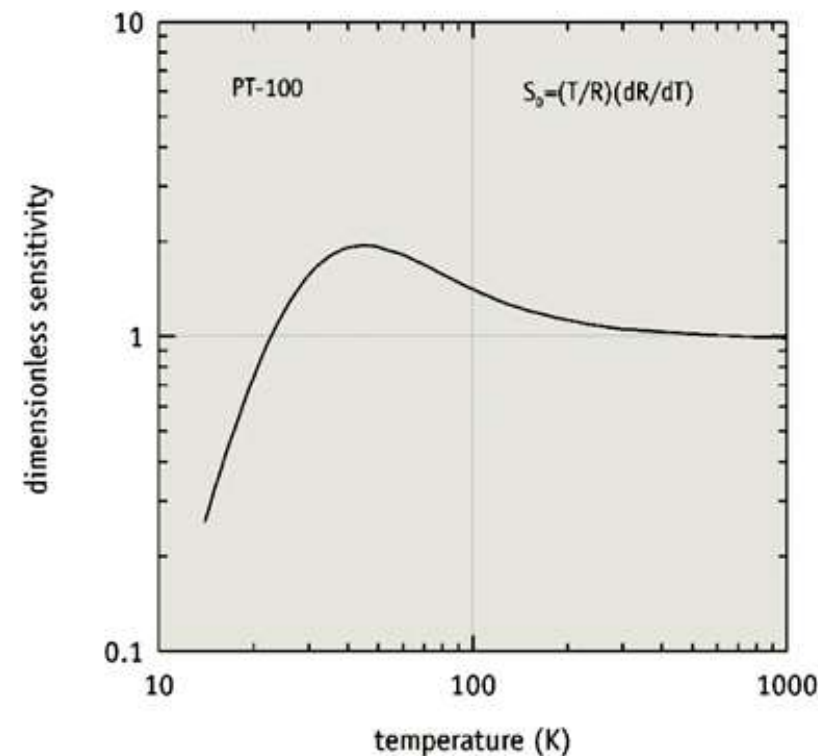
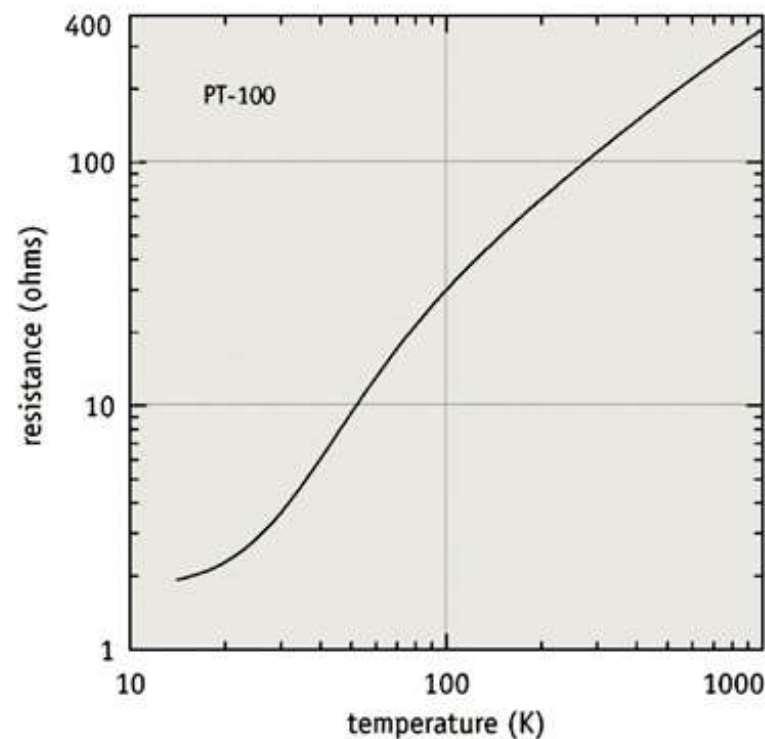
## III.2.3 Secondary Thermometers

- resistance thermometers
  - required: well established relation between resistance and temperature, sufficiently large  $dR/dT$
  - advantage: resistance easy to measure → resistance thermometry very popular
  - fact: temperature variation of resistance may have very different physical origin
  - commonly used:
    - Pt resistors (PT-100, PT-1000)
    - RhFe resistors
    - carbon resistors (Speer, Allen-Bradley)
    - carbon glass resistors
    - Ge resistors
    - RuO<sub>2</sub> resistors

# III.2.3 Secondary Thermometers

- Platinum resistors

The **platinum resistance thermometer (PRT)** is very widely used below 500 °C as a thermometric sensor. There is a wide range of quality of PRT available, from the standard instrument (SPRT) of the ITS-90 to some industrial types (IPRT) that are accurate only to within a few tenths of a kelvin or, perhaps, even a kelvin or more. The major difference of the industrial type of fabrication from the standard type is not just the purity of platinum, but also the less strain-free mounting of the film or wire which is embedded (partially or totally) in a cement (glass or refractory). Furthermore, in most cases, the thermometer body is not hermetically sealed.



Source: Lake Shore Cryotronics, Inc.

# III.2.3 Secondary Thermometers

- Platinum resistors

- ITS-90 requirement for Pt resistance thermometer (PRT)*

temperatures are determined in terms of the ratio of the resistance  $R(T_{90})$  at a temperature  $T_{90}$  and the resistance  $R(273.16 \text{ K})$  at the triple point of water:

$$W(T_{90}) = R(T_{90})/R(0.01^\circ\text{C}) \quad \longrightarrow \quad \begin{aligned} W(29.7646^\circ\text{C}) &\geq 1.118\,07 \\ W(-38.8344^\circ\text{C}) &\leq 0.844\,235 \end{aligned}$$

- industrial PRT*

for  $0 < T < 100^\circ\text{C}$

$$R = R_0 \cdot (1 + \alpha \cdot T)$$

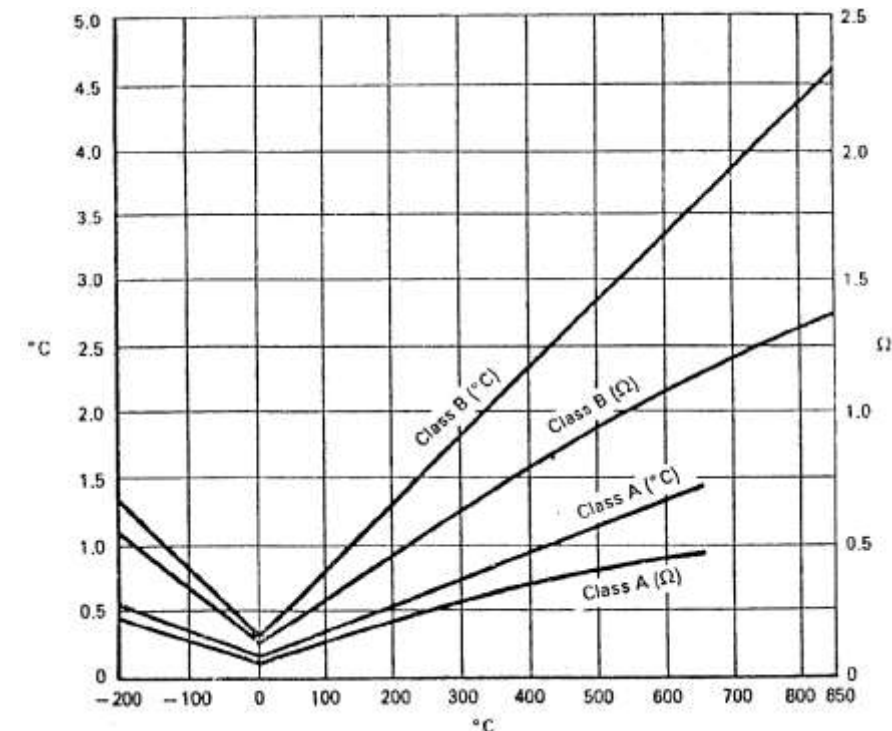
$$\alpha = 3.85 \cdot 10^{-3} / \text{K}$$

allowed errors in  $^\circ\text{C}$ :

Class A:  $dT = \pm (0.15^\circ\text{C} + 0,002 \cdot T)$

Class B:  $dT = \pm (0.30^\circ\text{C} + 0,005 \cdot T)$

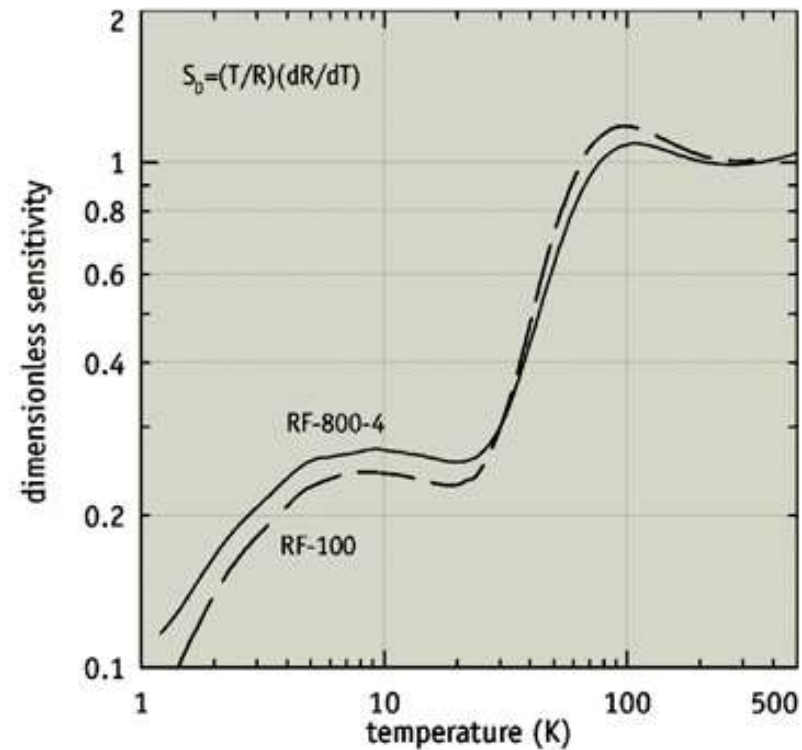
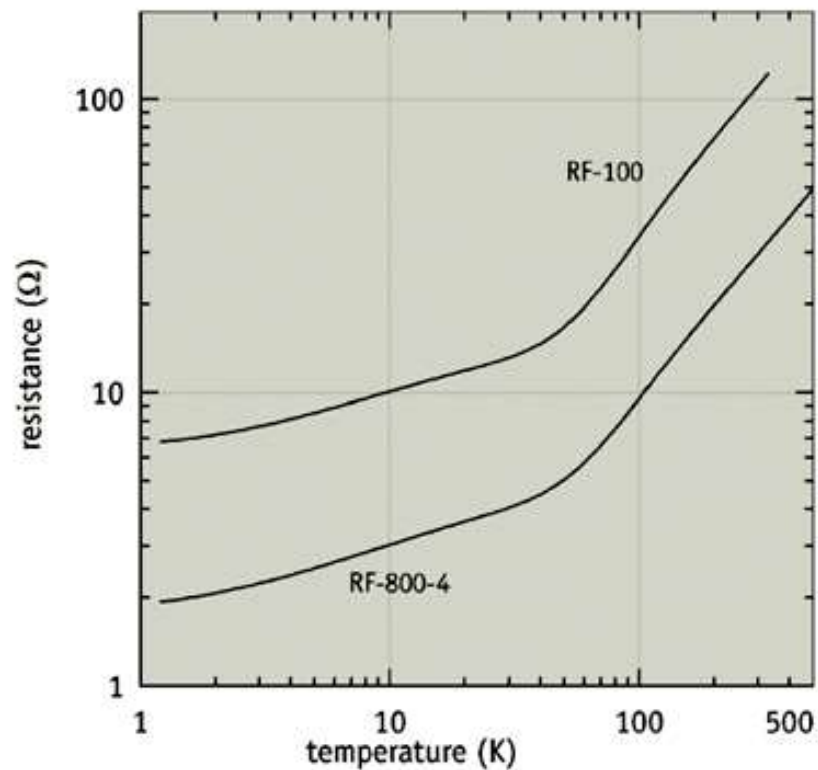
1/3 Class B:  $dT = \pm 1/3 \cdot (0.30^\circ\text{C} + 0.005 \cdot T)$



# III.2.3 Secondary Thermometers

- RhFe resistor thermometer

*Rhodium with 0.5% Fe*



Source: Lake Shore Cryotronics, Inc.

# III.2.3 Secondary Thermometers

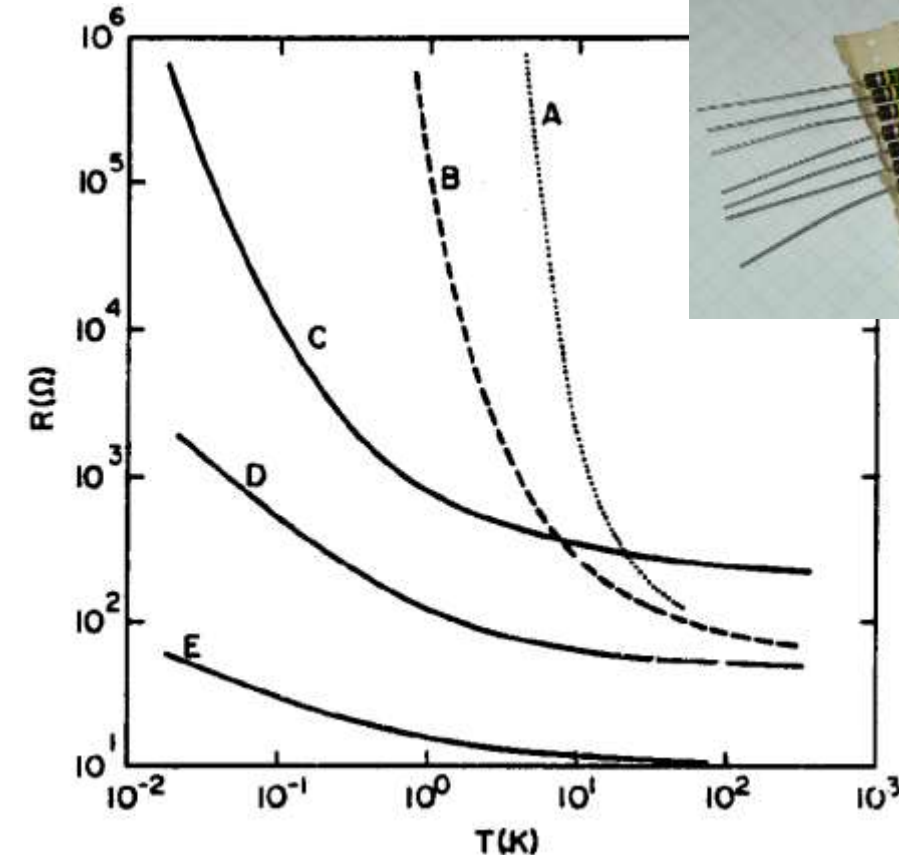
- **carbon resistors**

commercial carbon-composition resistors manufactured by Allen-Bradley were introduced as temperature sensors by Clement and Quinnell in 1952. The carbon composition resistor is a small cylinder consisting of graphite with a binder encased in an outer phenolic shell.

The carbon resistors used as thermometers are generally characterized by their room temperature resistance and their wattage [see Rubin (1980)], and have come largely from the following manufacturers:

- *Allen-Bradley*
- *Airco Speer* (usually referred to simply as *Speer*)
- *Ohmite*
- *Matsushita*,
- *CryoCal*

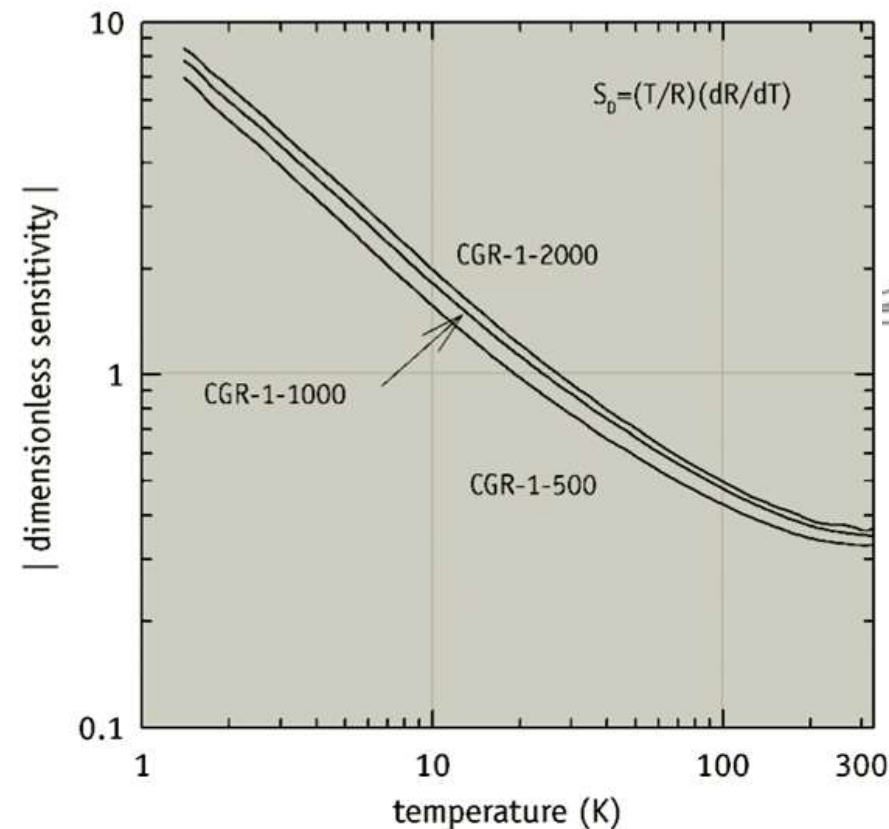
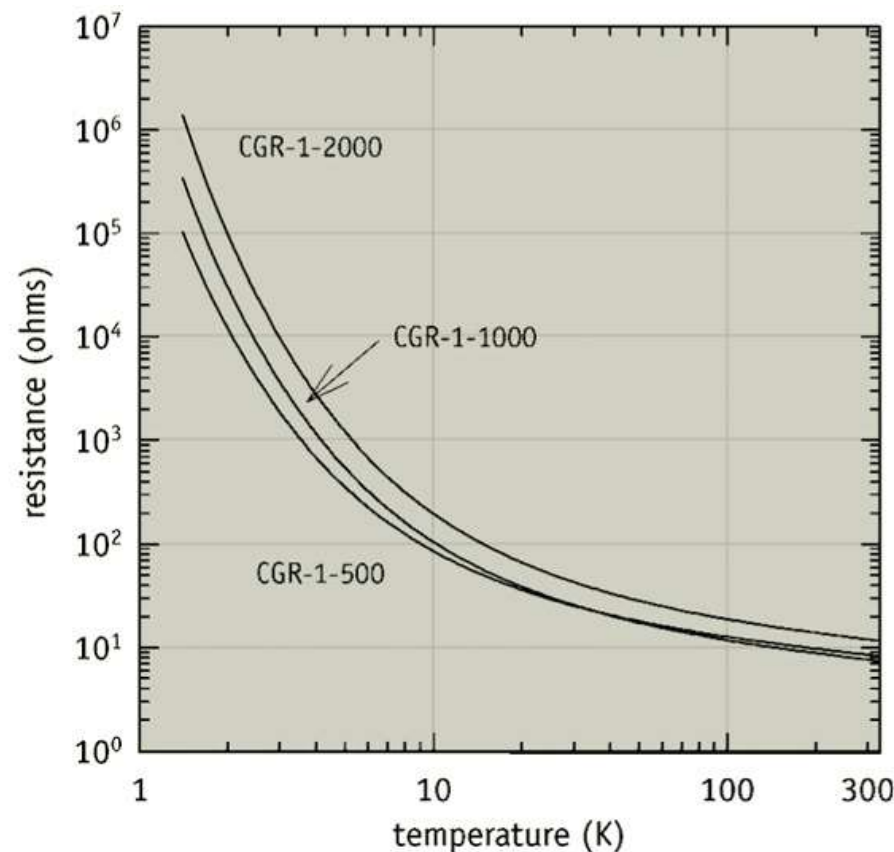
A: thermistor,  
 B: 68  $\Omega$  Allen-Bradley,  
 C: 220  $\Omega$  Speer (grade 1002),  
 D: 51  $\Omega$  Speer (grade 1002),  
 E: 10  $\Omega$  Speer (grade 1002)



# III.2.3 Secondary Thermometers

- carbon glass resistors

A porous glass is prepared by removing the boron-rich phase from a borosilicate alkaline glass to leave a material having the appearance of silicate spheres of about 30 nm diameter, randomly distributed and separated by 3 to 4 nm pores. The spaces are then partially filled with high-purity carbon to form amorphous fibres → better stability than carbon resistors.



Source: Lake Shore Cryotronics, Inc.



# III.2.3 Secondary Thermometers

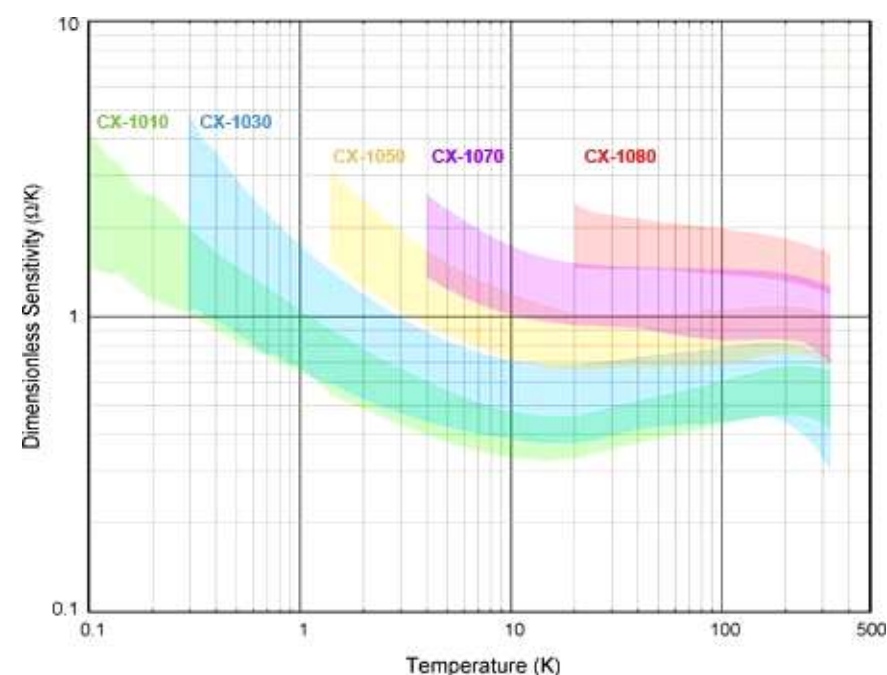
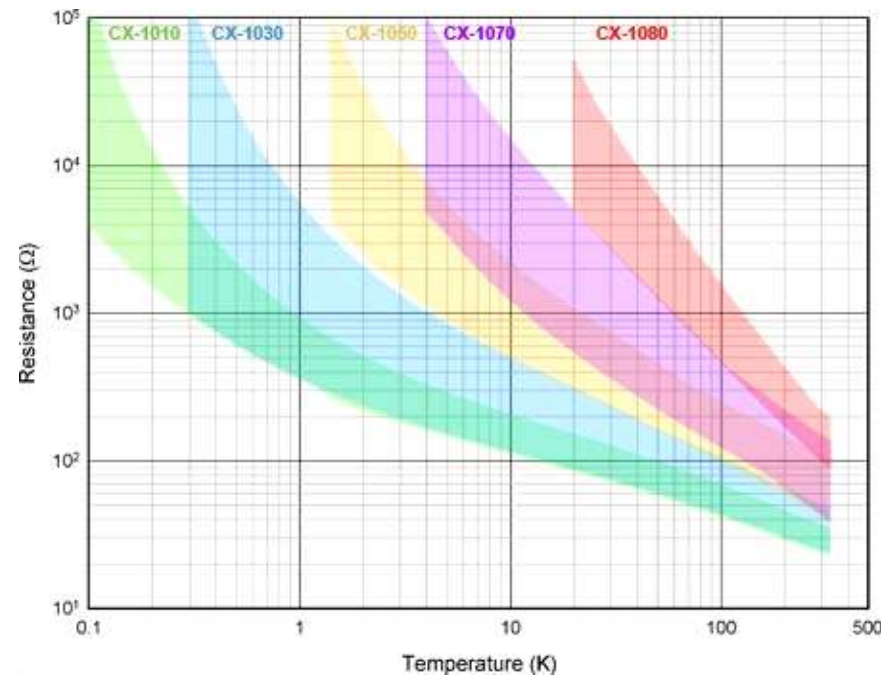
- Cernox resistors

Patents:

#5,363,084, Nov. 1994, "Film Resistors Having Trimmable Electrodes"

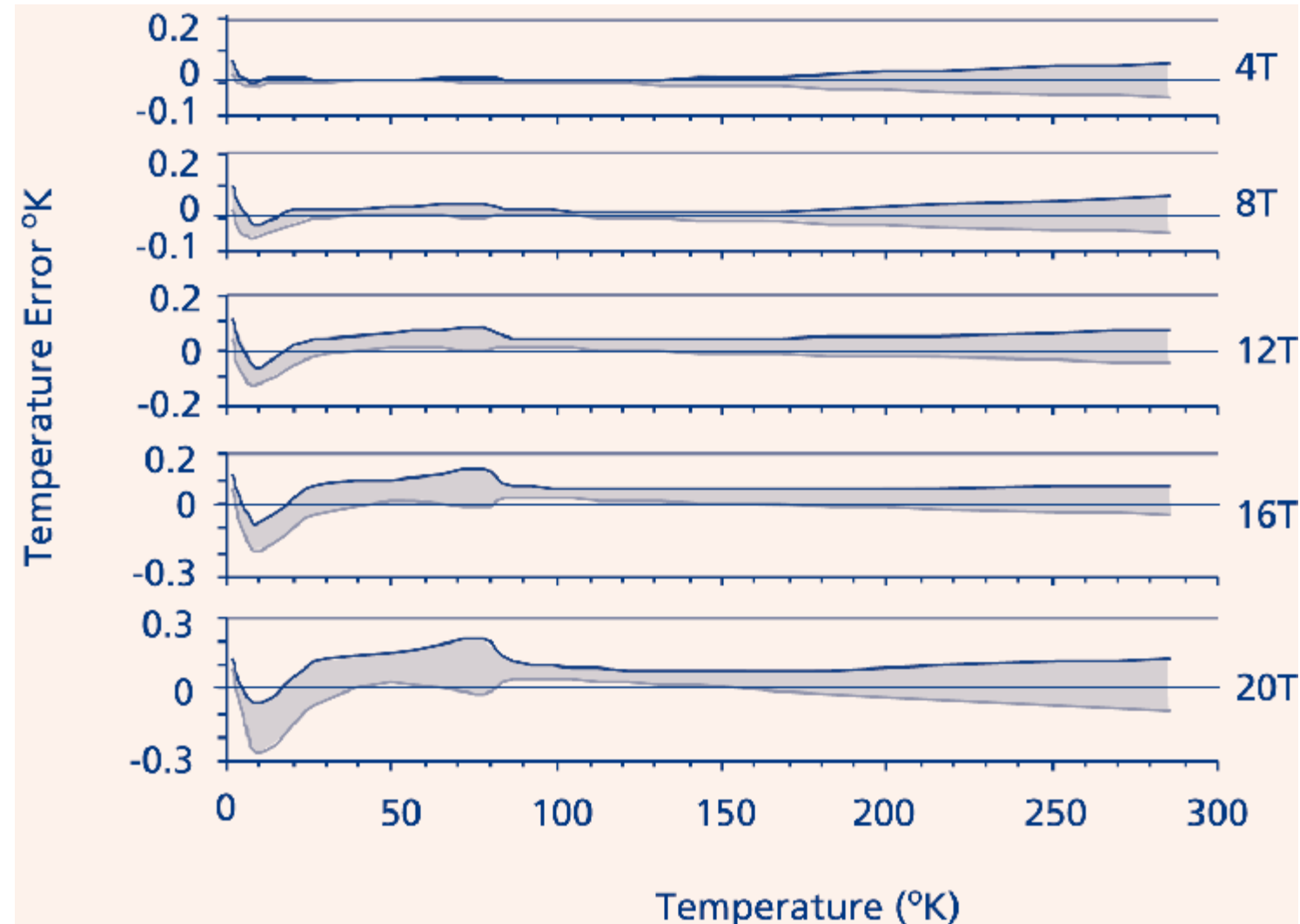
#5,367,285, Nov. 1994, "Cernox™", "Metal Oxy-nitride Resistance Films and Methods of Making the Same,"

→ **small magnetic field coefficient**:  $\Delta T/T$  typically smaller than 0.5% @ 19 T



## III.2.3 Secondary Thermometers

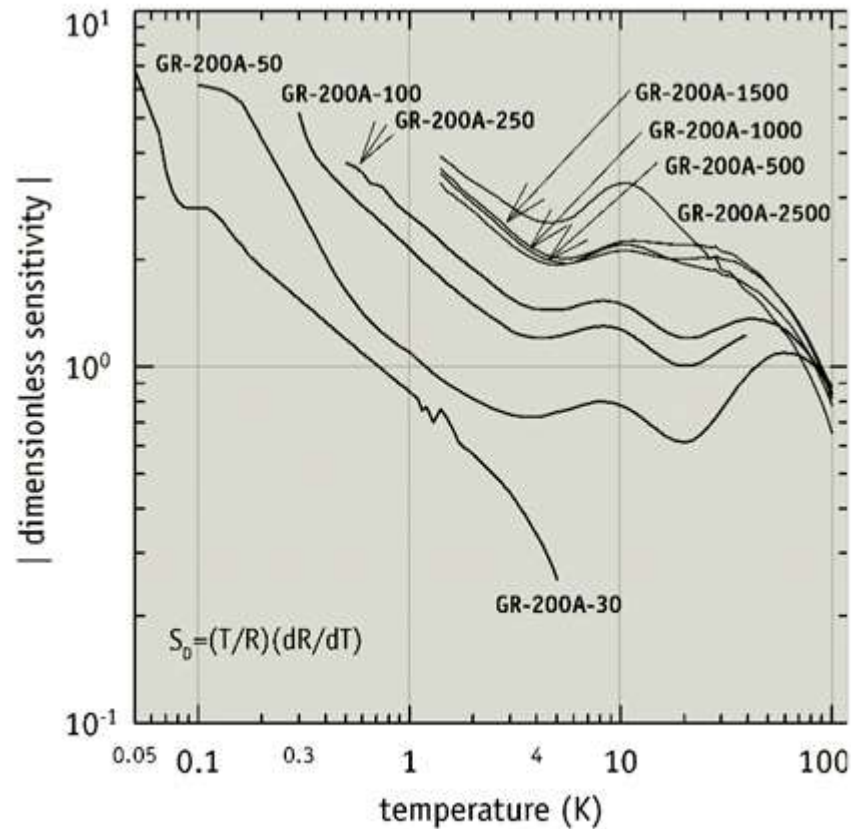
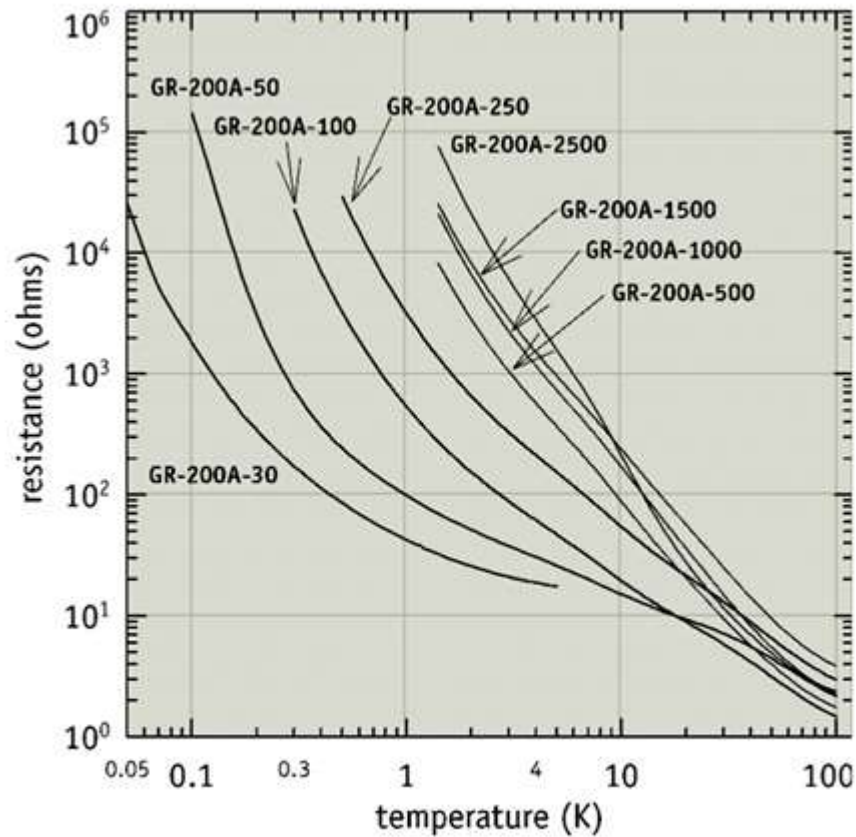
- Cernox resistors



Low temperature thermometry in high magnetic fields VII. Cernox sensors to 32 T, B.L. Brandt *et al*, *Rev. Sci. Instrum.*, vol 70, No 1, 1999, pp 104-110.

# III.2.3 Secondary Thermometers

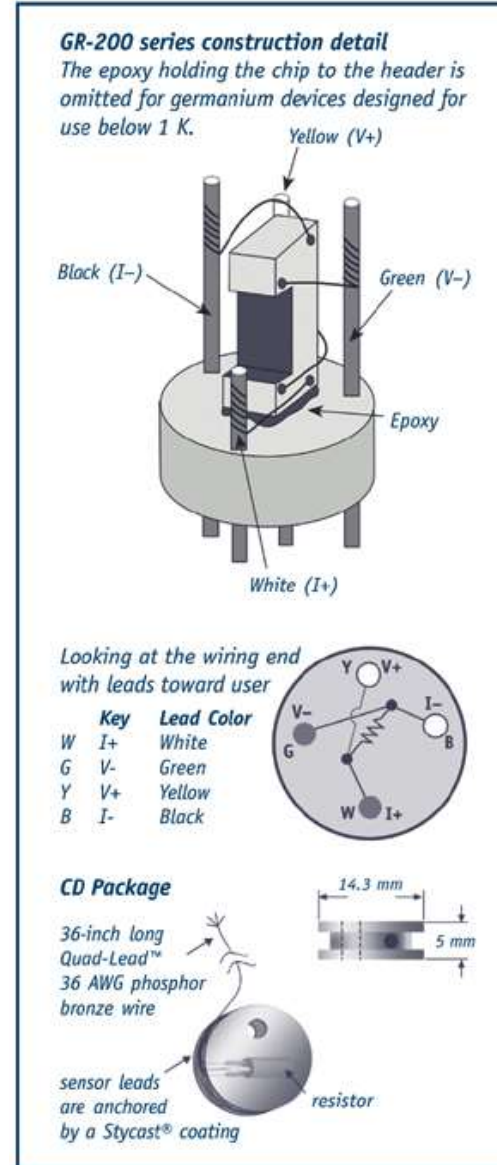
- Germanium resistors



Source: Lake Shore Cryotronics, Inc.

# III.2.3 Secondary Thermometers

- Germanium resistors



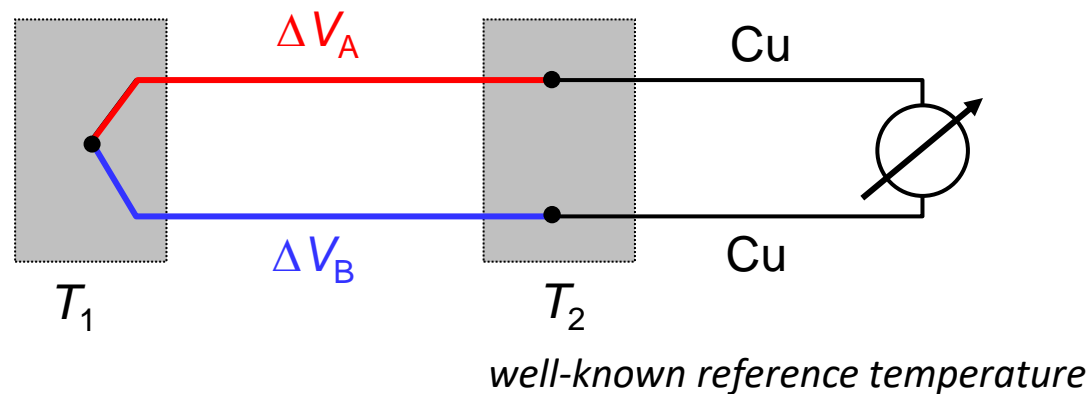
Source: Lake Shore Cryotronics, Inc.

# III.2.3 Secondary Thermometers

- Thermocouples

Thermocouples are pairs of dissimilar metal wires joined at least at one end, which generate a net thermoelectric voltage between the the open pair according to the size of the temperature difference between the ends, the relative Seebeck coefficient of the wire pair and the uniformity of the wire-pair relative Seebeck coefficient.

- based on Seebeck effect:  $\Delta V = S \cdot \Delta T$   $S$  = thermopower
- measurement of difference of thermovoltages of two different materials



# III.2.3 Secondary Thermometers

- Thermocouples

- **Chromel-Gold/Iron (0.07%)**

consists of a Gold (Au)-0.07 at % Iron (Fe) as the negative thermoelement and a Ni-Cr alloy (Chromel) as the positive thermoelement. This thermocouple is more widely used because of its relatively high thermoelectric sensitivity ( $>15 \mu\text{V/K}$  above 10 K).

- **Type E (Chromel (Ni-Cr-alloy) / Constantan (Cu-Ni-alloy) )**

has the **highest sensitivity** among the three standard thermocouple types typically used at **low temperatures** (types E, K, and T). The best choice for temperatures down to 40 K.

- **Type K (Chromel (Ni-Cr-alloy) / Alumel (Ni-Al-alloy) )**

recommended for continuous use in inert atmospheres. Has a sensitivity of **4.1 mV/K at 20 K** (about  $\frac{1}{2}$  of Type E).

- **Type T (Copper / Constantan)**

**many more !!**



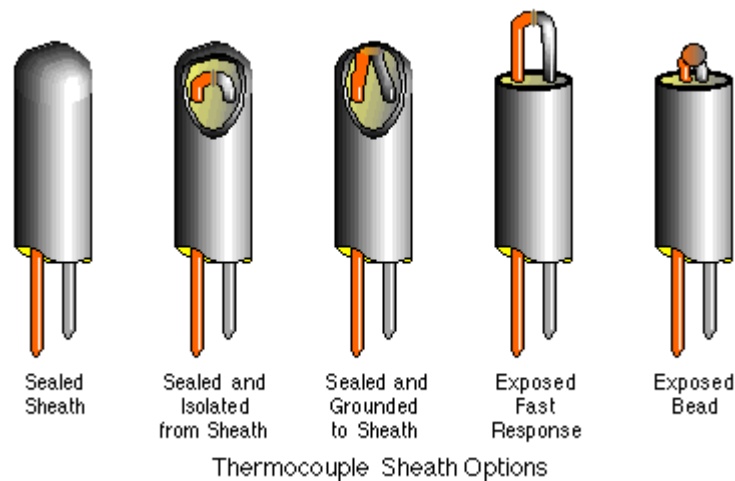
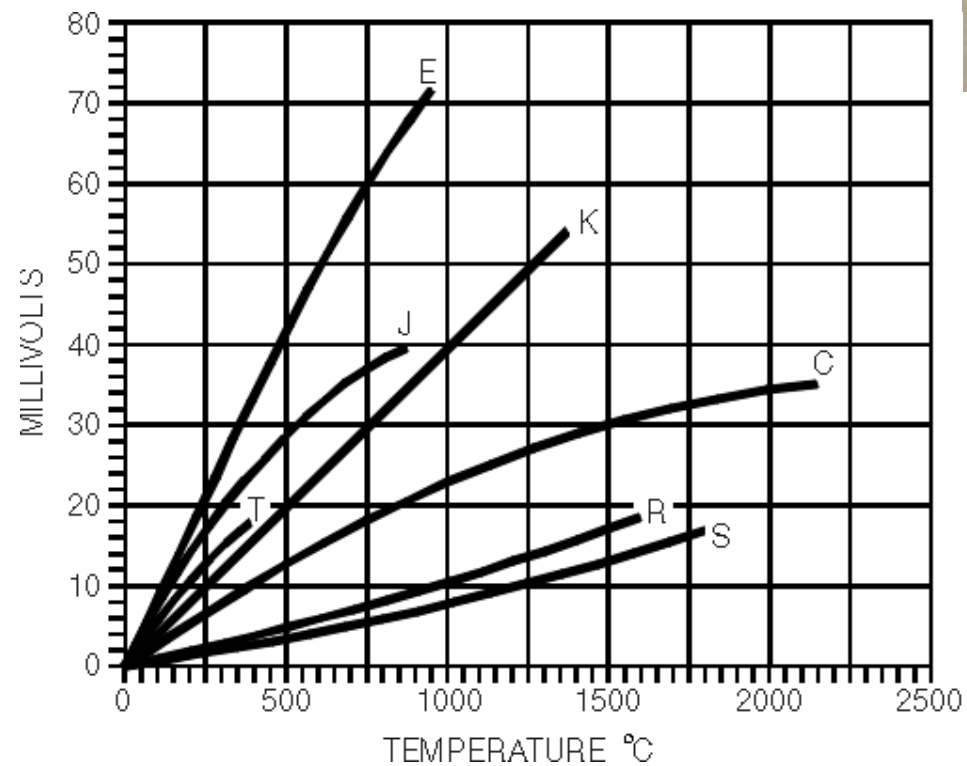
# III.2.3 Secondary Thermometers

- Thermocouples

Thermocouple Type	Names of Materials	Useful Application Range
<b>B</b>	Platinum30% Rhodium (+) Platinum 6% Rhodium (-)	2500 -3100F 1370-1700°C
<b>C</b>	W5Re Tungsten 5% Rhenium (+) W26Re Tungsten 26% Rhenium (-)	3000-4200F 1650-2315°C
<b>E</b>	Chromel (+) Constantan (-)	200-1650F 95-900°C
<b>J</b>	Iron (+) Constantan (-)	200-1400F 95-760°C
<b>K</b>	Chromel (+) Alumel (-)	200-2300F 95-1260°C
<b>N</b>	Nicrosil (+) Nisil (-)	1200-2300F 650-1260°C
<b>R</b>	Platinum 13% Rhodium (+) Platinum (-)	1600-2640F 870-1450C
<b>S</b>	Platinum 10% Rhodium (+) Platinum (-)	1800-2640F 980-1450°C
<b>T</b>	Copper (+) Constantan (-)	-330-660F -200-350°C

# III.2.3 Secondary Thermometers

- Thermocouples

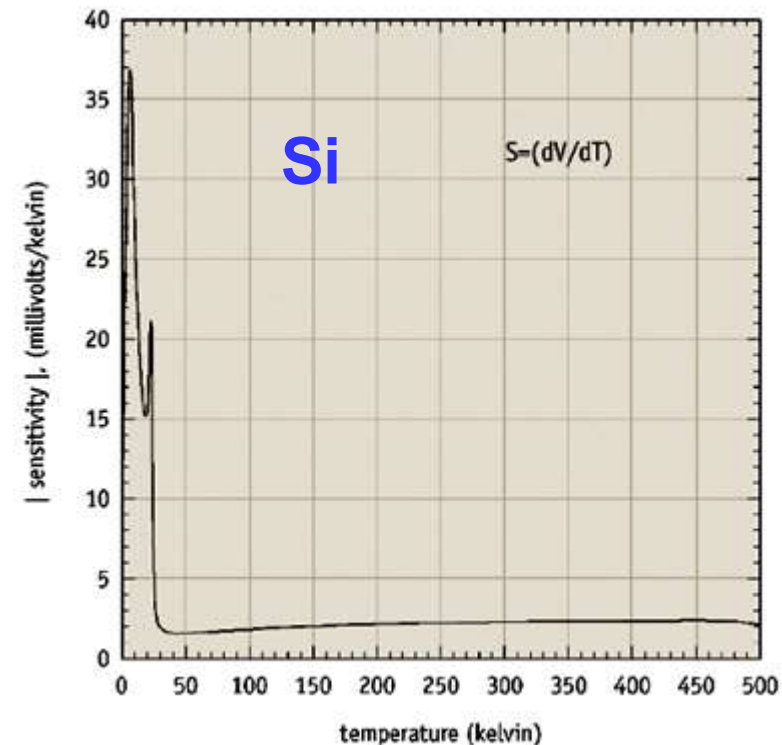
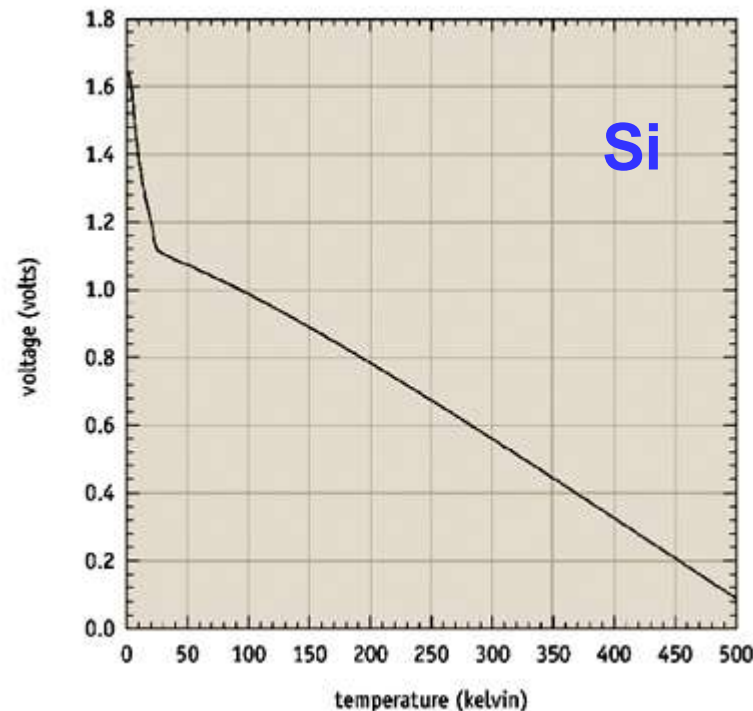


# III.2.3 Secondary Thermometers

- Diode Thermometers

The temperature-indicating parameter is the forward-biased junction voltage, which decreases approximately linearly with increasing temperature when the current is kept constant, since  $I \propto \exp(eV/k_B T)$

- The typical I-V characteristic is such as to make the internal impedance of the device very high (easily greater than 100 kΩ) at small currents; or else - using a larger current - one encounters unacceptably high power dissipation at low temperatures.
- There is a transition region in the conduction mechanism around 20 K that makes fitting a V-T characteristic over the whole temperature range difficult for GaAs and impossible for Si



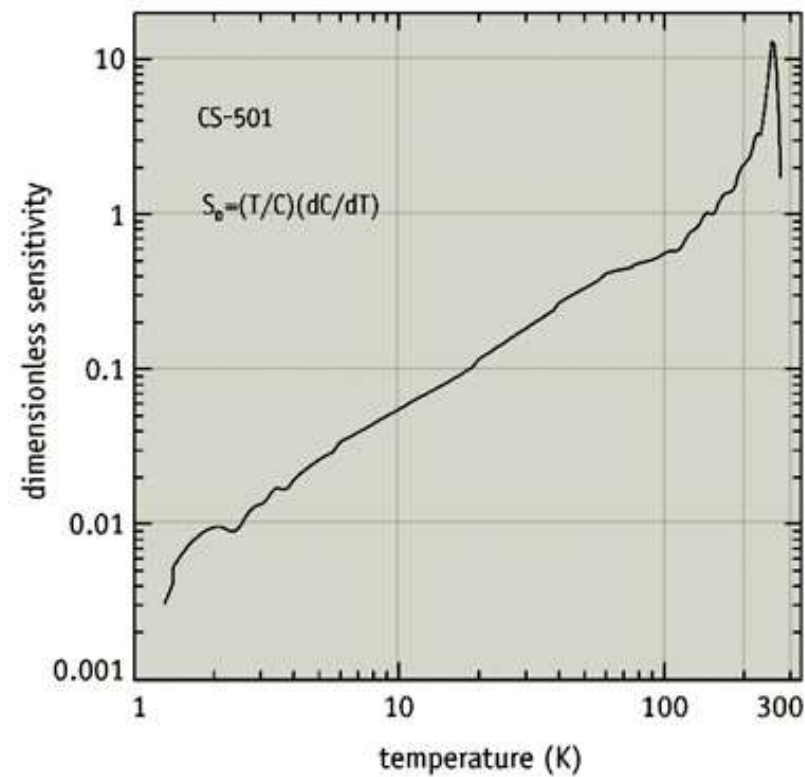
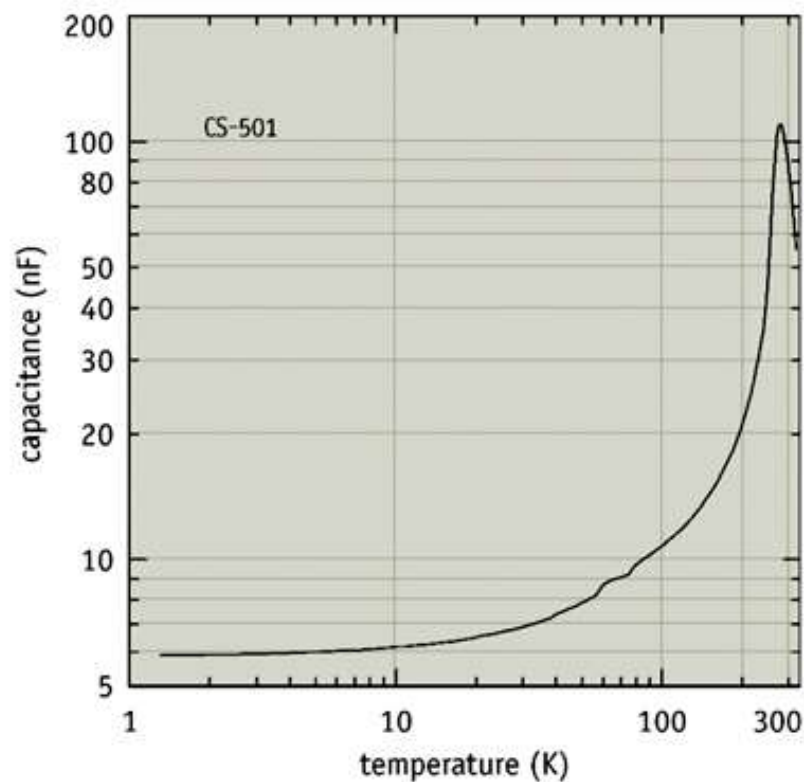
Source: Lake Shore Cryotronics, Inc.

## III.2.3 Secondary Thermometers

- **Capacitive Thermometers**
  - based on the well defined relation between the dielectric constant and temperature
  - temperature is determined via a capacitance measurement
  - advantage: virtually no magnetic field dependence

# III.2.3 Secondary Thermometers

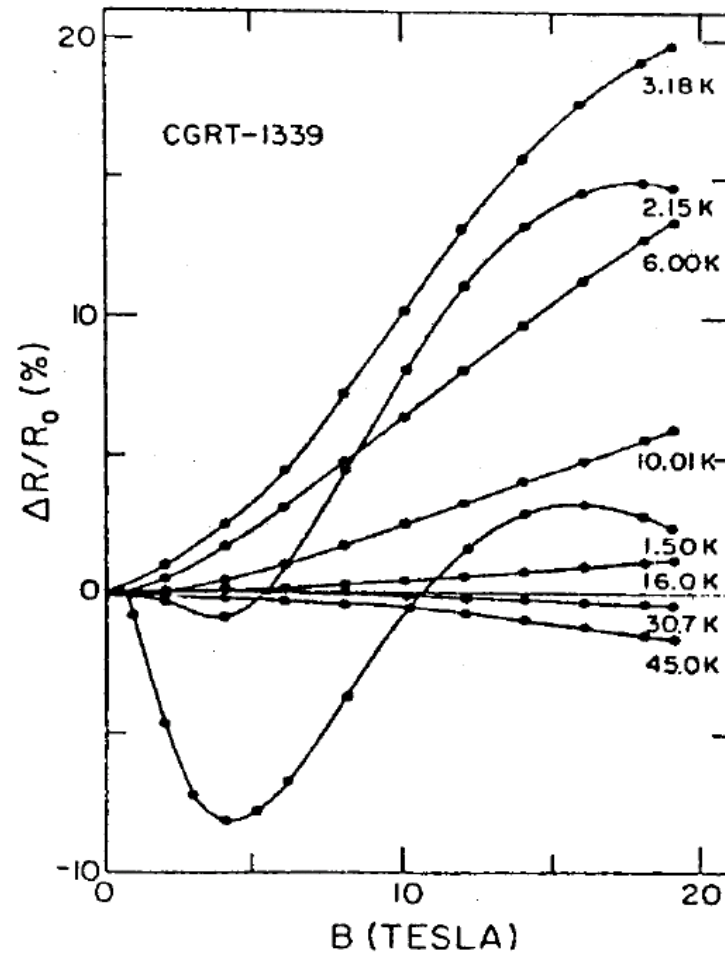
- Capacitive Thermometers



Source: Lake Shore Cryotronics, Inc.

# III.2.3 Secondary Thermometers

- magnetic field effects



Type of Sensor	T(K)	Magnitude of relative temperature error $ \Delta T/T $ (%) for values of B					Notes	References
		1 T	Magnetic Flux Density, B			19 T		
Carbon radio resistors Allen-Bradley (2.7, 3.9, 5.6, 10 C)	0.5		2-4	5-13	7-20		a	Sample and Rubin (1977)
	1.0		2-4	6-15	9-25			
	2.5		1-5	6-18	10-30			
	4.2		1-5	5-20	10-35			
Allen-Bradley (47, 100, 220 $\Omega$ )	4.2		<1	5	10		a	ibidem
	10		<1	3	5			
	20		<1	1	2			
Speer, Grade 1002 (100, 220, 470 C)	0.5		0-2	0-1	0-6		b	ibidem
	1.0		1-2	2-4	3-9			
	2.5		3-5	1-4	7-14			
	4.2		4-9	2-5	4-13			
Matsushita (68, 200, 510 C)	1.5		1-2	10-15			c	ibidem
	2.1		1	10-15				
	4.2		2-3	4-8				
KVM carbon composite resistors	2.4		3	8 (5T)			d	Astrov et al. (1977)
	4.2		1.5	5				
	10		0.4	1.4				
	20		0.1	0.4				
	80		<0.01	<0.01				
Carbon-Glass Resistors	2.2		0.1	1.5	3	4	e	Rubin and Brandt (1986)
	4.2		0.5	2	5	7		
	10		0.2	1.1	3	4		
	20		<0.01	0.02	0.03	0.13		
	45		0.07	0.5	1.3	2		
	88		0.06	0.5	1.3	2		
	190		0.04	0.3	1.0	1.7		
Thermistors	310		<0.01	0.2	0.6	1.1	f	Sample and Rubin (1977)
	4.2		<0.05	1	3			
	10		<0.05	0.3	1			
	20		<0.05	0.1	0.5			
	40		<0.05	0.1	0.5			
Germanium Resistors	60		<0.05	0.1	0.3		g	ibidem
	2.0		8-10	60				
	4.2		5-20	30-55	60-70			
	10		4-15	25-60	60-75			
	20		3-20	15-35	50-80			
Germanium Resistors TSG-2	70		3-10	15-30	25-50		g	Astrov et al. (1977)
	4.2		30	120				
	20		2.5	6				
Specially doped Ge resistors KG	4.2		<0.2	0.5 (6T)			h	ibidem; Matarotta et al. (1984)
	10		<0.2	<0.5				
	20		<0.5	2-3				
	30		<0.5	5				
	80		0.15	0.5				
Platinum Resistors	10		100	250			i	Pavese & Cresto (1984); Neuringer et al. (1971); Rubin & Brandt (1986)
	20		2-8	25	100	250		
	40		0.5	<1	3	5		
	68		0.1	<0.5	0.8	2		
	87		0.04	<0.5	0.4	1		
	110		0.02		0.2	0.6		
	190		<0.01		0.06	0.2		
	300		<0.01		0.02	0.07		
			(a)	(b)	(a)	(b)		
			(a)	(b)	(a)	(b)		
			(a)	(b)	(a)	(b)		



# III.2.3 Secondary Thermometers

- magnetic field effects

Type of Sensor	T(K)	Magnetic Flux Density, B					Notes	References
		1 T	2.5 T	8 T	14 T	19 T		
Rhodium-Iron resistors	2.0		22				j	Pavese & Cresto (1984); Rusby (1972); Rubin & Brandt (1986)
	4.2	2	11	40(8T)				
	20	0.8	4	10(5T)				
	40		1.5	12	30	40		
	66		0.3	2.5	6	9		
	87		0.2	1.5	4	8		
	110		0.1	0.9	2.4			
	190		0.03	0.3	0.9			
	300		<0.01	0.1	0.4			
Platinum-Cobalt resistors	2	25	30				k	Shiratori et al. (1982); Pavese & Cresto (1984)
	4.2	8	3	40 (5T)				
	10	1	<0.1	12 "				
	20	0.2	1	3.5 "				
	30	0.2	0.3	1.5 "				
Cryogenic linear resistance sensor	4	20	250				l	McDonald (1973)
	10	17	100					
	20	8	50					
	30	5	30					
KELTIP resistors (Au/Mn)	4.2		4	13	20		m	Rubin & Brandt (1986)
	40		2	30	70	110		
	66		0.4	4	12	20		
	87		0.15	1.5	5	10		
	110		0.03	0.25	1			
	190		0.02	0.2	0.5			
	300		0.02	0.1	0.4			
SrTiO <sub>3</sub> capacitors	2.2		<0.02	<0.02	0.02		n	ibidem
	4.2		<0.01	<0.01	0.01			
	20		<0.05	<0.05	<0.05			
	50		<0.05	<0.05	<0.05			
	88		<0.01	<0.01	<0.01	<0.01		
	110		<0.01	<0.01	<0.01	<0.01		
	190		<0.01	<0.01	<0.01	<0.01		
Si Diodes	4.2		75				o	Sample & Rubin (1977)
	10		20	30	50			
	20		4	7	10			
	30		3	4	5			
	77		0.2	0.5	0.5			
GaAs Diodes	4.2		2-3	30-50	100-250		p	ibidem
	10		1.5-2	25-40	75-200			
	20		0.5-1	20-30	60-150			
	40		0.2-0.3	4-6	15-30			
	80		0.1-0.2	0.5-1	2-5			
Au + 0.07 % Fe/ Chromel P thermocouple	5		2	10	15		q	Sample et al. (1974)
	10		3	20	30			
	20		2	15	20			
	45		1	5	7			
	100		0.1	0.8				
Chromel P/Constantan thermocouple (Type E)	10		1	3	7		r	ibidem
	20		<1	2	4			
	45		<1	<1	2			
Cu + 0.01 % Fe/Cu thermocouple	5		2	3.5 (5T)			q,f	Astrov et al. (1977)
	10		0.8	2 "				
	20		0.6	1.5 "				
	50		0.3	0.6 "				
Vapour pressure thermometers	no intrinsic error except with O <sub>2</sub>						s	
Helium gas thermometer	no intrinsic error						s	Van Degrift et al. (1980)

# III.2.3 Secondary Thermometers

Table 1.1: Summary of Some Properties of Most-Commonly-Used Thermometers

Thermometer	Usual Temperature Range	Thermometric Quantity	Typical Uncertainty
Germanium	1 K to 100 K	elec. resist.*	$\Delta T / T < 2 \times 10^{-4}$
Rhodium-Iron	0.5 K to 30 K	elec. resist.*	0.3 mK
Platinum-Cobalt (industrial type)	2 K to 20 K	elec. resist.*	10 mK
Carbon	0.5 K to 30 K	elec. resist.*	$\Delta T / T < 5 \times 10^{-3}$
Carbon-glass	0.5 K to 100 K	elec. resist.*	$\Delta T / T < 1 \times 10^{-3}$
Diode	4 K to 300 K	junction voltage	-50 mK
Vapour-pressure	various subranges between 0.5 K and 100 K	pressure	-1 mK
Mercury-in-glass	-50 °C to 250 °C	thermal expansion of mercury	0.1 K
Thermocouples	4 K to 2500 °C	thermoelectromotive force	
Type S	-50 °C to 1600 °C		0.3 K < 1000 °C 1 K > 1000 °C
Type R	-50 °C to 1600 °C		0.3 K < 1000 °C 1 K > 1000 °C
Type B	300 °C to 1800 °C		0.5 K to 2 K
Type T	-200 °C to 350 °C		0.1 K
Type E	-200 °C to 870 °C		0.1 K < 300 K 1 K > 300 K
Type J	0 °C to 760 °C		0.5 K < 300 °C 2 K > 300 °C
Type K	-200 °C to 1260 °C		0.1 K < 200 °C 1 K 200-1000 °C 3 K > 1000 °C
Type N	0 °C to 1300 °C		0.1 K < 200 °C 0.5 K 200-1000 °C 3 K > 1000 °C
W/Re	1000 °C to 2400 °C		3-10 K

## III.2.3 Secondary Thermometers

Thermometer	Usual Temperature Range	Thermometric Quantity	Typical Uncertainty
Thermistor	-80 °C to 250 °C	elec. resist.*	0.1 K (much better if use confined to very small temperature range)
Platinum: SPRT IPRT	14 K to 630 °C 20 K to 600 °C	elec. resist.*	0.5 mK 50 mK
Radiation	100 °C to 3000 °C	spectral radiance of source	1 K < 1000 °C 5 K > 1000 °C

# III.2.3 Secondary Thermometers

- **Magnetic Susceptibility Thermometers**

- Curie-Weiss law:  $\chi = \frac{C}{T - T_C}$

- measure  $\chi$  as a function of  $T$ , calibration requires determination of  $C$  and  $T_C$

- experimental techniques:

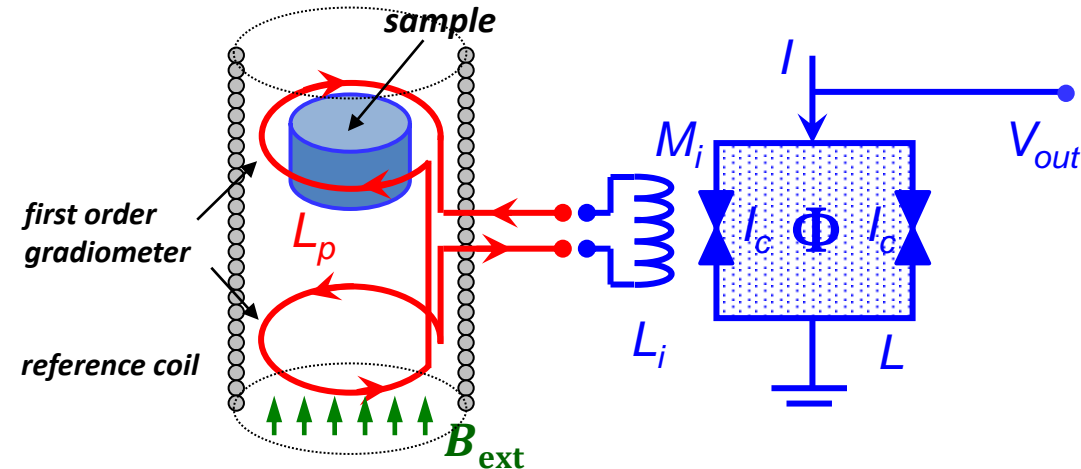
- **inductance bridge**: compare inductance of coil containing the magnetic material to empty reference coil

- **SQUID magnetometer**:

resolution:

→  $\Delta T/T \approx 10^{-10}$  @ 1 K

→ increases  $\approx 1/T$



T.C.P. Chui et al., Phys. Rev. Lett. **69**, 3005 (1992)

# III.2.3 Secondary Thermometers

- **Magnetic Susceptibility Thermometers**

- materials (use of electronic magnetic susceptibility):

→ paramagnetic salts:

e.g. Ce-Mg-nitrate (CMN)

++ very low ordering temperature:  $T_c \sim 2$  mK

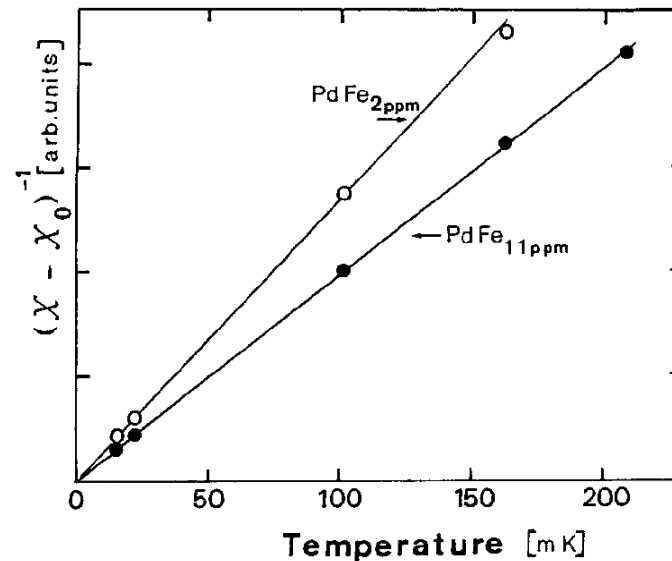
++ large Curie constant

-- long time constant at low  $T$  ( $\sim 100$  s)

-- cannot be used in vacuum (instable)

→ nonmagnetic metals:

e.g. PdFe or AuEr



- usable down to about 0.3 mK

- response time: 1 s @ 10 mK

M. Jutzler, B. Schröder, K. Gloos, F. Pobell,  
Z. Phys. B64, 115 (1986)

## III.2.3 Secondary Thermometers

- Magnetic Susceptibility Thermometers

- materials (use of nuclear susceptibility):

- use of nonmagnetic metals: e.g. Cu

- - nuclear moments much smaller → sensitive SQUID magnetometer

- - perturbing magnetic impurities → very pure materials

- (e.g. 1 ppm Fe in Cu gives similar signal as all Cu nuclear spins)*

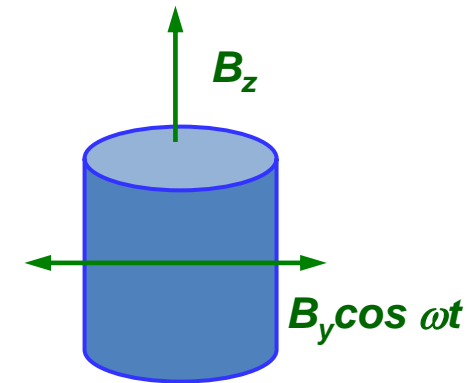
- ++ can be used below 1 mK



# III.2.3 Secondary Thermometers

- Nuclear Magnetic Resonance Thermometers

- are not based on static orientation of nuclear moments as in susceptibility thermometers
- have selectivity to specific nuclear moments (less sensitive to magnetic impurities)
- usable at temperatures below 1 mK
- materials: predominantly  $^{195}\text{Pt}$
- experimental techniques: stationary and pulsed NMR



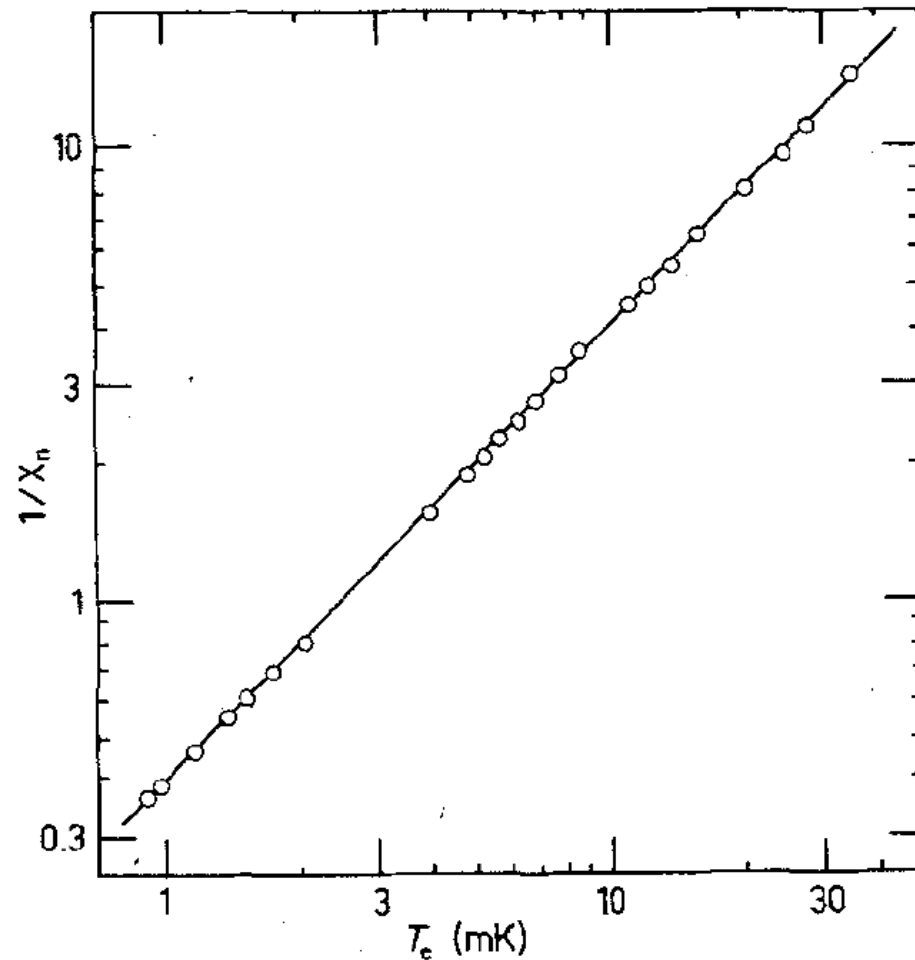
→ measures resonance absorption of high frequency signal  
 → allows to determine induced  $M_y \propto M_0(T) \propto 1/TN$  at resonance  
 → requires small  $B_y \approx 1 \mu\text{T}$  to avoid saturation effects

→ measures decay of induced  $M_y(t)$  after  $90^\circ$  pulse  $\boxed{?}$   
 → amplitude  $dM_y(t = 0)$  or integral of decay curve  $\propto M_0(T) \propto 1/TN$

→ **general problem:** one measures temperature  $T_N$  of nuclear spins  
 → are they in thermal equilibrium with lattice ??

# III.2.3 Secondary Thermometers

- Nuclear Magnetic Resonance Thermometers



comparison of the Curie and Korringa temperatures of a Pt sample:

the inverse nuclear susceptibility  $1/\chi_N$  (*arb. units*) is plotted against the electron temperature

$$T_e = \left( \frac{1}{t_1} \right) \cdot 29.9 \times 10^{-3} \text{ K sec.}$$

A.I. Ahonen et al.,  
J. Low Temp. Phys. **25**, 421 (1976)