Vacuum and Cryoliquids

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Production and Measurement of Low Pressure

Pumping

pressure ranges
pluming
pumps

Measuring Pressure

mechanical
thermal conductivity
viscocity
ionisation

Vacuum flansh

feed throughs
seals
leaks
leak detection
diffusion
outgasing

Units

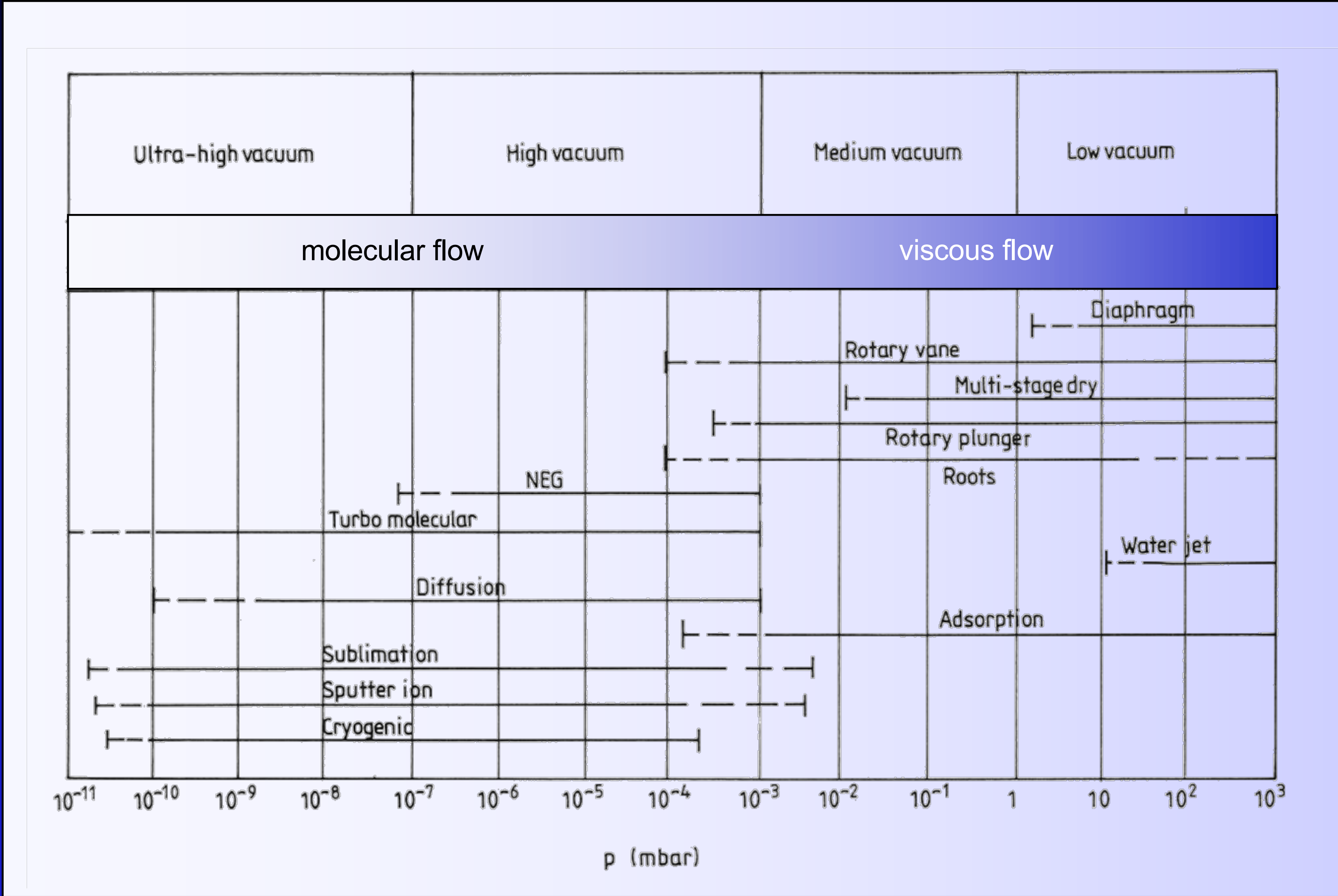
$1\ N/m^2 = 1\ Pa = 10^{-5}\ bar$

1 Torr	= 4/3 mbar	
1 dyn/cm²	$= 10^{-5}\ N/cm^2 = 0.1\ Pa$	
1 atm.	= 760 Torr	physical atmosphere
1 at.	$= 1\ kp/cm^2 = 0.981\ bar$	technical atmosphere
1 psi	$= lb/in^2$	
1 μ	$= 10^{-3}$ Torr	

Mean Free Path Depending on Pressure (Ideal Gas Law)

Vacuum range	*p* [mbar]	Molecules / cm^3	mean free path
Ambient pressure	1013	2.7×10^{19}..	68 nm
Low vacuum	300 … 1	10^{19}… 10^{16}	0.1 ... 100 µm
Medium vacuum	1 … 10^{-3}	10^{16}… 10^{13}	0.1 ... 100 mm
High vacuum	10^{-3}… 10^{-7}	10^{13}… 10^{9}	10 cm ... 1 km
Ultra high vacuum	10^{-7}… 10^{-12}	10^{9}… 10^{4}	1 km ... 10^{5} km
Extremely high vacuum	$< 10^{-12}$	$<10^{4}$	$> 10^{5}$ km

Vacuum Pumps and Pressure Ranges



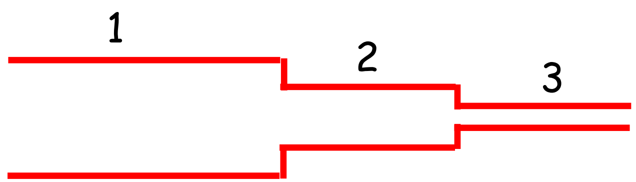
Pumping Lines

throughput

$$q = p\frac{\mathrm{d}V}{\mathrm{d}t} \quad \left[\frac{\mathrm{mbar}\ \ell}{\mathrm{s}}\right]$$

Flow resistance: $W = \dfrac{\Delta p}{q}$

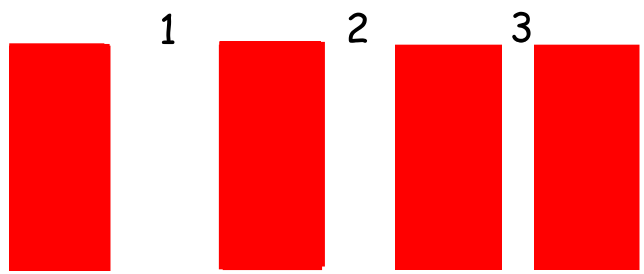
Flow conductance: $F = \dfrac{1}{W} = \dfrac{q}{\Delta p}$



serial

$$W = W_1 + W_2 + W_3 + \ldots$$

$$\frac{1}{F} = \frac{1}{F_1} + \frac{1}{F_2} + \frac{1}{F_3} + \ldots$$

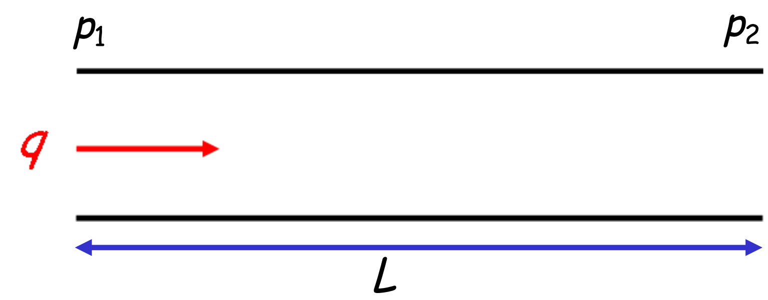


parallel

$$\frac{1}{W} = \frac{1}{W_1} + \frac{1}{W_2} + \frac{1}{W_3} + \ldots$$

$$F = F_1 + F_2 + F_3 + \ldots$$

Flow conductance



Viscous regime

$$q = p_\mathrm{a}\,\frac{\mathrm{d}V}{\mathrm{d}t} = p_\mathrm{a}\frac{\pi r^4}{16\eta L}\,(p_1 - p_2)$$

average pressure

viscosity

$$F = \frac{q}{\Delta p} = p_\mathrm{a}\frac{\pi r^4}{16\eta L}$$

Molecular flow (long tube, $L/r > 5$)

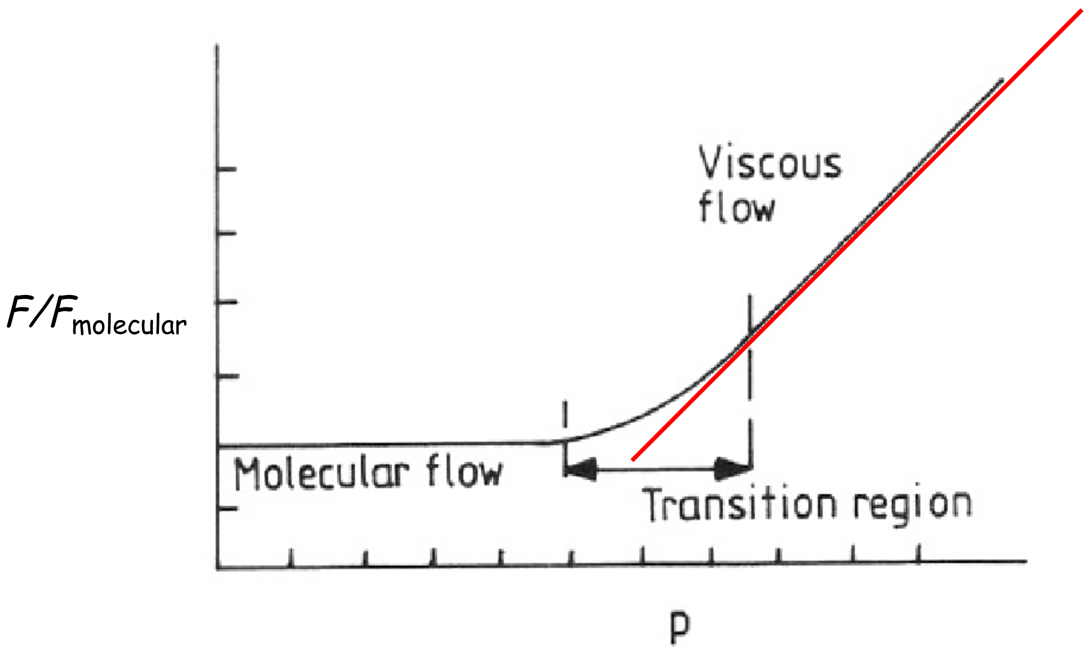
$$F_\mathrm{long} = \frac{4r^3}{3L}\sqrt{\frac{2\pi k_\mathrm{B} T}{m}}$$

short tube, $L/r < 5$

$$F_\mathrm{short} = \frac{3L}{8r} K F_\mathrm{long}$$

Clausius factor

Flow conductance



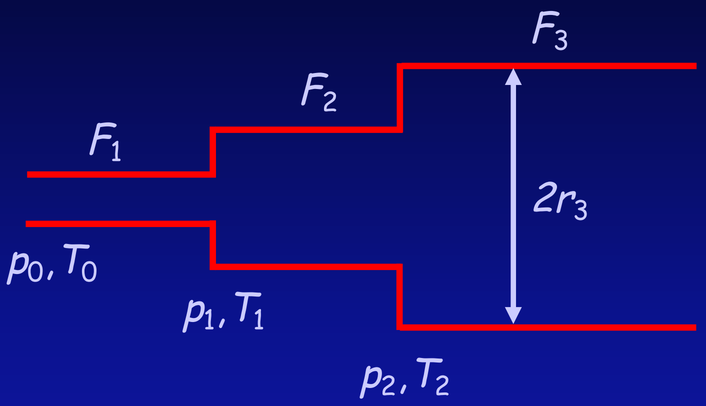
Transition region: $F = F_{\mathrm{viscous}} + \alpha\, F_{\mathrm{molecular}}$

$\alpha(T, p, \eta, r)$ = 0.81 … 1

$\alpha \approx 1$

Design of Pumping Systems

most cases: molecular flow



$$\frac{p_0}{\sqrt{T_0}} - \frac{p_1}{\sqrt{T_1}} = A\frac{L_1}{r_1^3}$$

$$\frac{p_1}{\sqrt{T_1}} - \frac{p_2}{\sqrt{T_2}} = A\frac{L_2}{r_2^3}$$

$$\frac{p_{n-1}}{\sqrt{T_{n-1}}} - \frac{p_n}{\sqrt{T_n}} = A\frac{L_n}{r_n^3}$$

$$A = \frac{3}{4}q_{\mathrm{m}}\sqrt{\frac{k_{\mathrm{B}}}{2\pi m}}$$

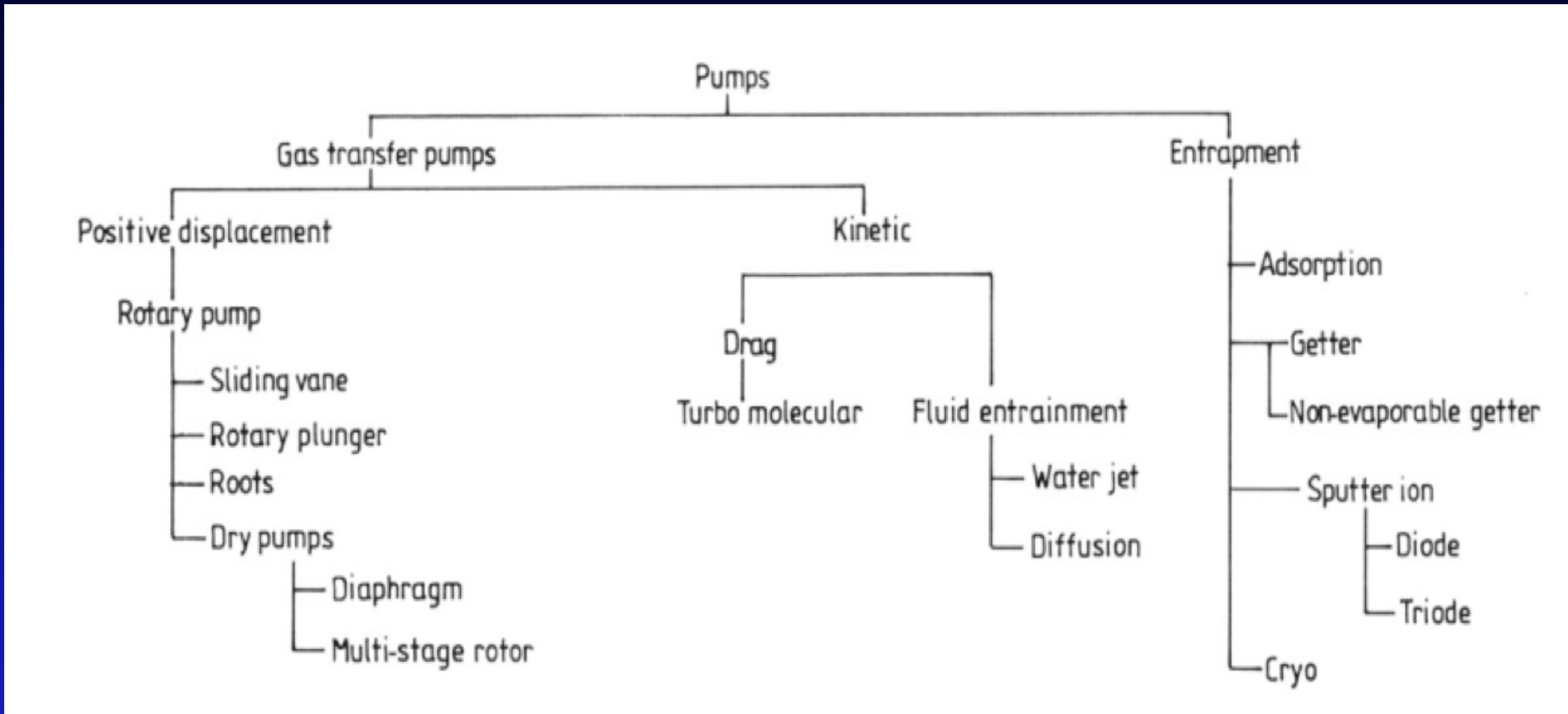
$$\frac{p_0}{\sqrt{T_0}} - \frac{p_n}{\sqrt{T_n}} = A\sum_{i=1}^{n}\frac{L_i}{r_i^3}$$

final segment at room temperature

$$\frac{p_0}{\sqrt{T_0}} \approx \frac{3}{4}q_{\mathrm{m}}\sqrt{\frac{k_{\mathrm{B}}}{2\pi m}}\sum_{i=1}^{n}\frac{L_i}{r_i^3}$$

if p_0, T_0, q_m are known, the design of the pumping system is straight forward

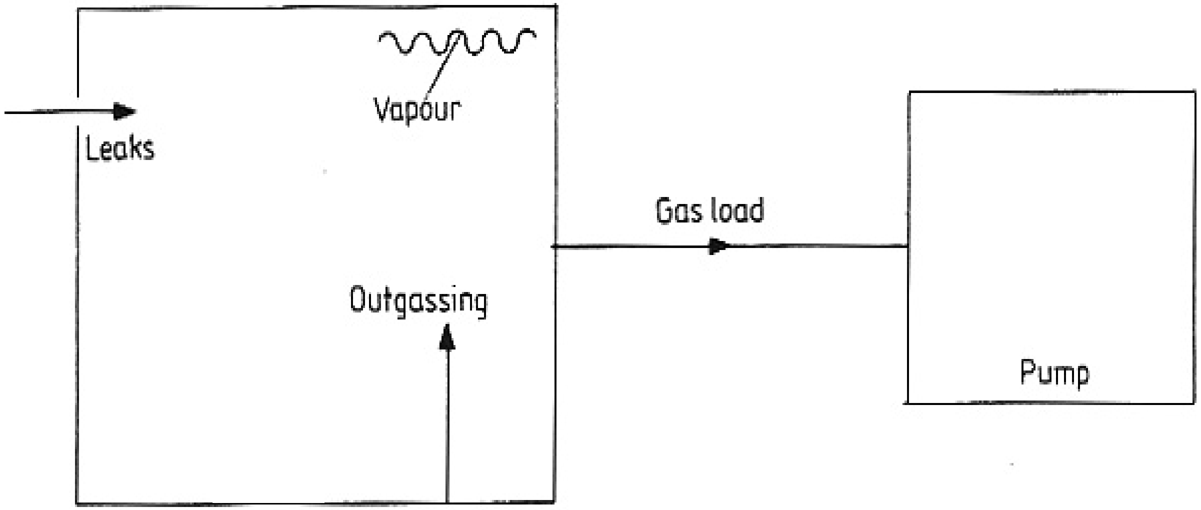
Types of Pumps

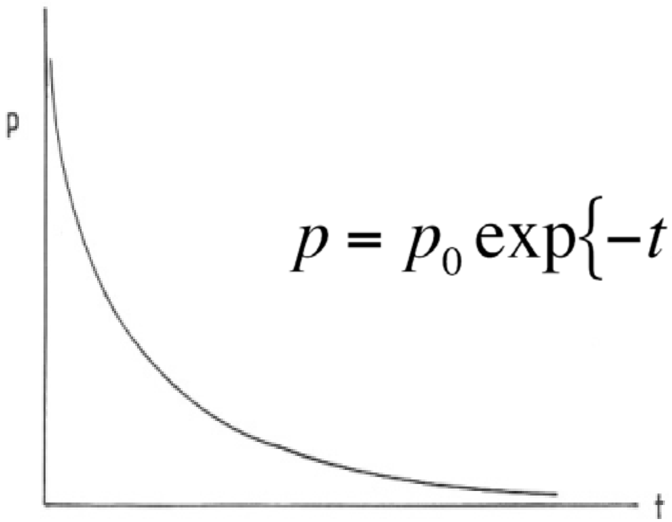


relevant parameters:

- pumping speed liters/second ℓ/s
- throughput
- minimal pressure
- operating range
- inlet pressure
- oil or dry

Pumping Process





$$p = p_0 \exp\{-t/\underbrace{(V/S)}_{\text{pumping time constant}}\}$$

Gas Transfer Pumps

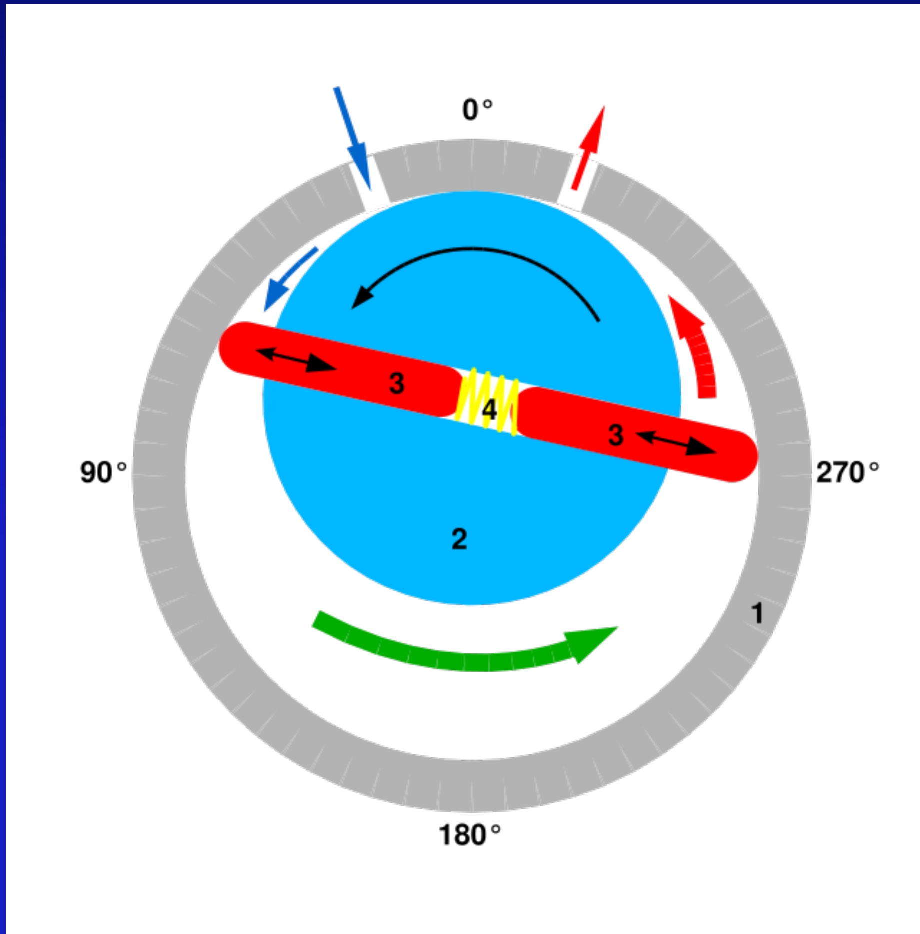
Rotary pumps
Scroll pumps

Roots pumps

Rotary Pumps

Principle: Displacement pump for viscous flow / Knudsen regime

- suction phase
- compression phase
- discharge phase



oil lubricated

low wear

ultimate pressure 10^{-4}-10^{-3} mbar

volume flow rate 1-100 m^3/h

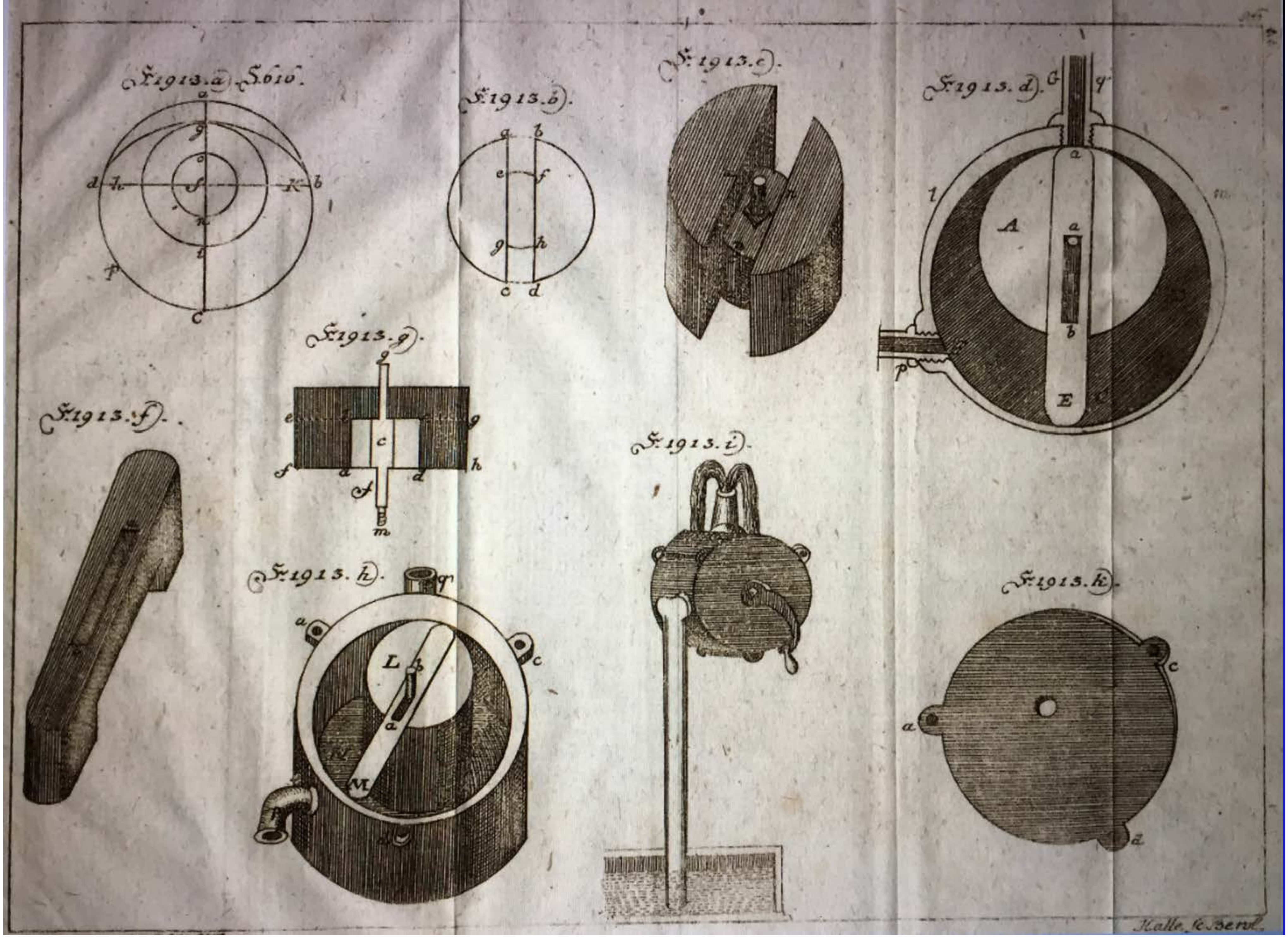
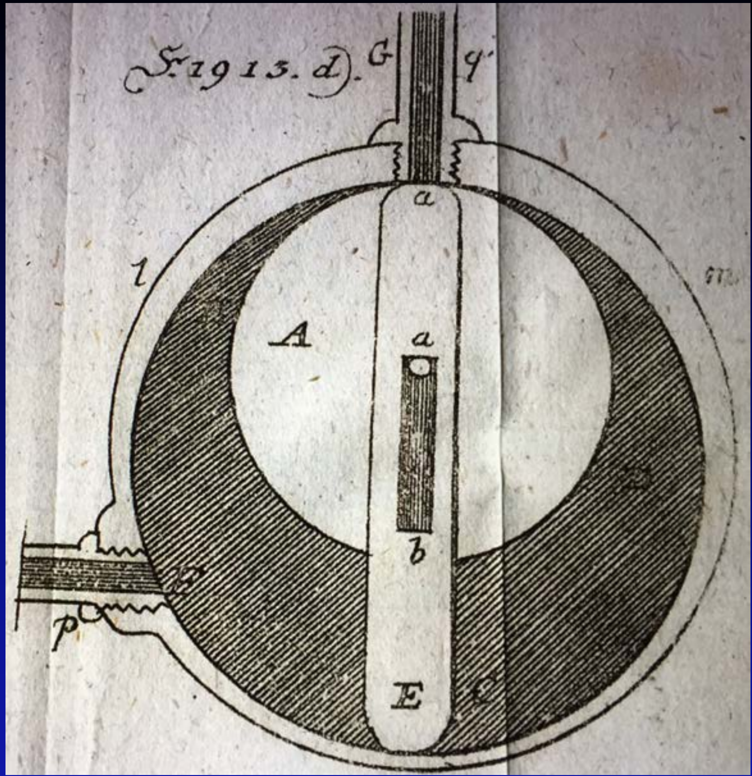


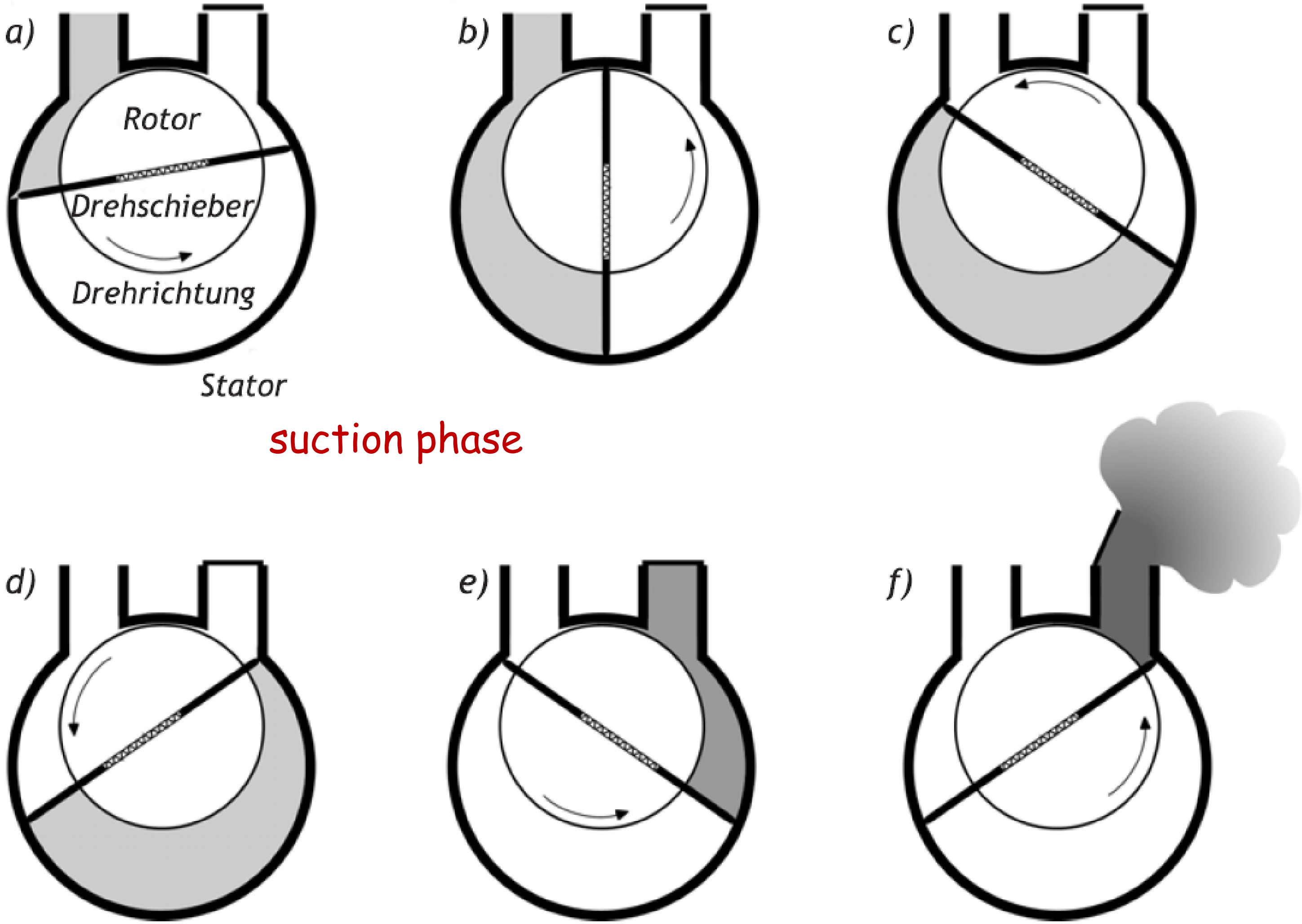
Fig. 1913. a)
Fig. 1913. b)
Fig. 1913. c)
Fig. 1913. d)
Fig. 1913. f)
Fig. 1913. g)
Fig. 1913. h)
Fig. 1913. i)
Fig. 1913. k)
Halle sc. Bern.



Ramelli vane pump

Invented by Agostino Ramelli 1588

Operating Principle of a Rotary Pump

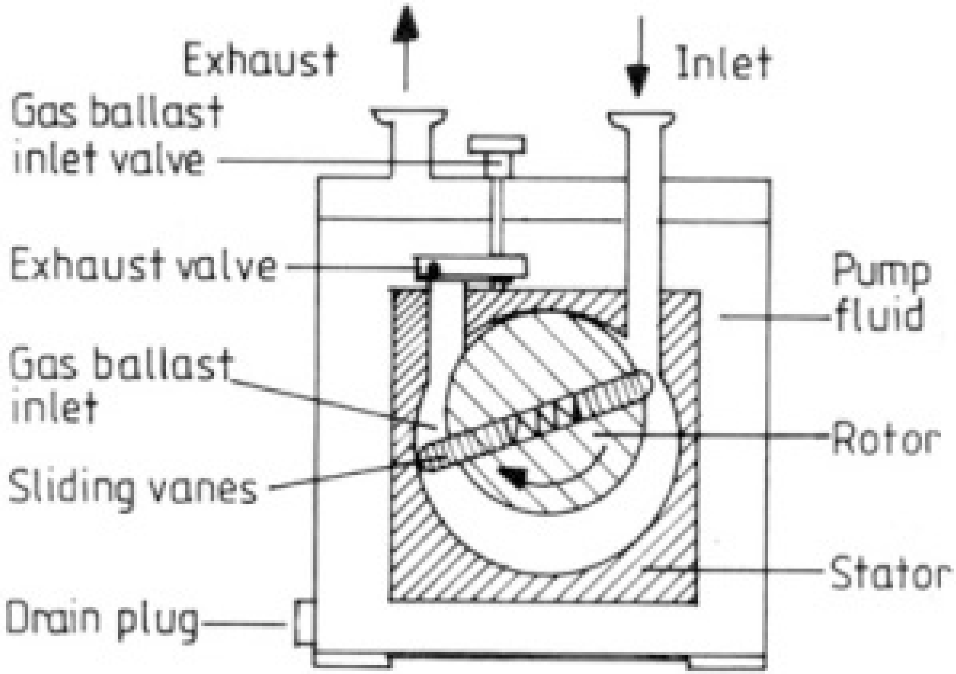


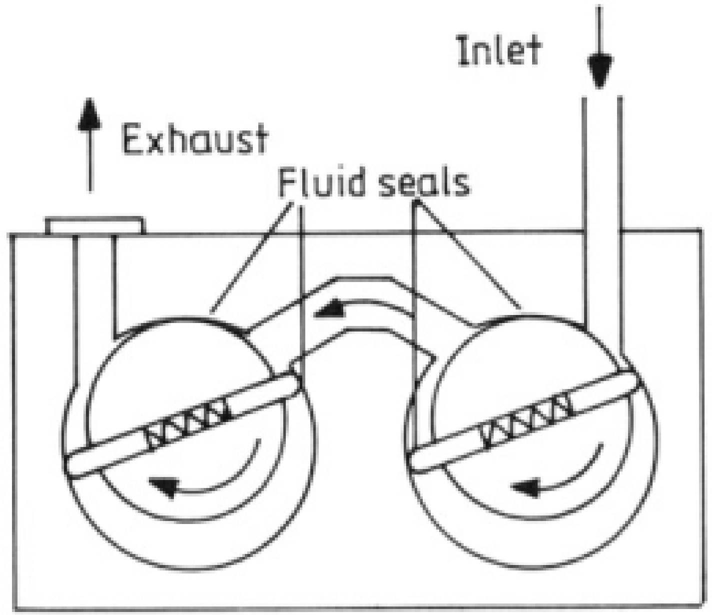
suction phase

compression phase

discharge phase

Rotary Pumps

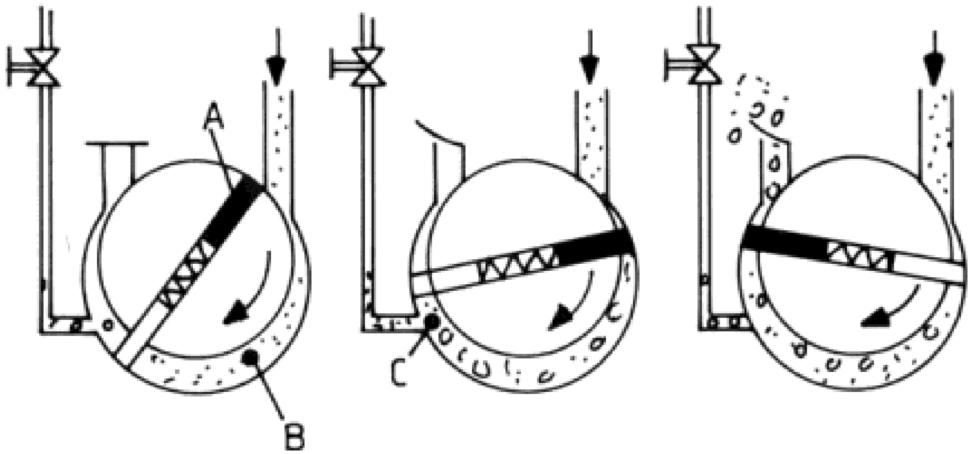


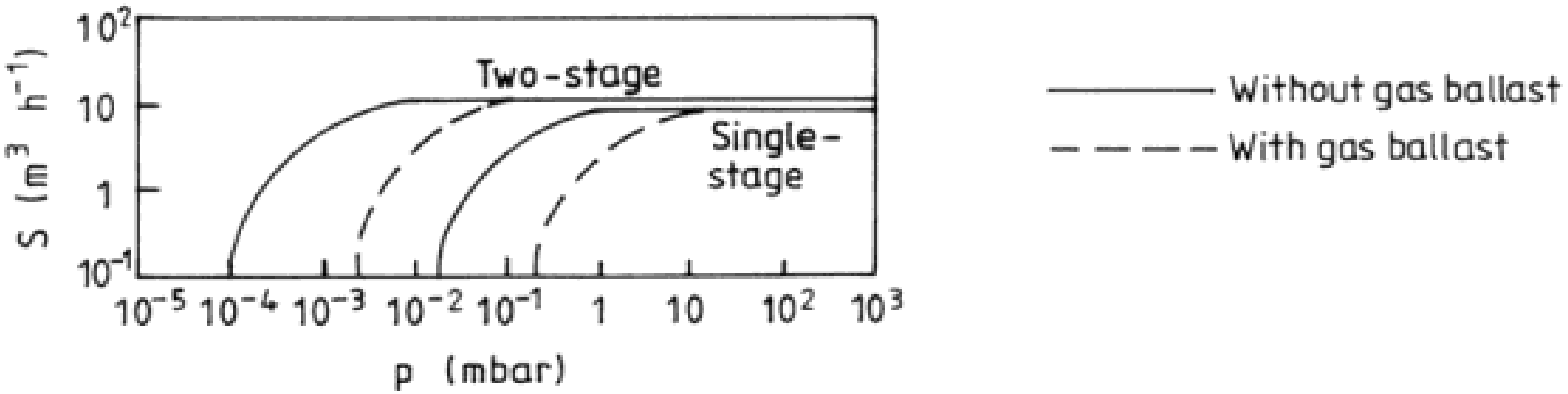


two stage rotary pump

Rotary Pumps With Gas Ballast

gas ballast is used when the evacuated vessel contains condensable vapours





Scroll Pumps

dry mechanical pump

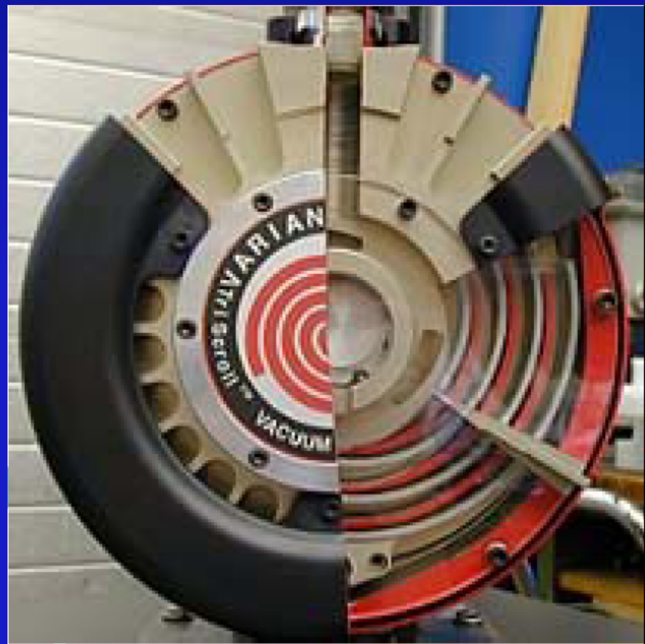
used as backing pump in dry pumping systems

oil-free

sliding PTFE seals, wear

volume flow rate 1-100 m^3/h

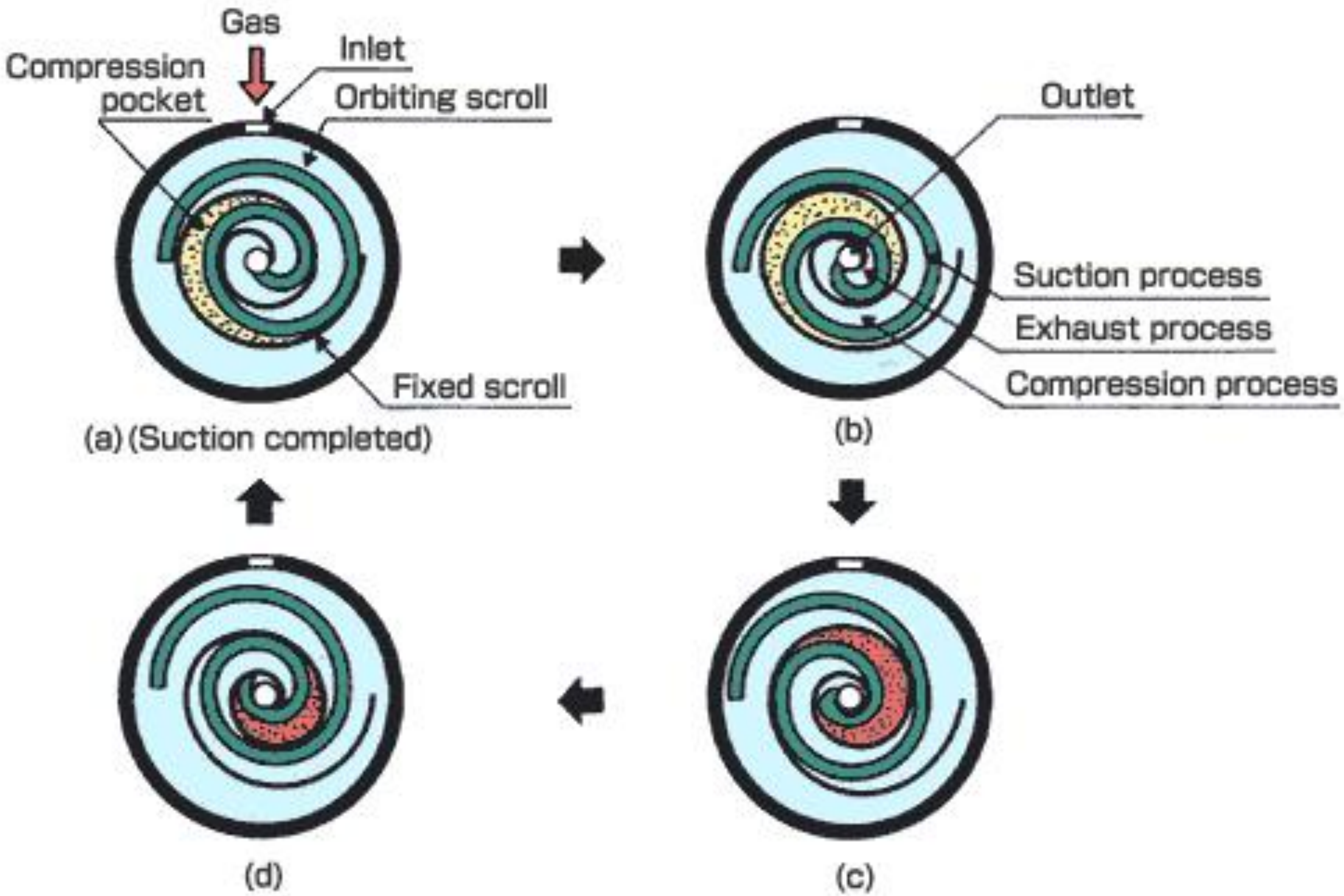
ultimate pressure 0.1-0.01 mbar



other application:

scroll compressor as charger for car engines (VW G-charger)

Scroll Pumps Operation



Root Pumps

Displacement pumps with large
large throughput ~ 250 torr ℓ/s

typical application: $^3He/^4He$-Kryostat

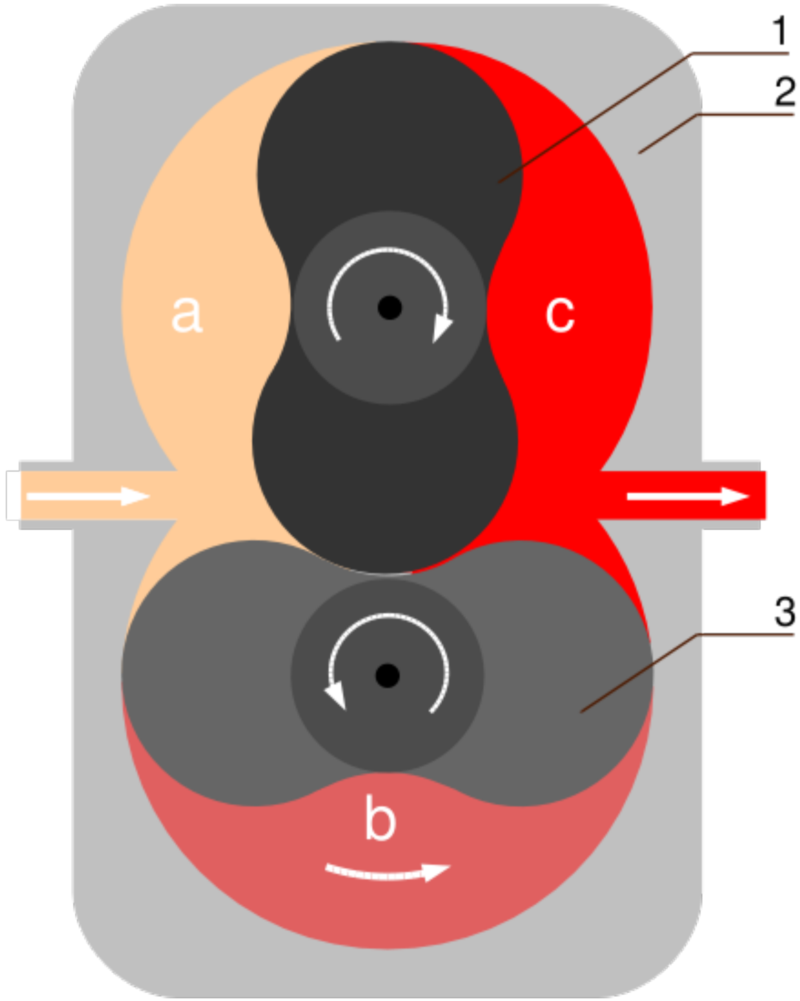
backing pump needed
inlet pressure <100 mbar!
thermal problem

no sealing fluid → dry
no sliding seals
(exception shaft feed trough if present)

compression ratio typically 10:1
volume flow rate 100-1000 m^3/h
ultimate pressure 10^{-4} mbar

potential trouble spots:
rotating seal
close mechanical tolerance 0.3 mm

two lobed rotors interlocked
and synchronised



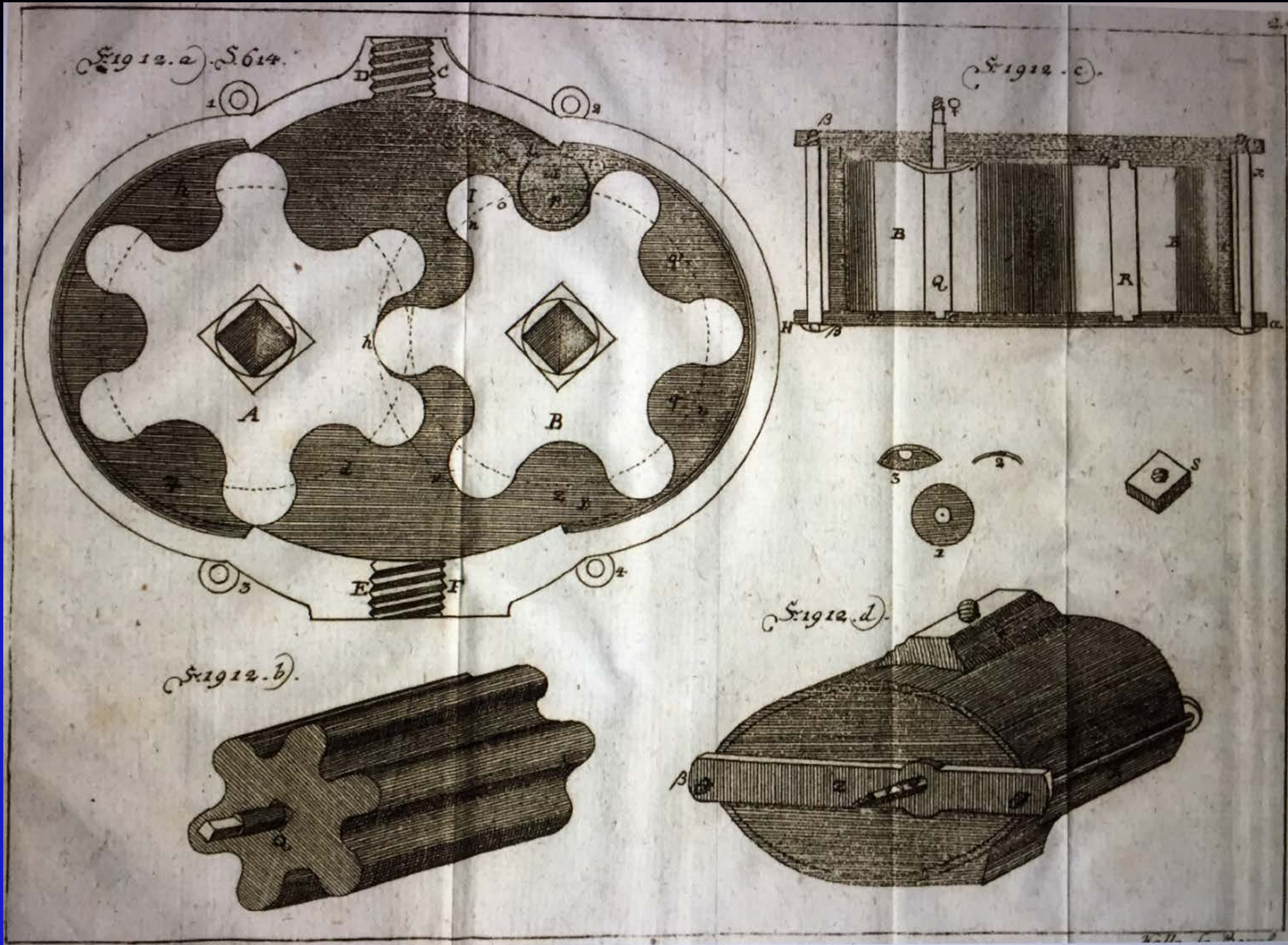
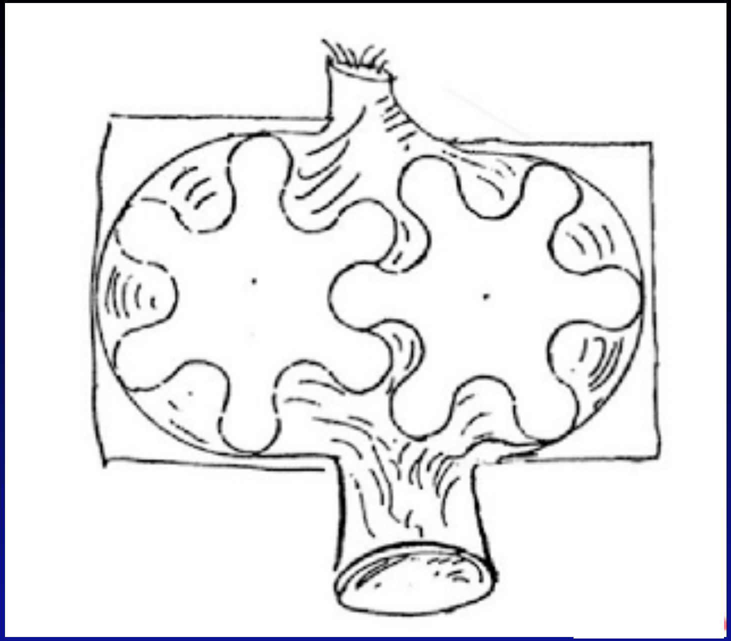
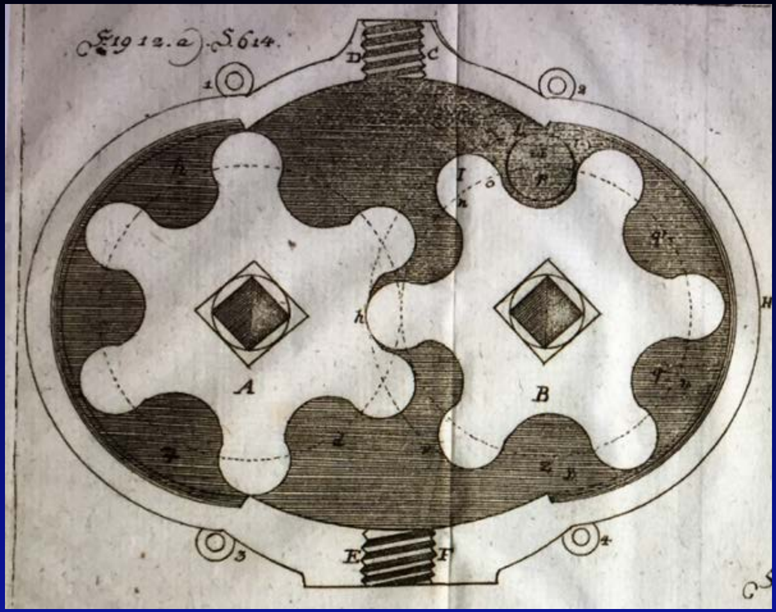


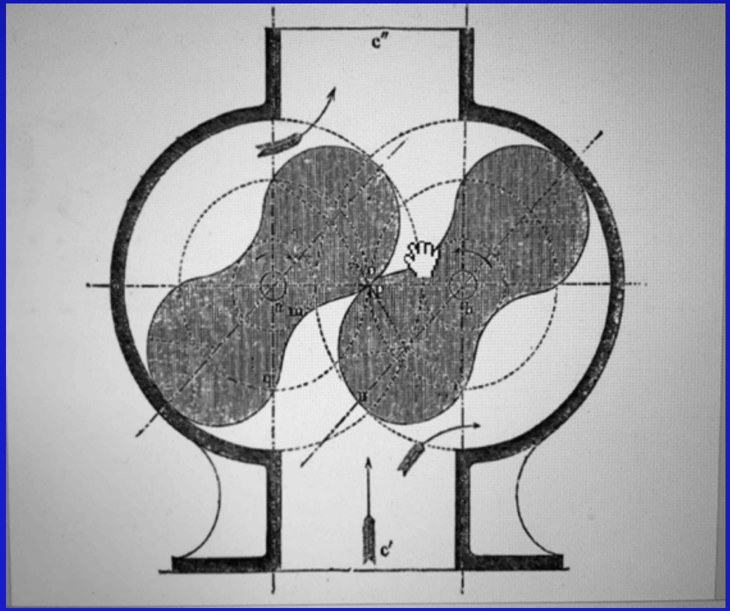
Fig. 1912. a). S. 614.
Fig. 1912. c).
Fig. 1912. b).
Fig. 1912. d).
A
B



Keplers gear pump 1600
First prototype 1604 by Jost Buergi



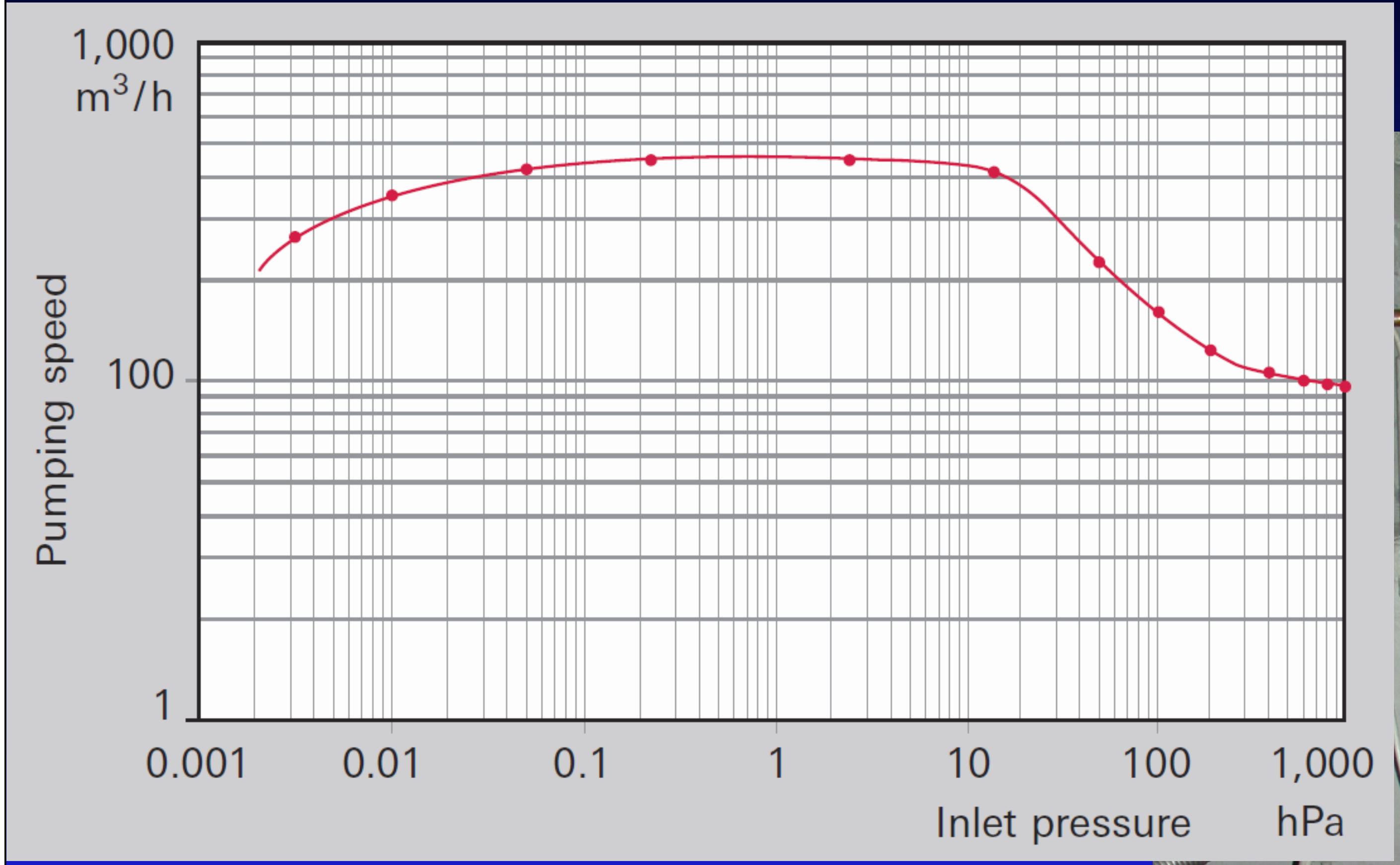
Papenheim Machine
designed around 1630

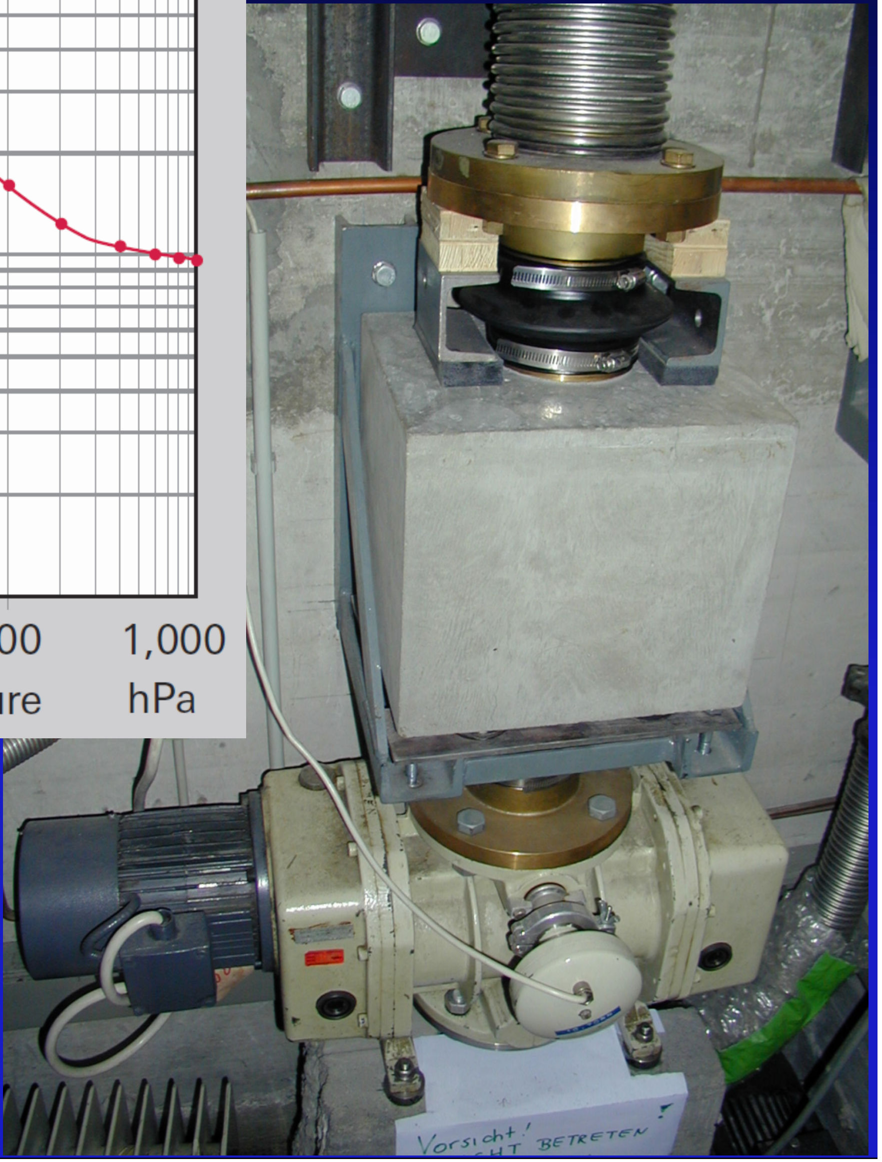


Rotary vacuum pump

around 1800, developed by Fabry

Pumping Speed of a Roots Pump





Kinetic Pumps:

Drag and Turbo Pumps

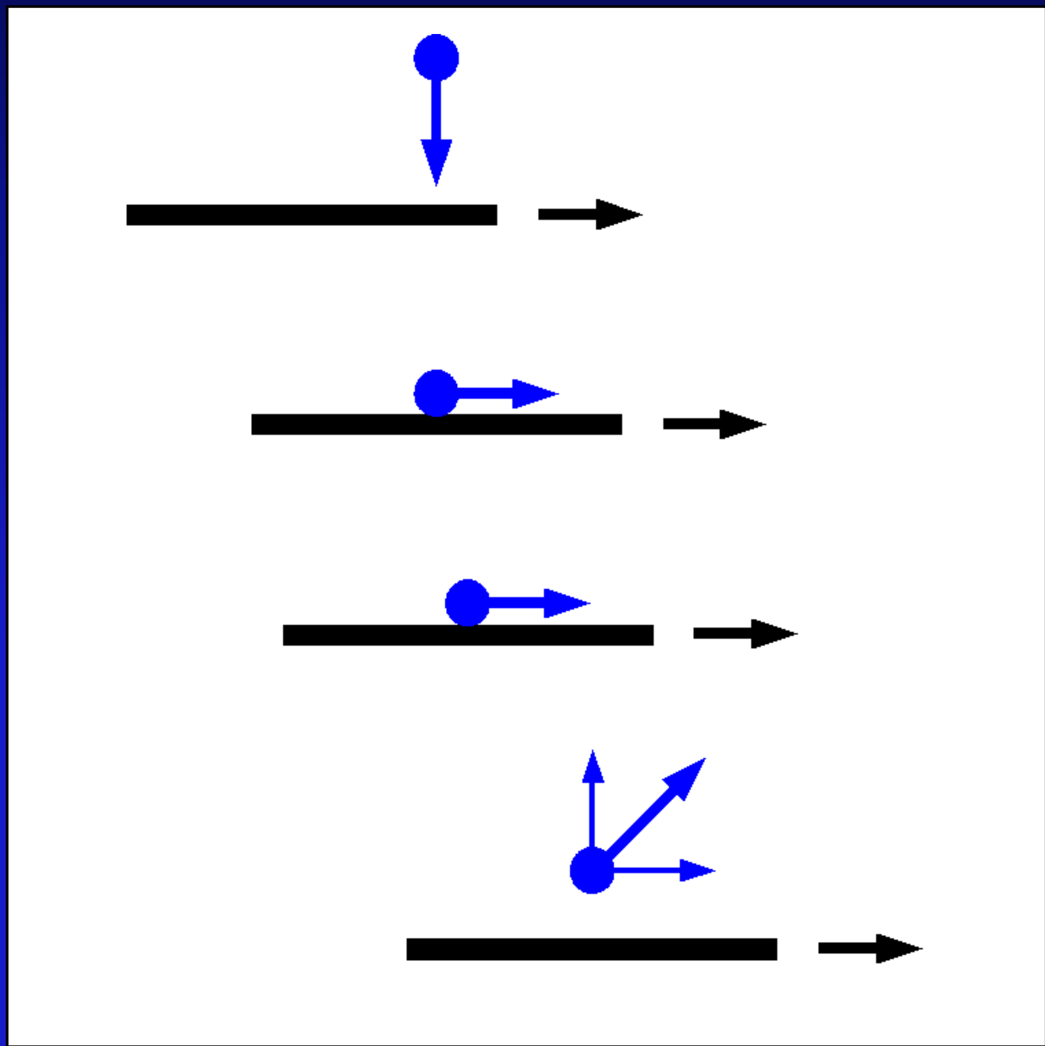
Oil Diffusion Pumps

Molecular Pumps: Drag and Turbo

Principle: Quickly moving wall, momentum transfer

molecular flow regime

needs backing pump



light gases (H_2, He)

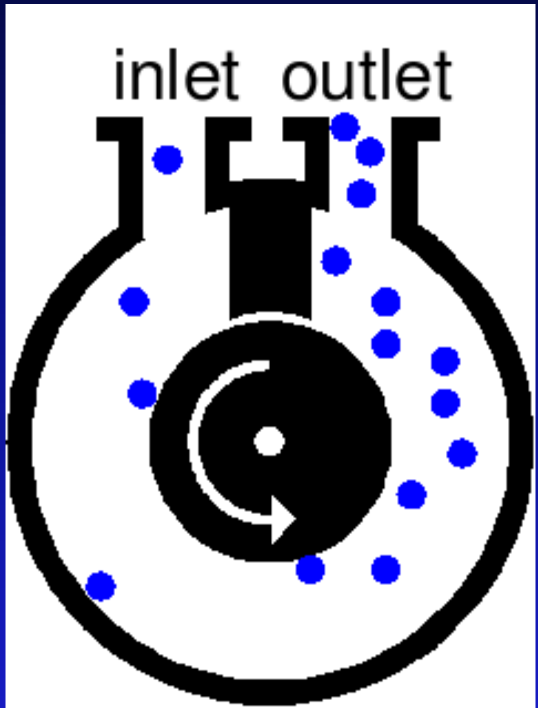
difficult to pump:

high sound velocity,

high particle velocity

Molecular pumps: Drag and turbo

Gaede pump (Drag pump)



Gaede 1913

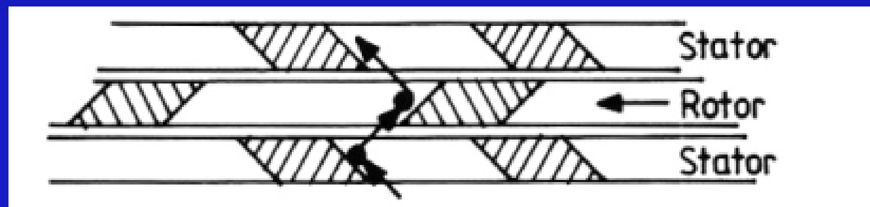
10.000-30.000 rpm

10-100 l/s

needs fore pump (max 0.1 mbar)

Turbomolecular pump (TMP)

Becker 1958

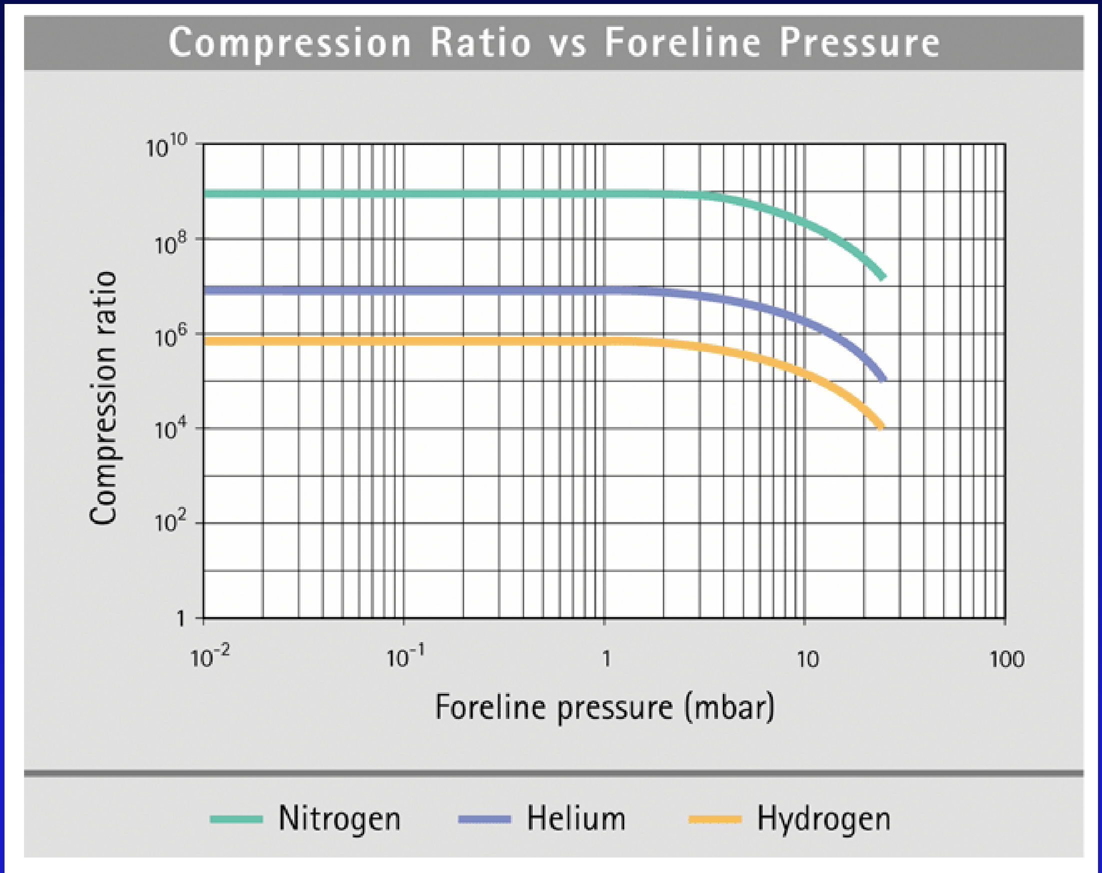


10.000-30.000 rpm

10-1000 l/s

needs fore pump (max 10^{-2} mbar)

Compression Ratio

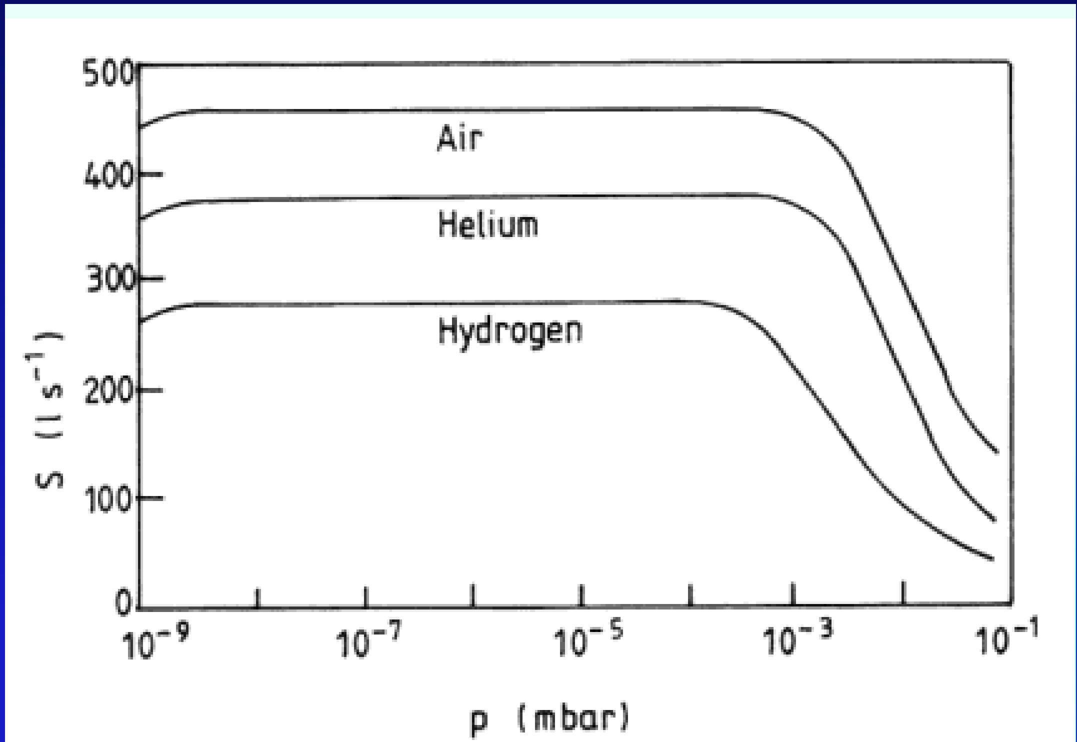


$K = p_{in} / p_{out}$

gas	compression ratio
H_2	10^3 - 10^6
He	10^4 - 10^7
N_2	10^8 - 10^9

Pumping Speed

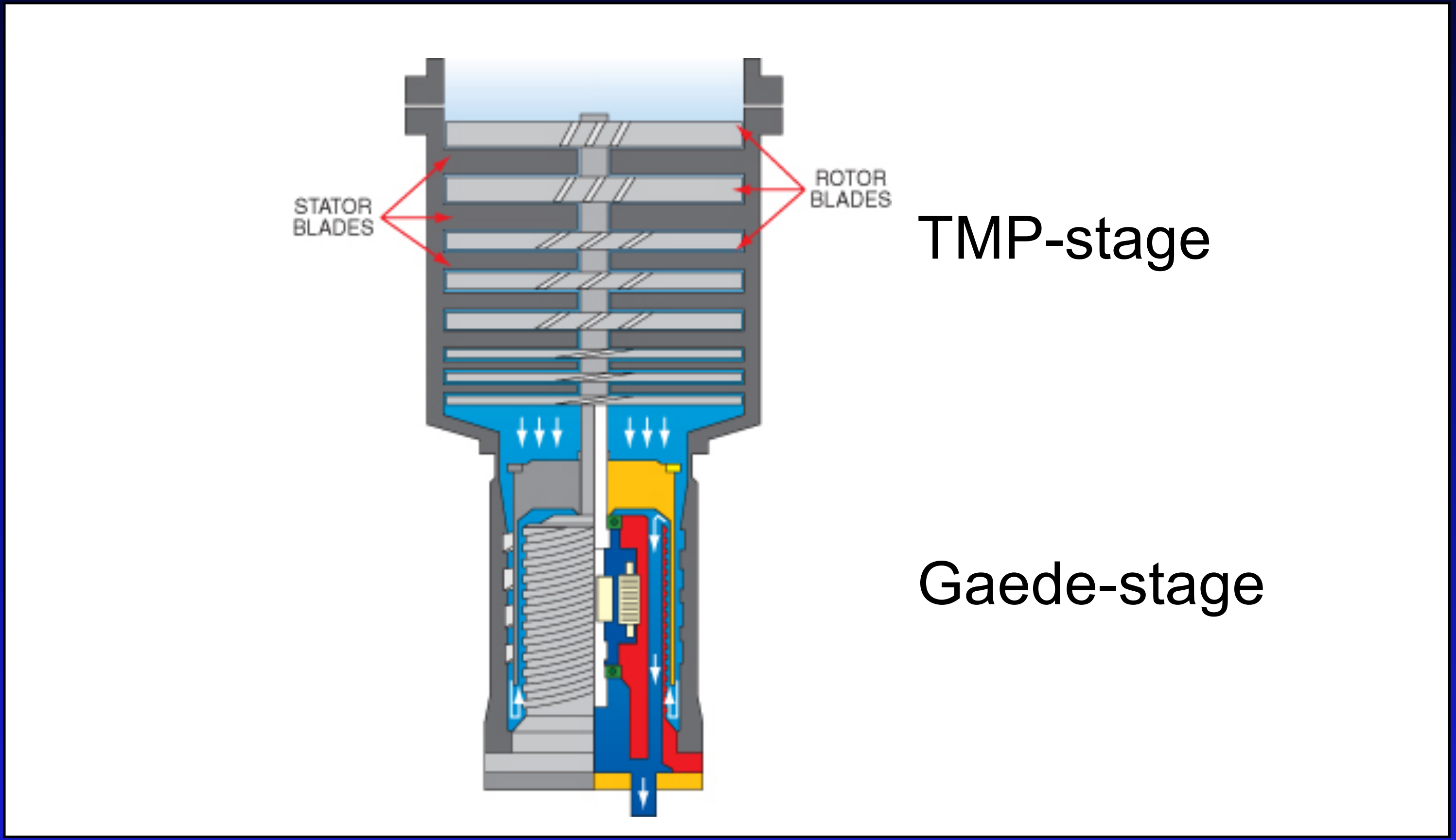
Pumping Speed



rotary backing pump is required producing a pressure of 10^{-2} mbar

magnetically levitated bearings → no oil or grease

Compound TMP: TMP + Gaede



Advantage: low requirements for ultimate fore-pump pressure:

e.g. oil free scroll pumps / membrane pumps possible

Diffusion pump

Principle: Diffusion pumps are vapor jet pumps

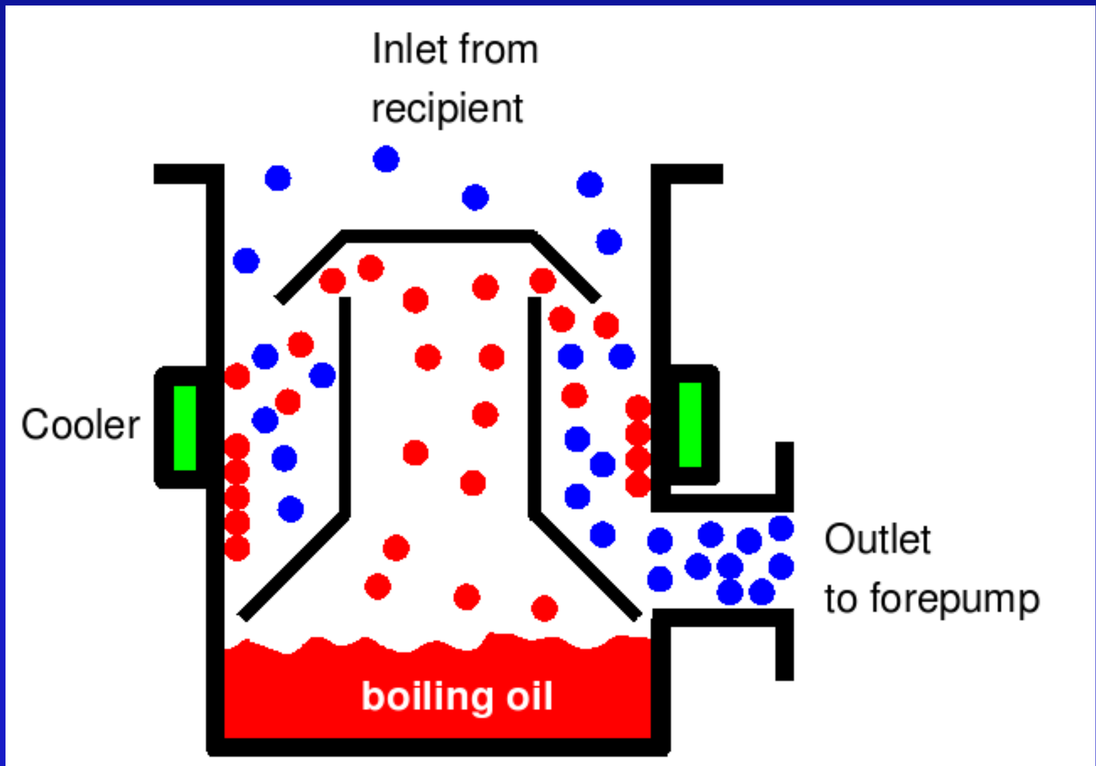
momentum transfer from a heavy high speed vapor jet

to gas molecule will be moved through pump

molecular flow regime, needs good fore pump

Pumping speed: 100 – 10.000 l/s

excellent pump for light gases Pumping speed: $S(H_2)/S(air) = 3$

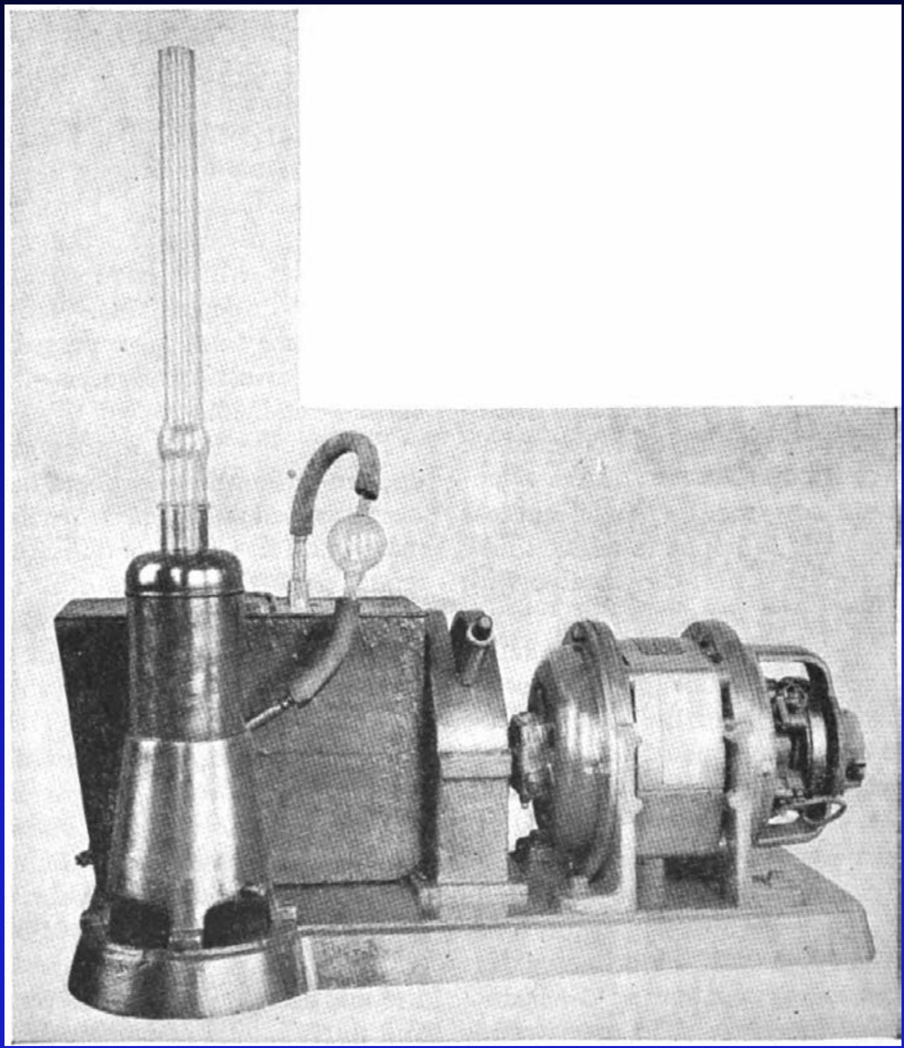


First high-vacuum pump

Invented 1915 by Wolfgang Gaede

Improved 1916 by
Irving Langmuir and W. Crawford

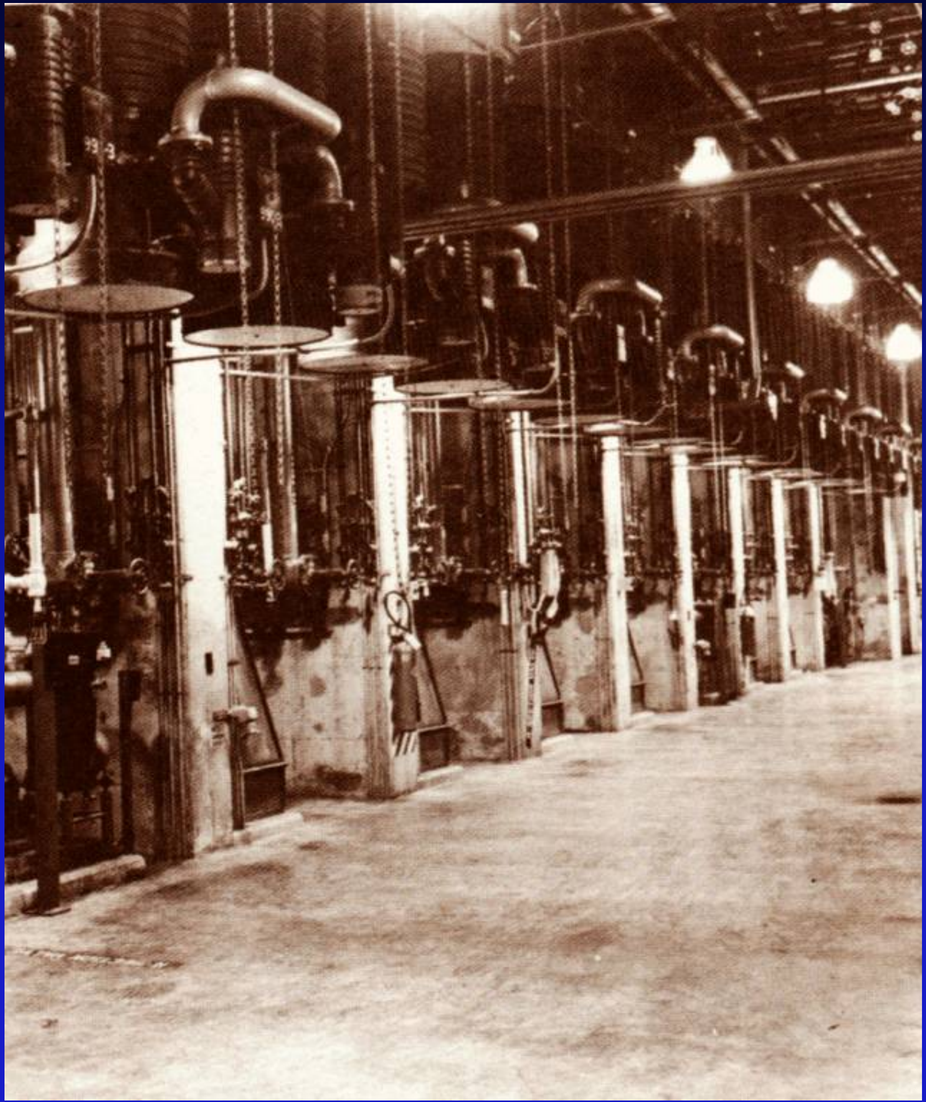
Diffusion pump



Early Langmuir mercury diffusion pump

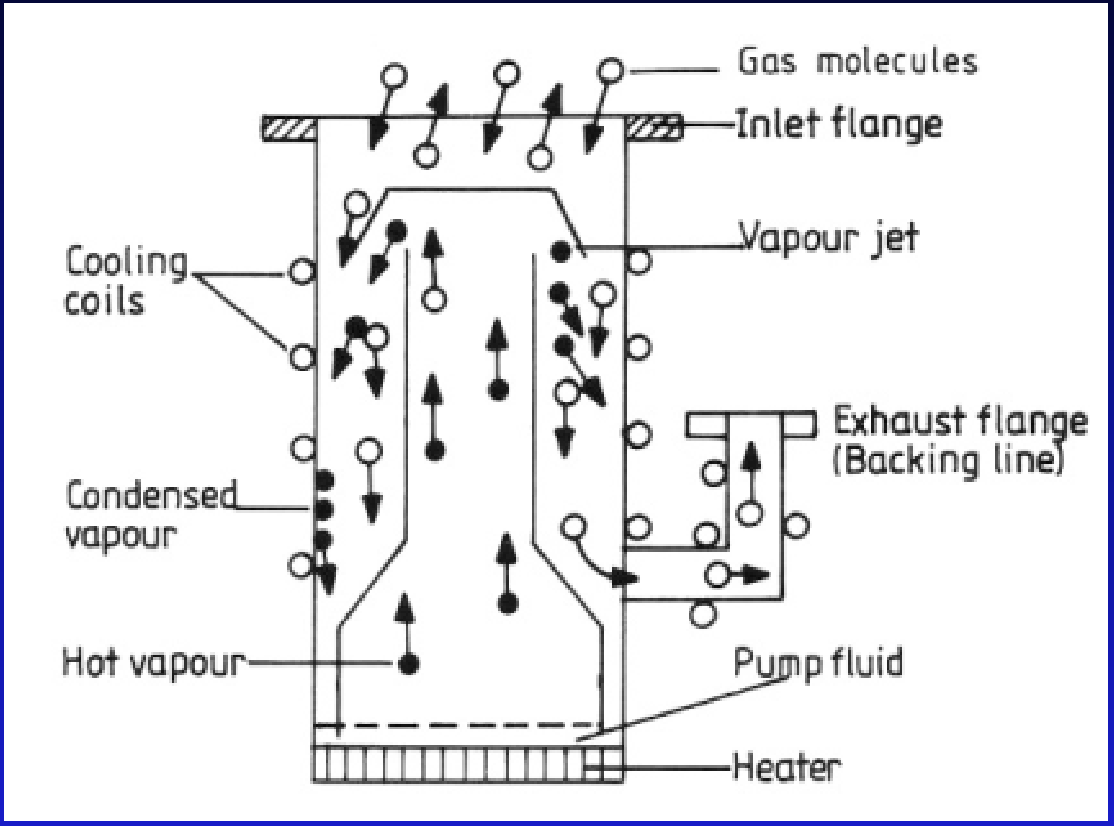
modern version of a diffusion pump

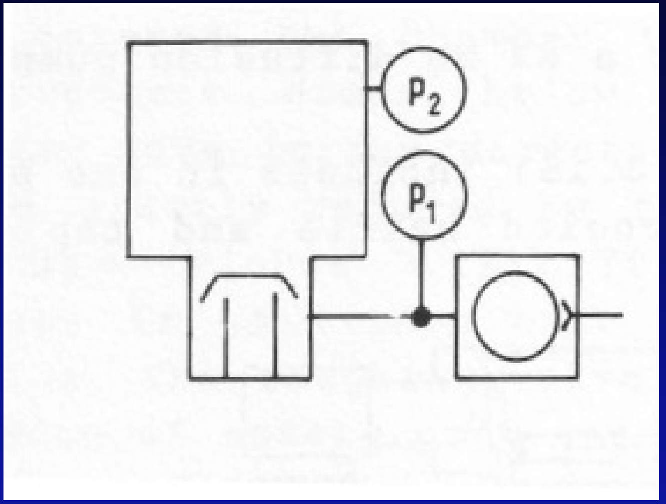
Diffusion pump

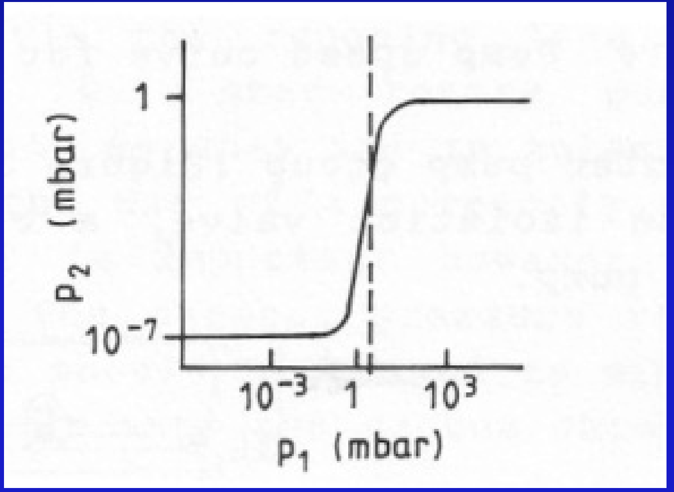


Battery of Diffusion Pumps used in the Manhatten Project

Diffusion Pumps





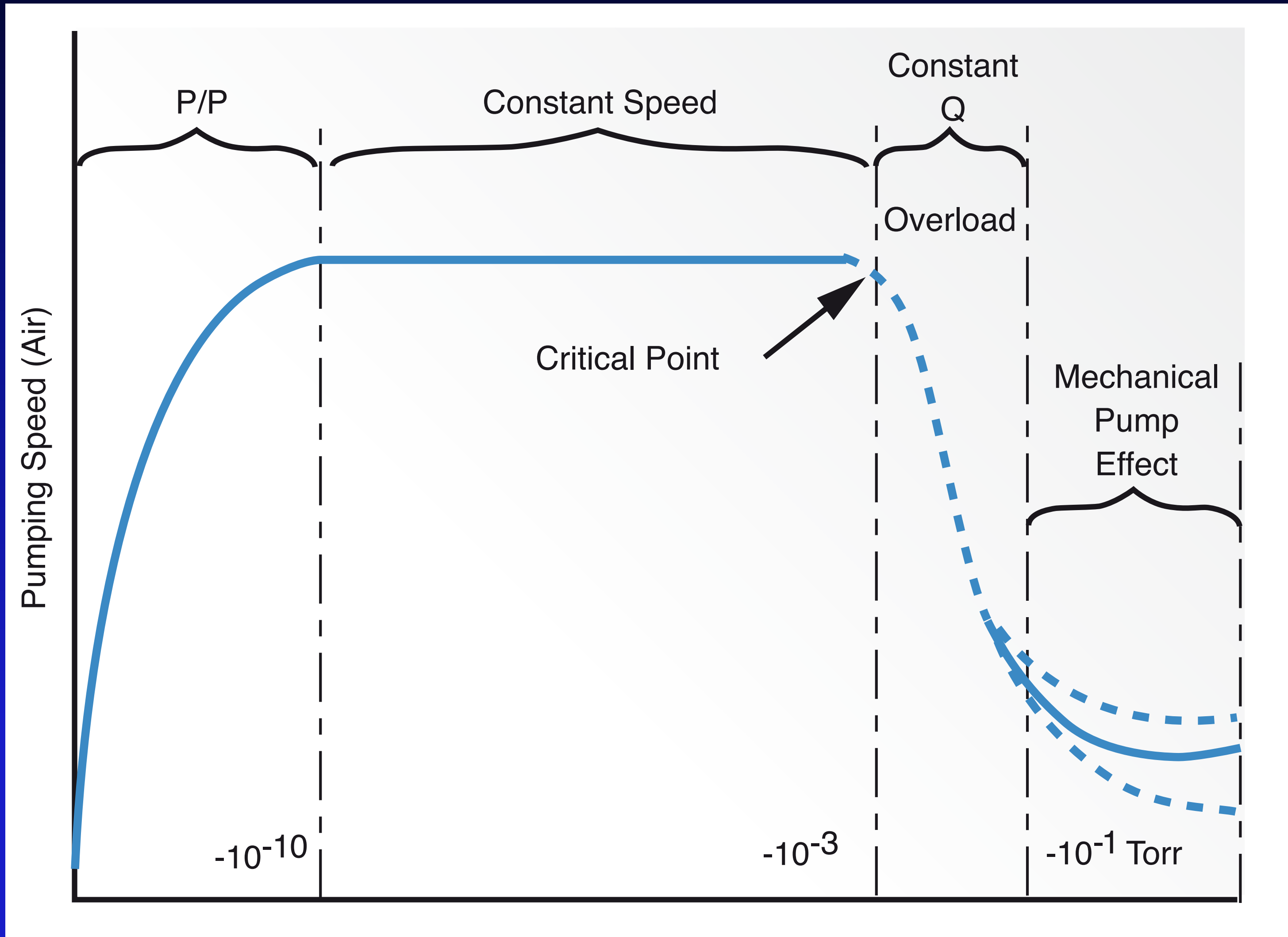


trapping the pumped gas molecules in a high velocity stream of oil vapor

low ultimate pressure, high pumping rate, small cost

pumping speed between 10^2 … 10^4 ℓ/s

Pumping Speed

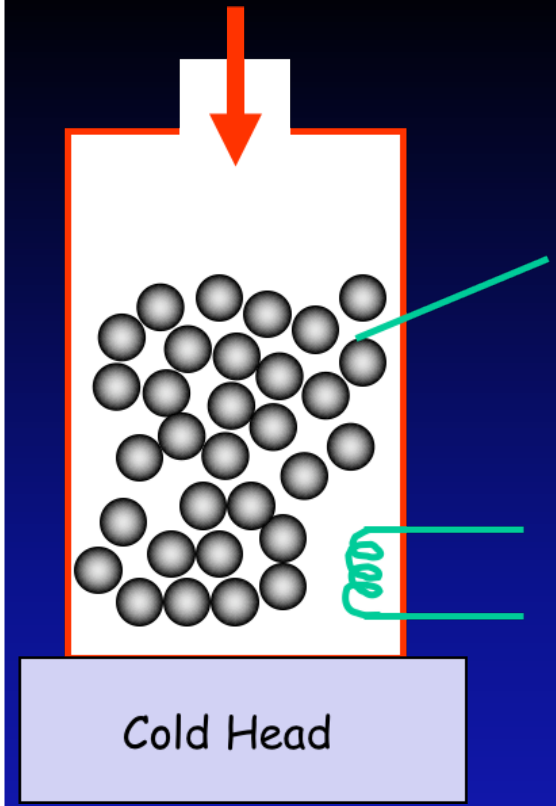


Entrapment Pumps:

Getter Pumps

Adsorption Pumps

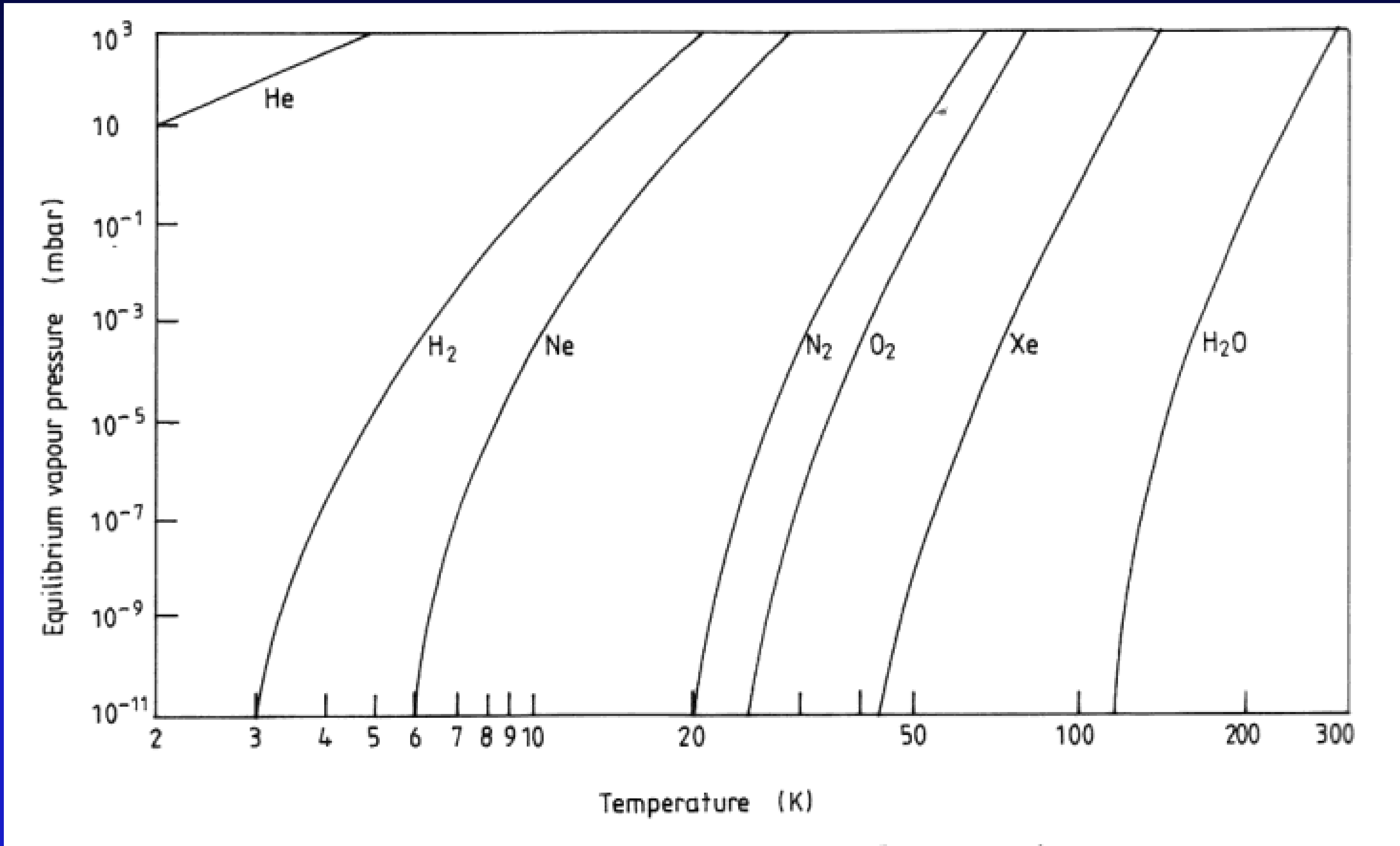
Adsorption Pumps



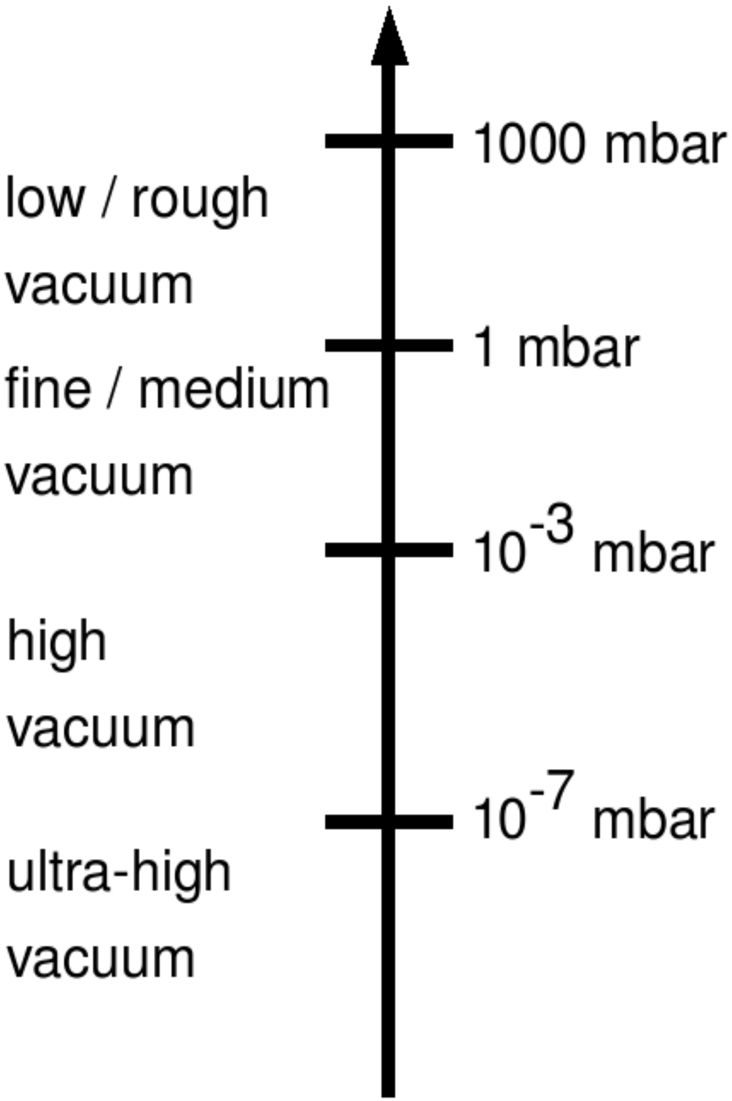
NO gas transfer pump, but getter principle:

- gas atoms / molecules adsorbed at cold surface
- best vacuum at all
- large volume flow rate (up to 10^6 l/s) for limited times

Vapour Pressure of Different Gases



Measurements of Pressure

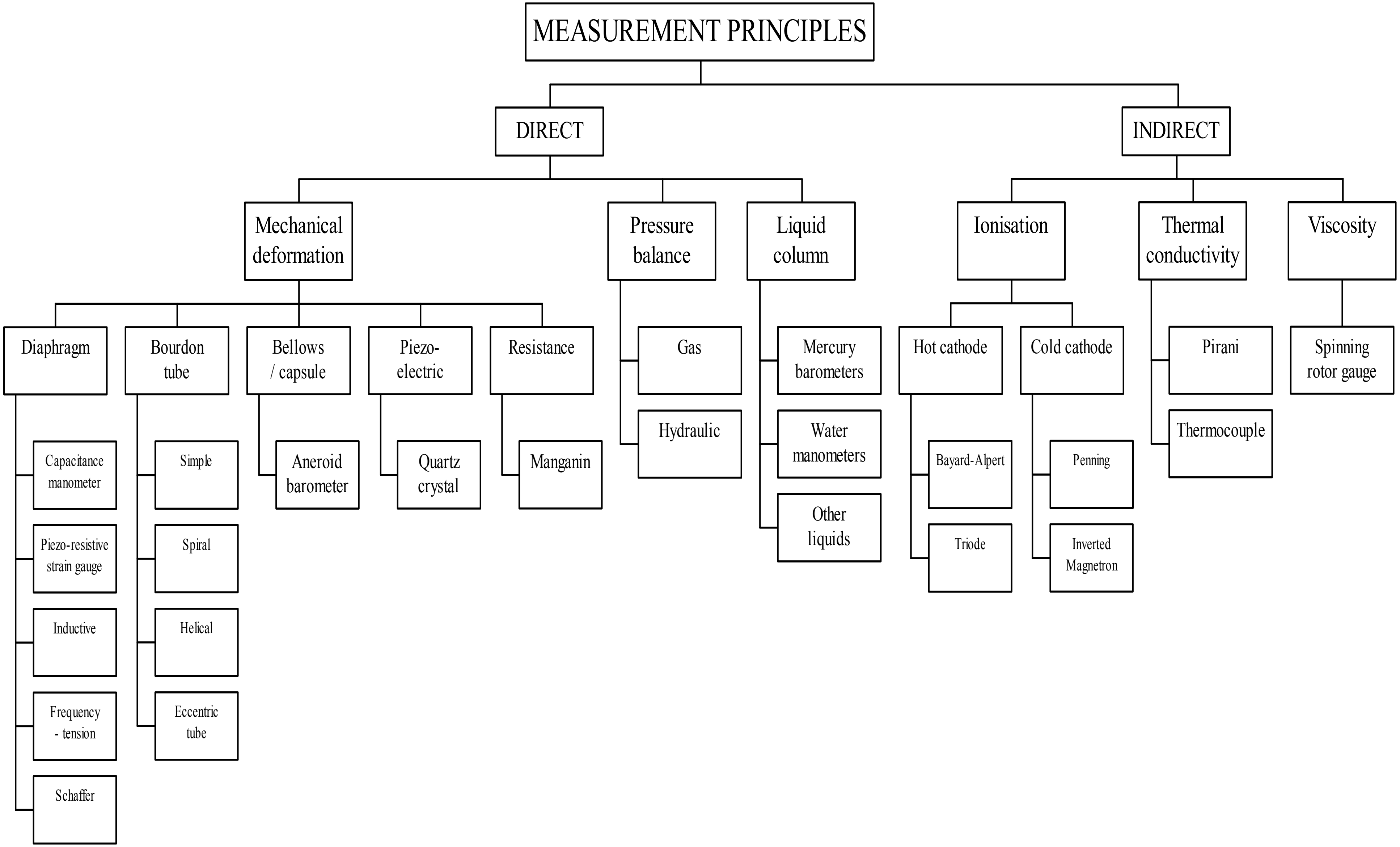


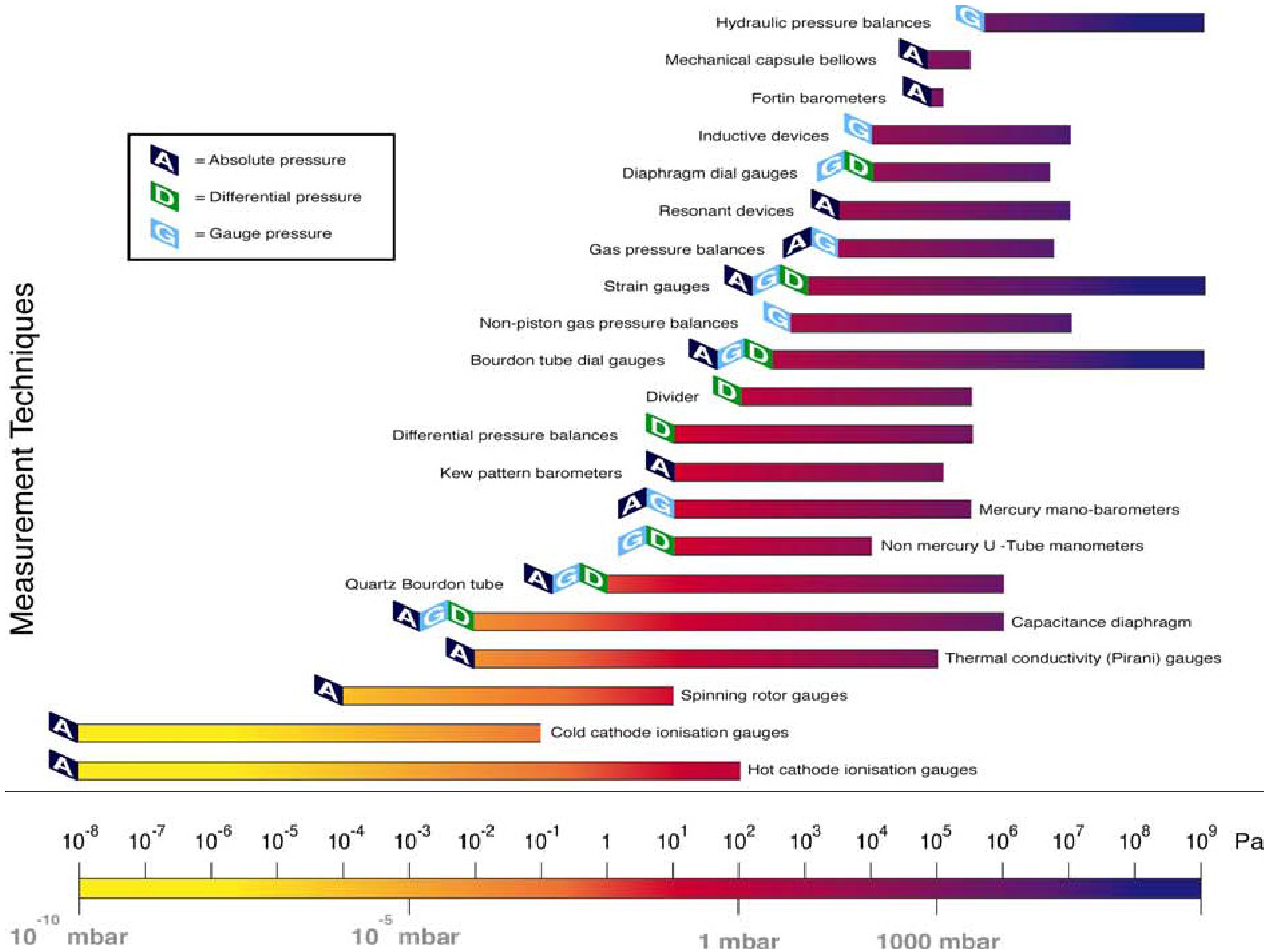
- mechnical gauges
  (e.g. Bourdon gauge)

- McLeod compression gauge
- Pirani gauge

- Ionization gauge
- Penning gauge

Measurements of Pressure

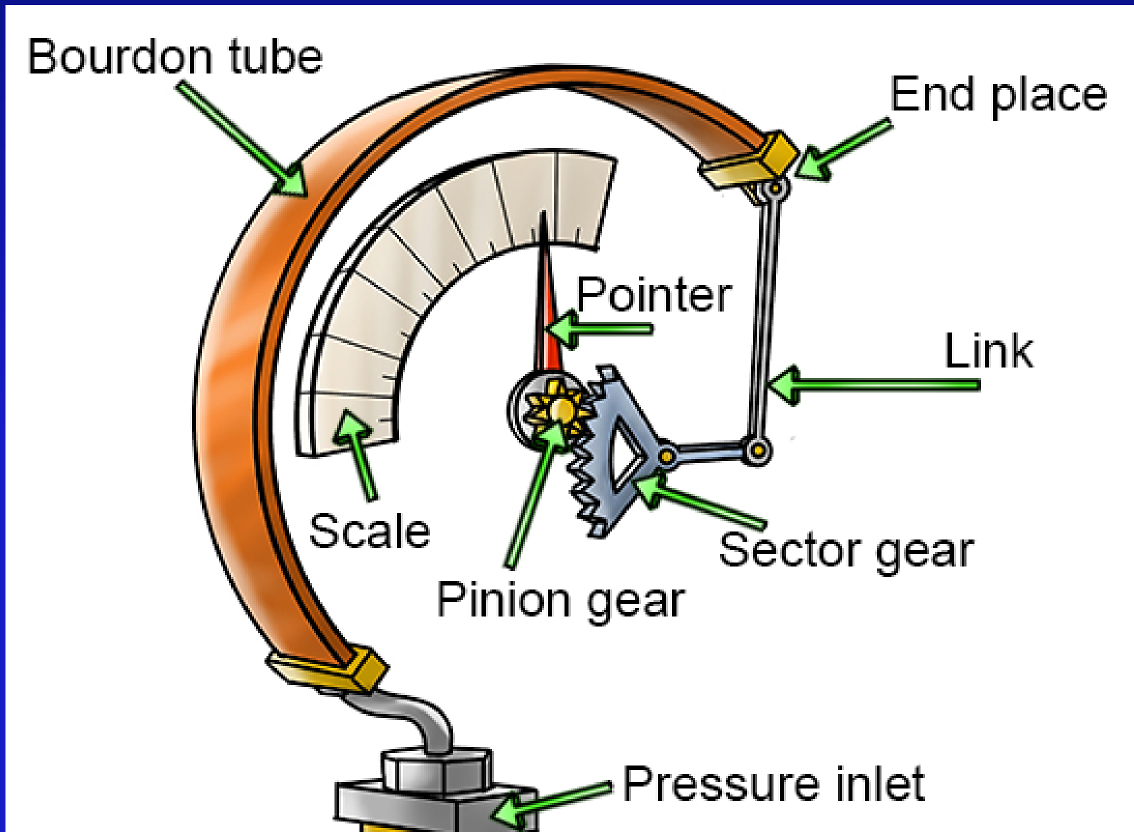


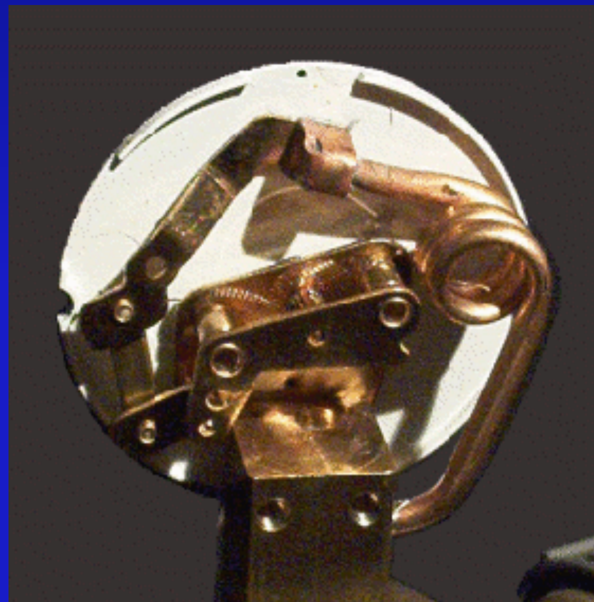


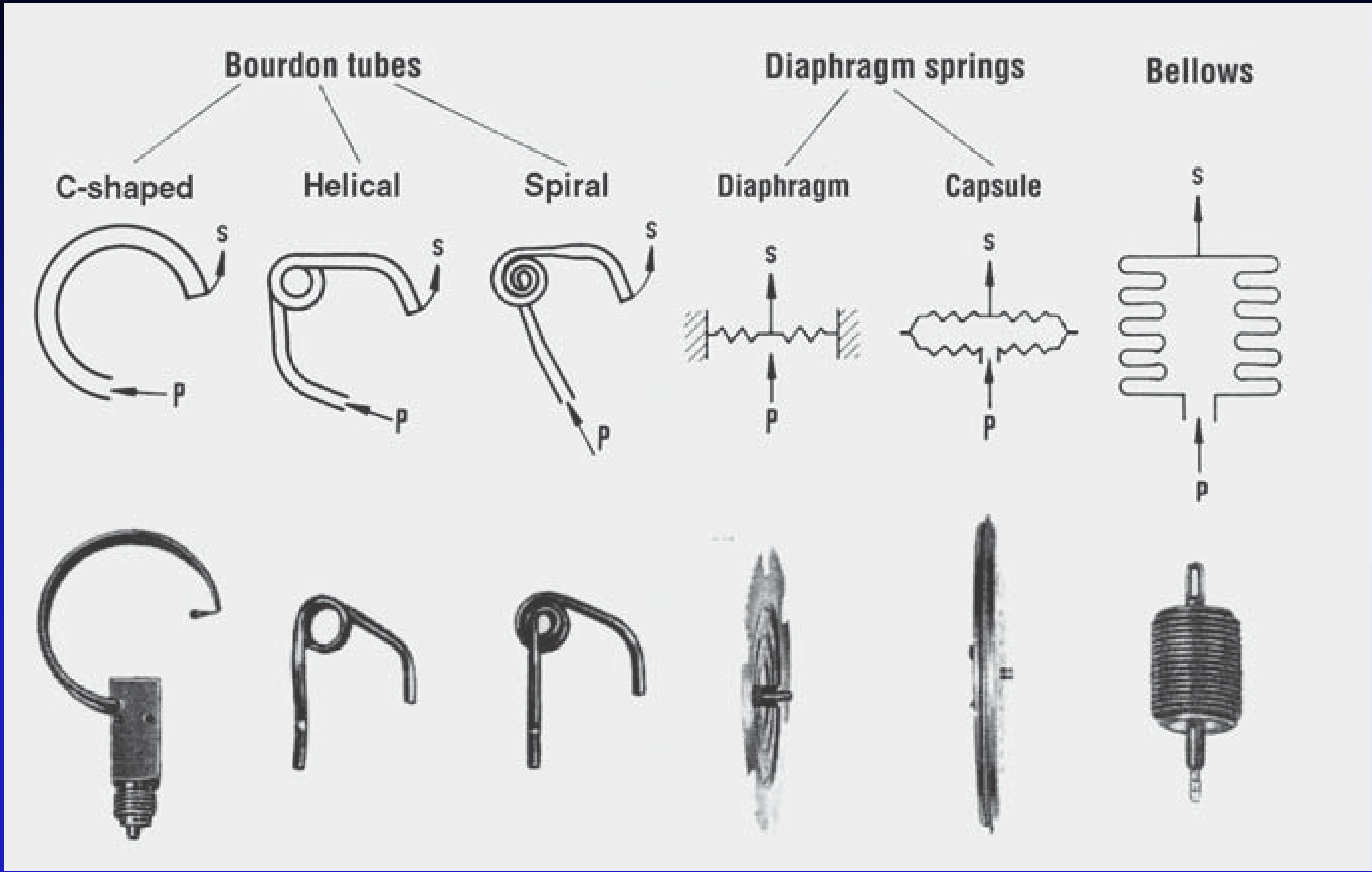
Measurement Techniques
= Absolute pressure
= Differential pressure
= Gauge pressure
Hydraulic pressure balances
Mechanical capsule bellows
Fortin barometers
Inductive devices
Diaphragm dial gauges
Resonant devices
Gas pressure balances
Strain gauges
Non-piston gas pressure balances
Bourdon tube dial gauges
Divider
Differential pressure balances
Kew pattern barometers
Mercury mano-barometers
Non mercury U -Tube manometers
Quartz Bourdon tube
Capacitance diaphragm
Thermal conductivity (Pirani) gauges
Spinning rotor gauges
Cold cathode ionisation gauges
Hot cathode ionisation gauges
10^{-8} 10^{-7} 10^{-6} 10^{-5} 10^{-4} 10^{-3} 10^{-2} 10^{-1} 1 10^{1} 10^{2} 10^{3} 10^{4} 10^{5} 10^{6} 10^{7} 10^{8} 10^{9} Pa
10^{-10} mbar
10^{-5} mbar
1 mbar
1000 mbar

Mechanical Gauges

Bourdon gauge (10 mbar to 1 bar)

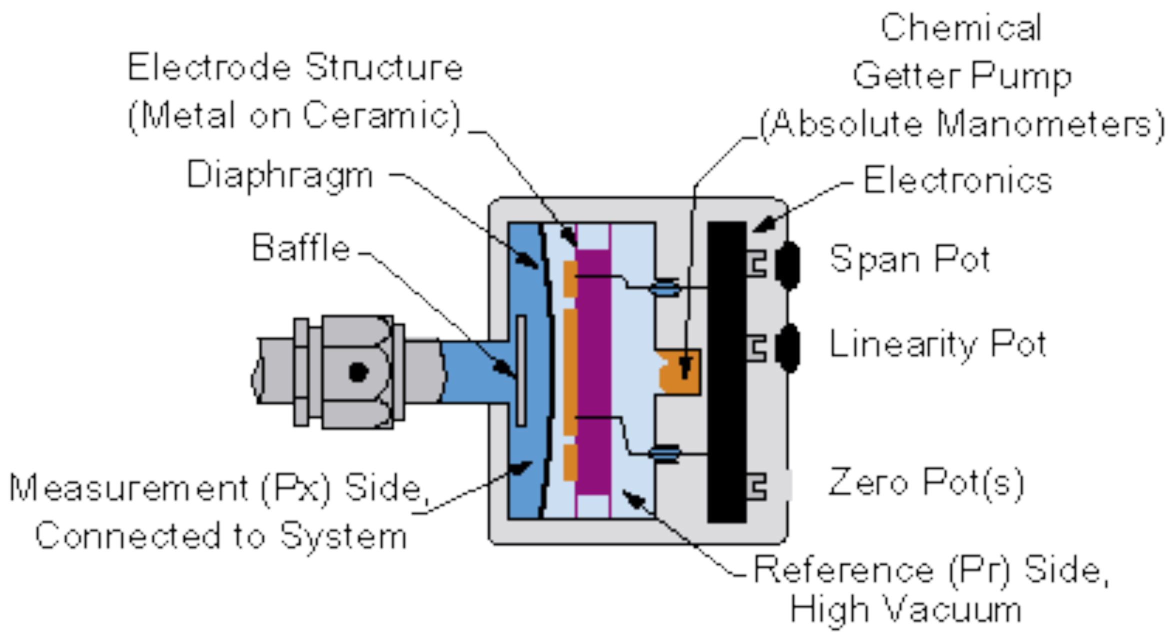





Bourdon tubes
Diaphragm springs
Bellows
C-shaped
Helical
Spiral
Diaphragm
Capsule
S
P
S
P
S
P
S
P
S
P
S
P

Capacitance Gauge

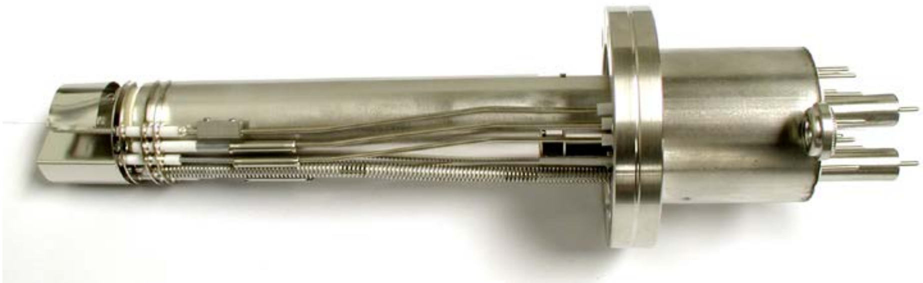
(10^{-4} mbar to 100 mbar)

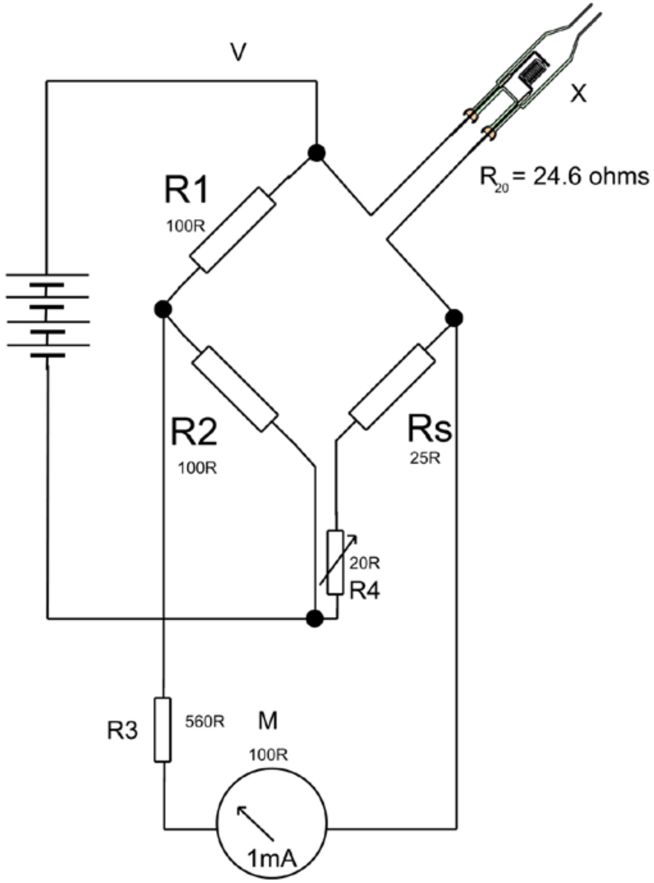


Thermal Conductivity Gauges

Pirani Gauge (10^{-4} mbar to 1 bar) 1906 Marcello Stefano Pirani

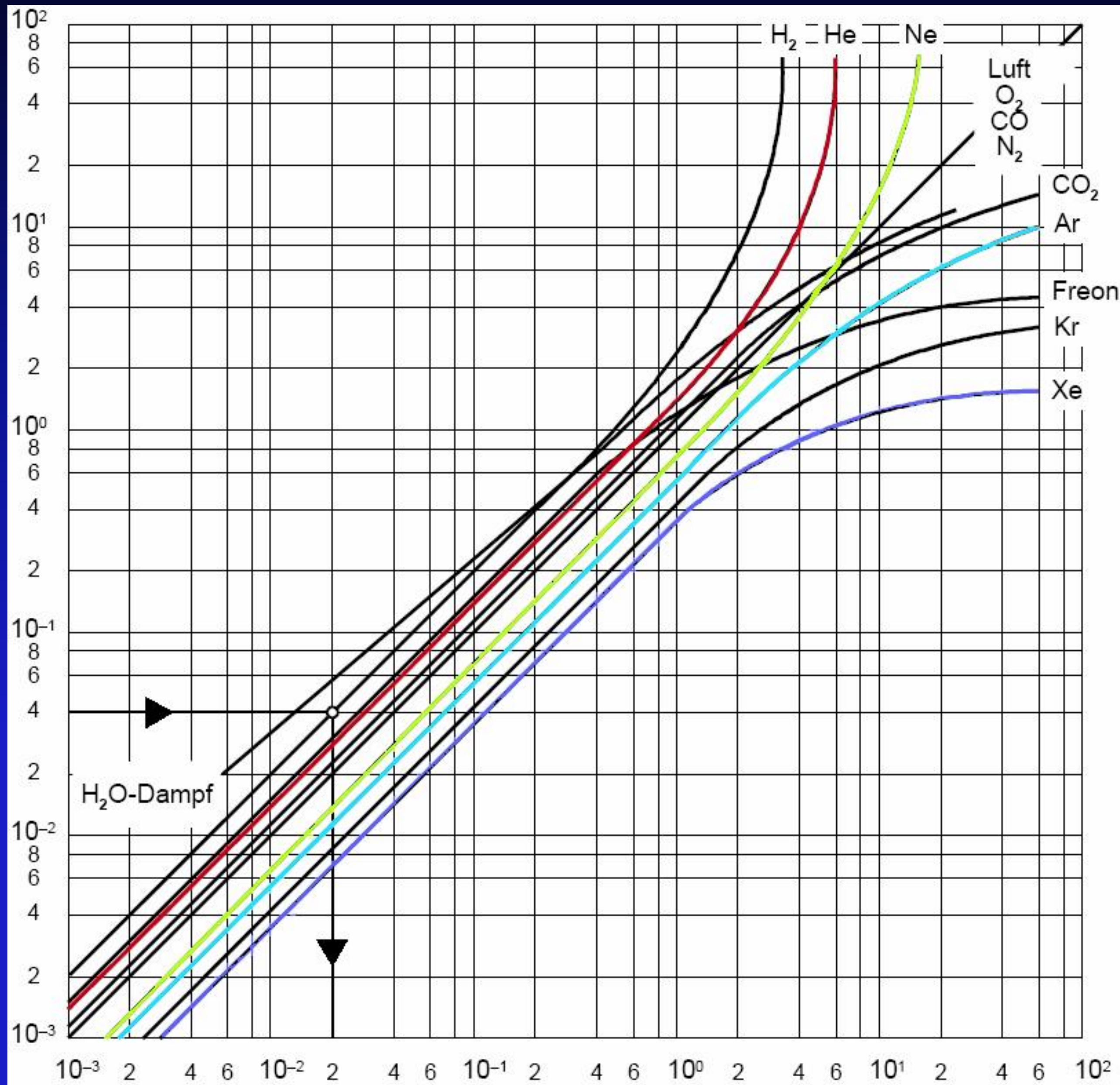
Resistance change of a gas cooled wire is measured with a bridge circuit under constant joule heating





Thermal Conductivity Gauges

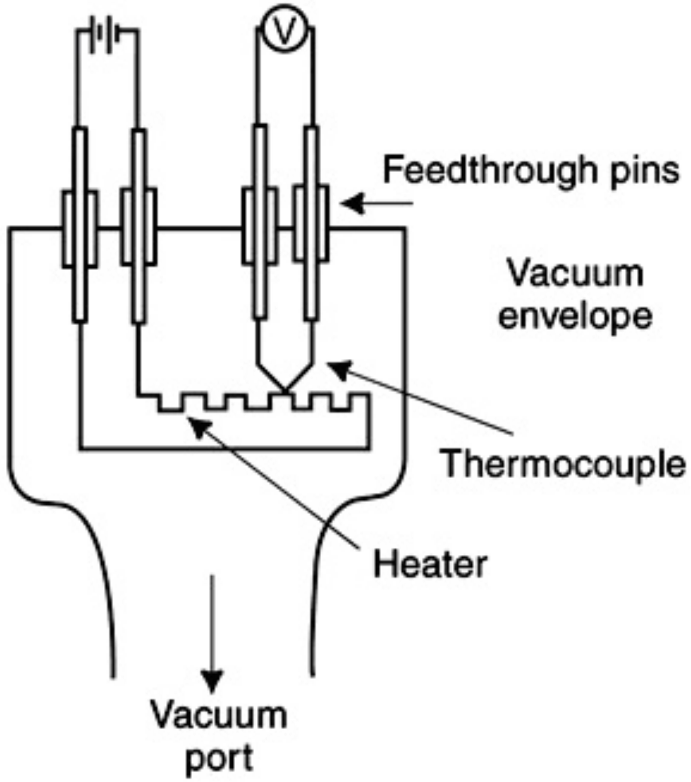
depends of gas type



Thermocouple Gauge

(10^{-4} mbar to 1 bar)

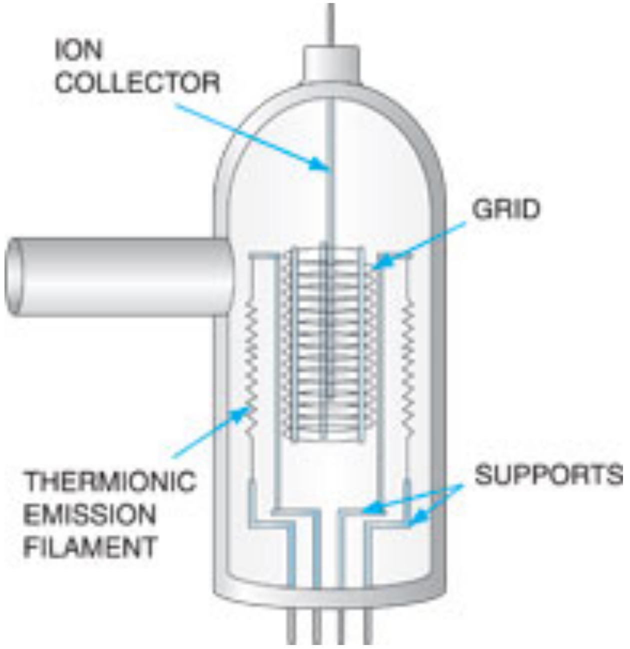
Temperature of gas cooled resistive wire is measured via thermocouples

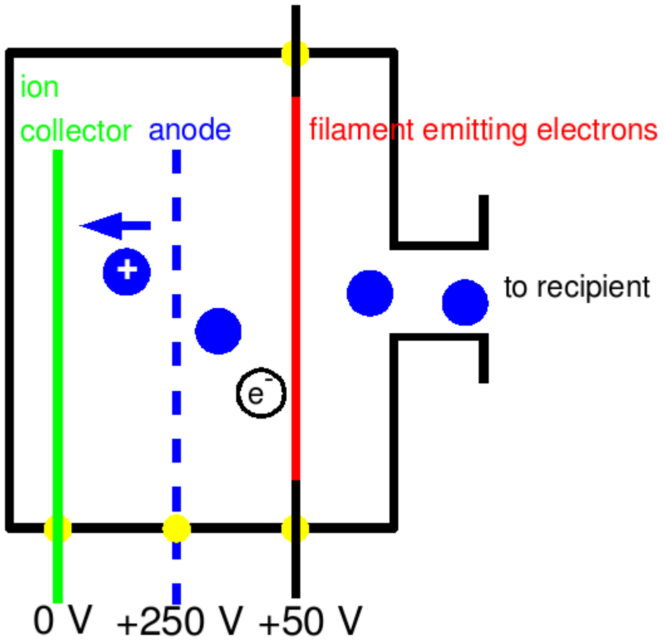


Ionisation Gauges

Hot cathode ionisation (10^{-12} mbar to 10^{-2} mbar)

create gas ions by electron bombardment stemming from a hot filament

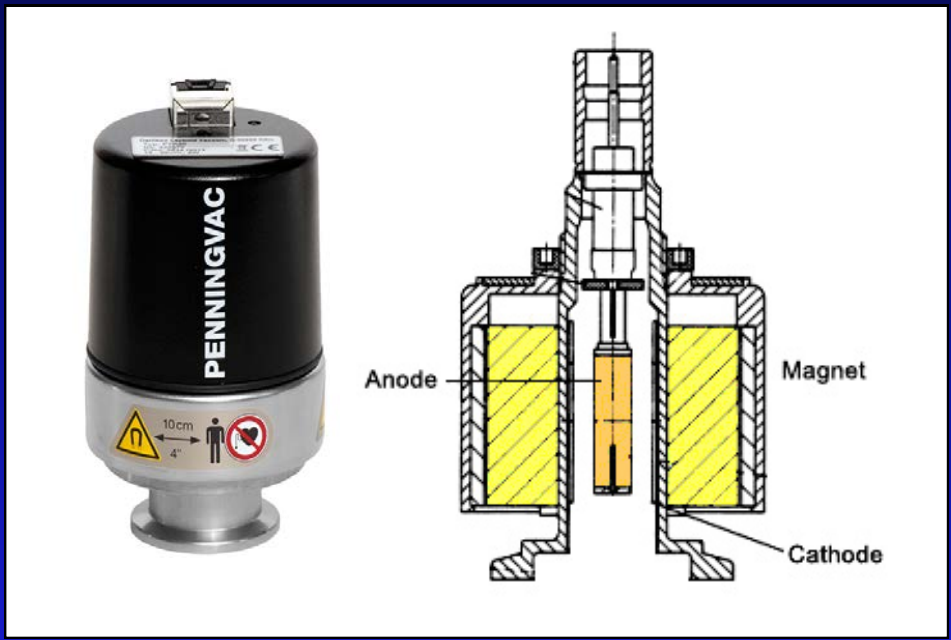


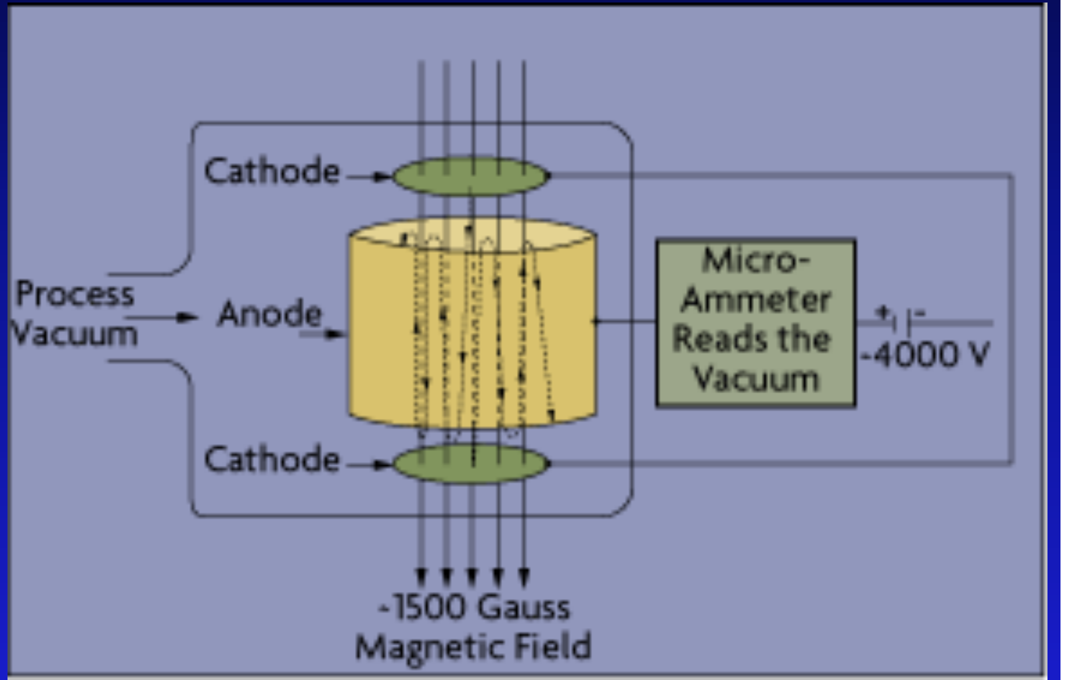


Ionisation Gauges

Penning cold cathode ionisation (10^{-12} mbar to 10^{-4} mbar)

high voltage → gas ionisation → plasma → measurement of ionisation current





advantages:

rushing in of air in no problem

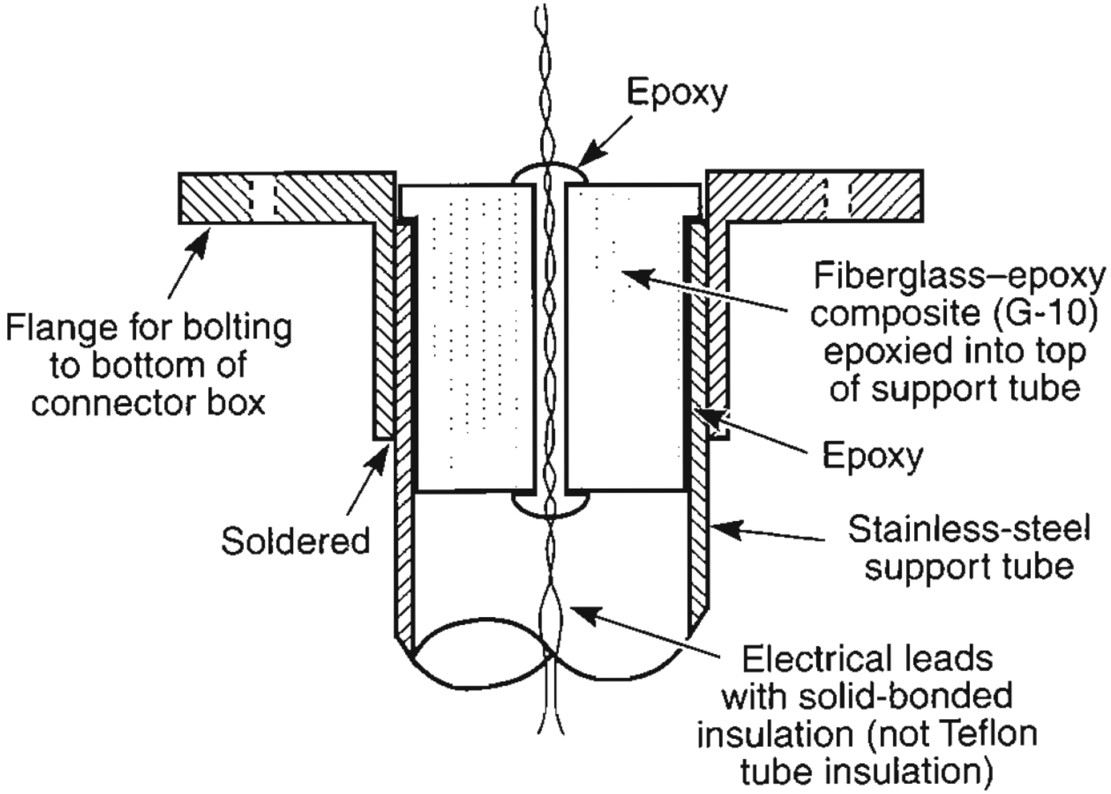
insensitive to mechanical vibrations

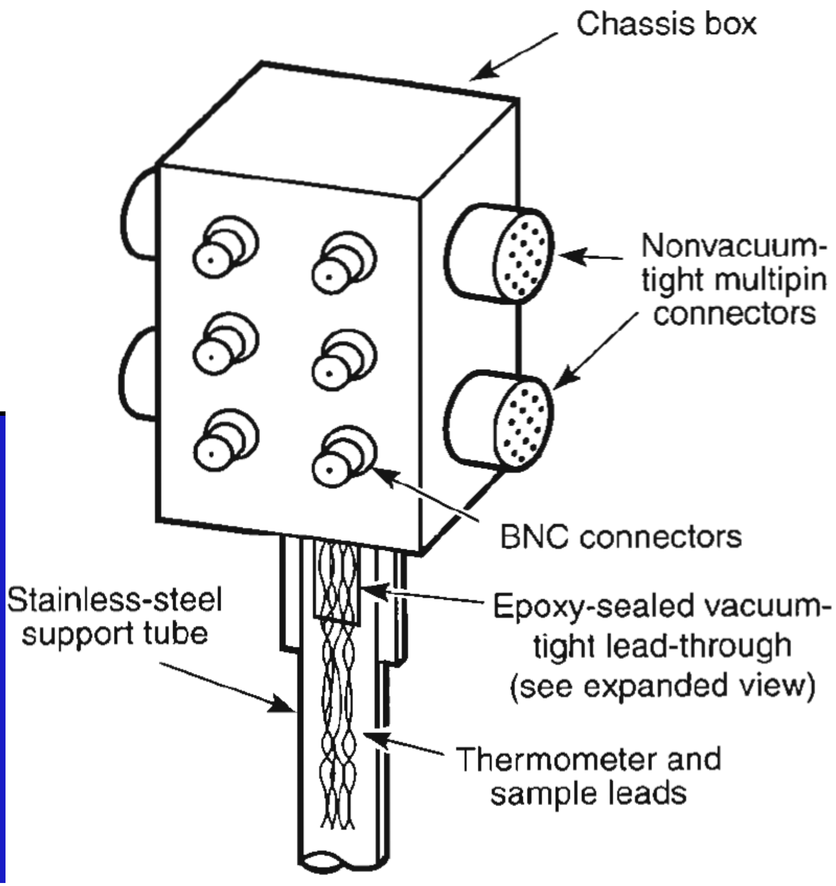
Vacuum Flansch

Seals and Feed Throughs

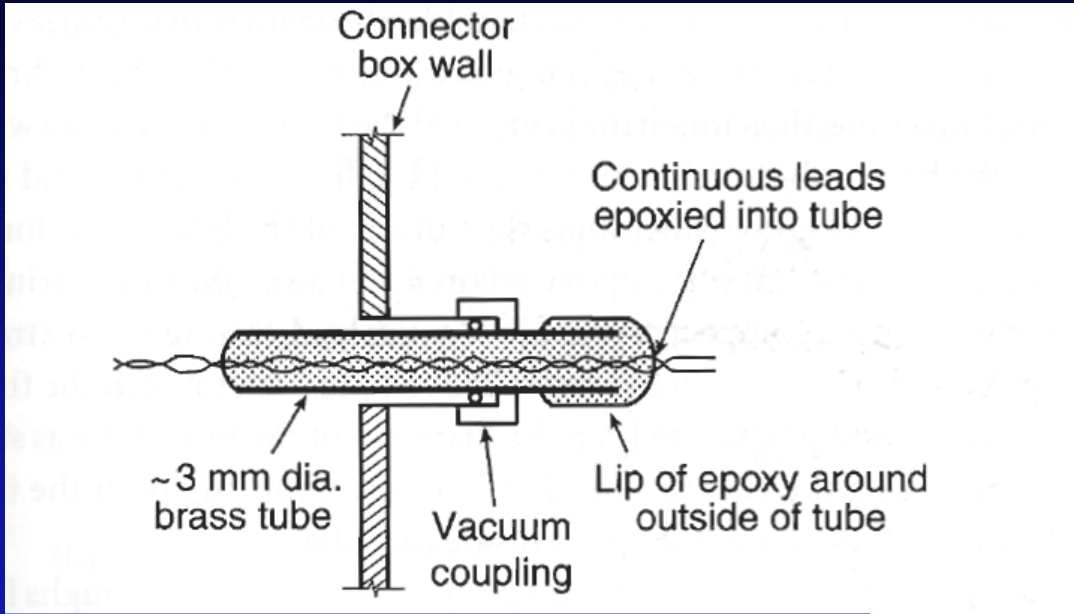
Leak Detection

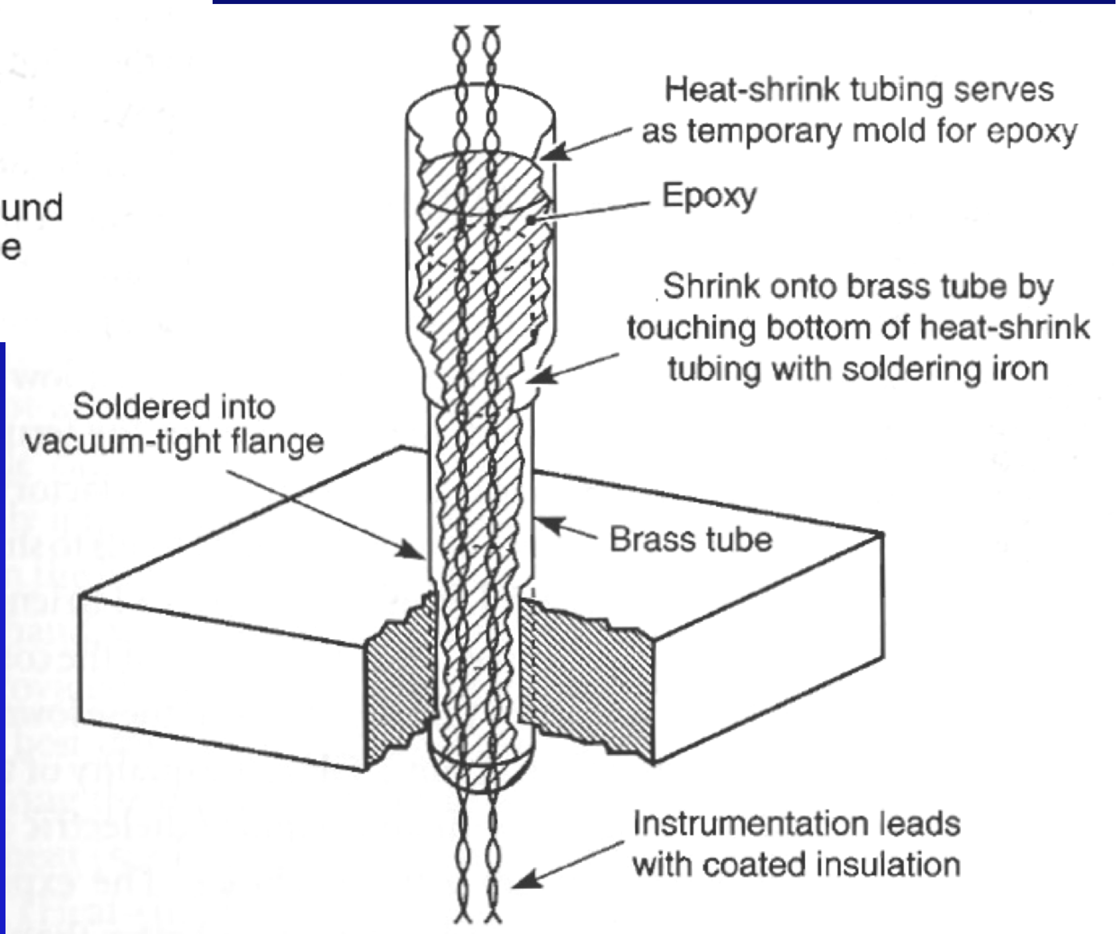
Seals and Feed Throughs



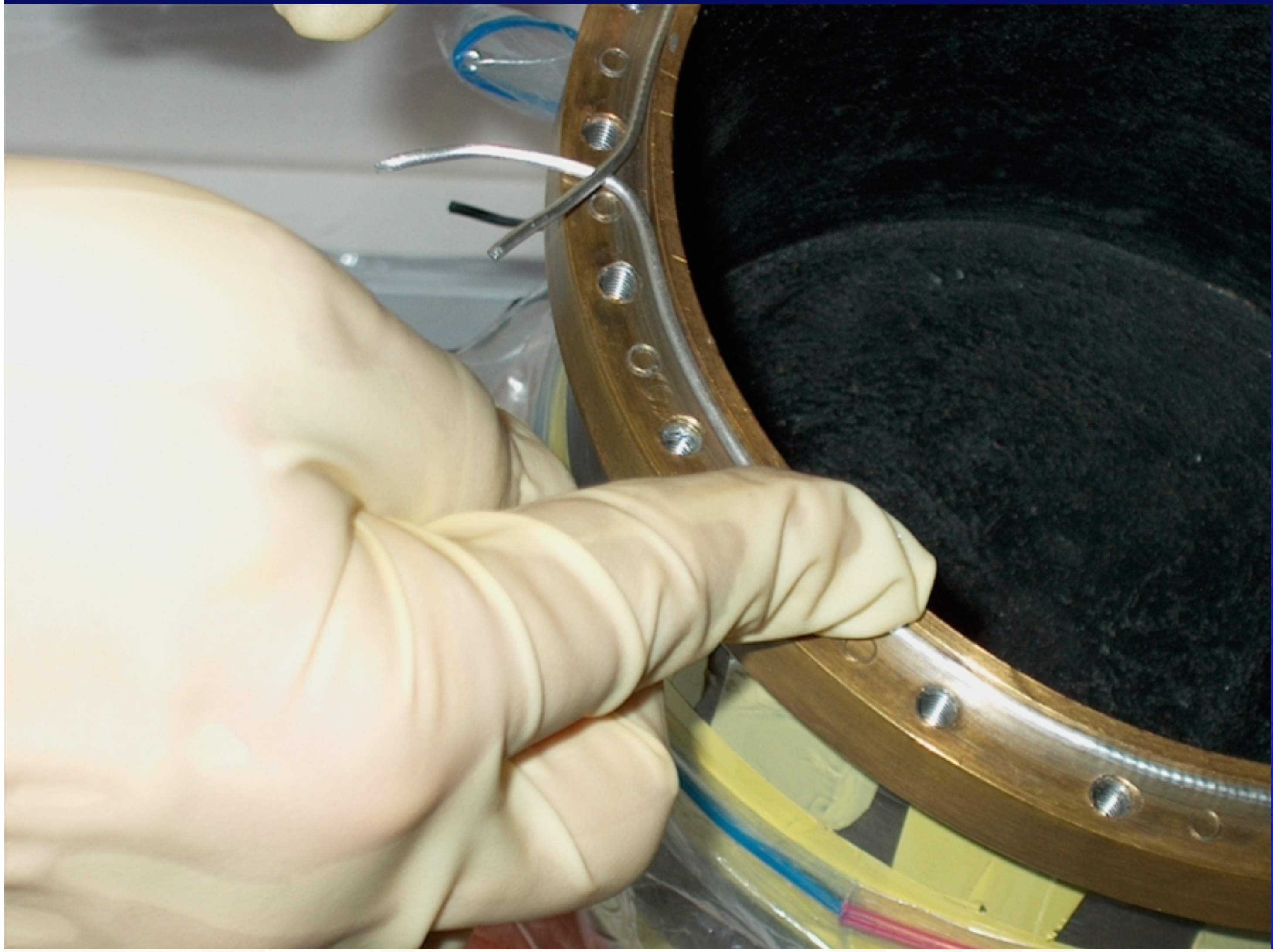


Seals and Feed Throughs





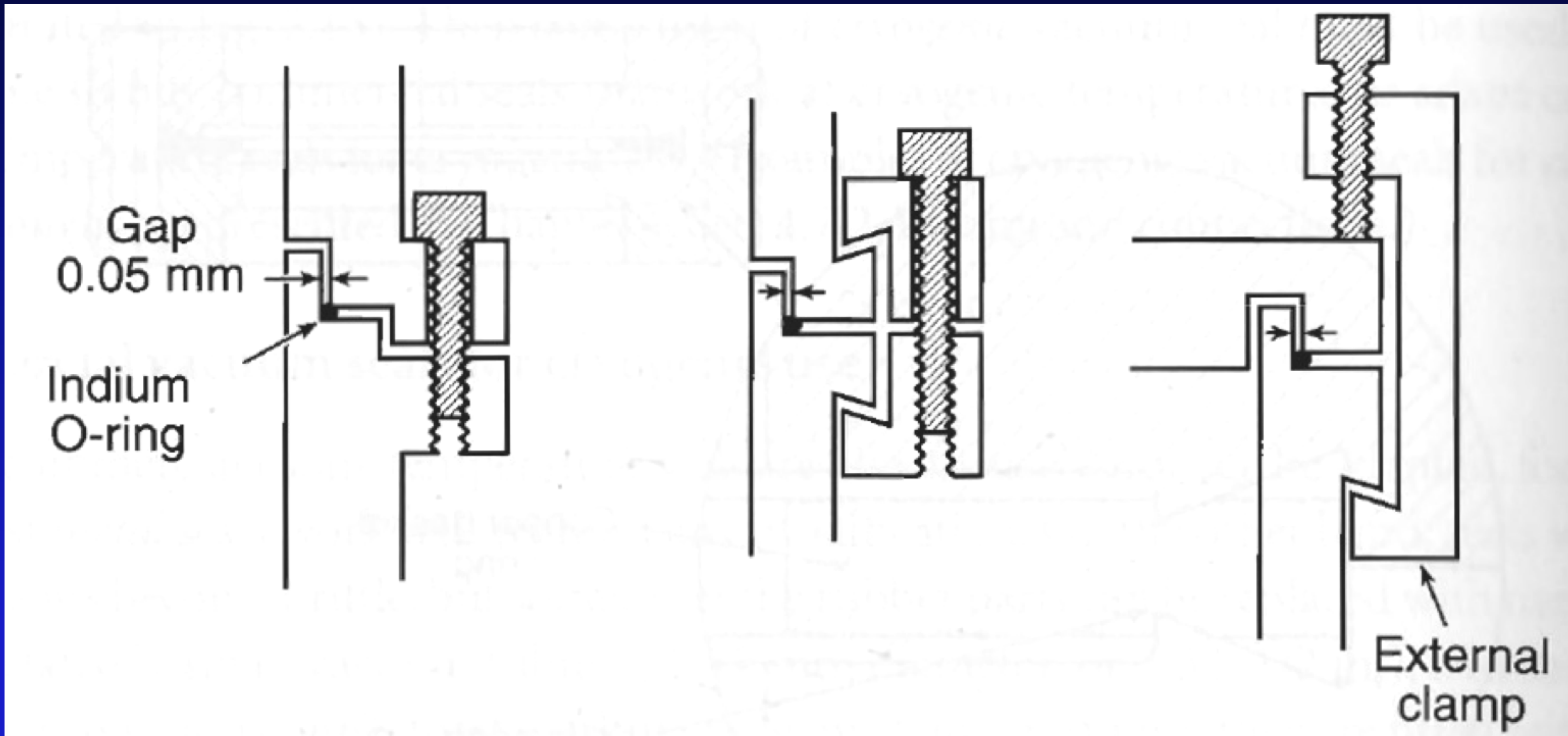
Indium Seals



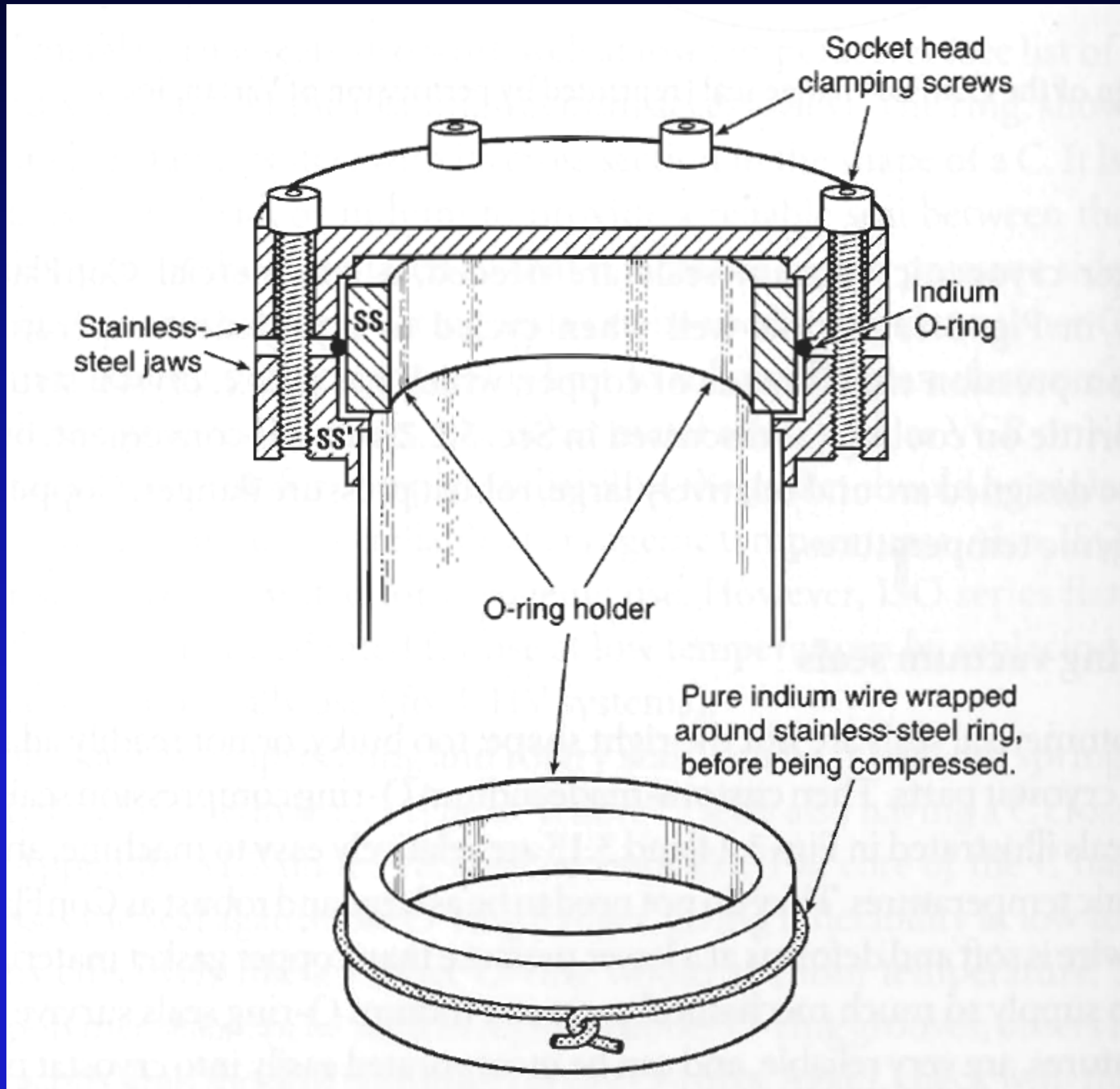
Indium Seals

Indium Seals

Indium Seals



Indium Seals



Leaks and Leak Detection

Indications to have a leak in a cryostat:

oscillating base temperature

higher 1 K pot temperature

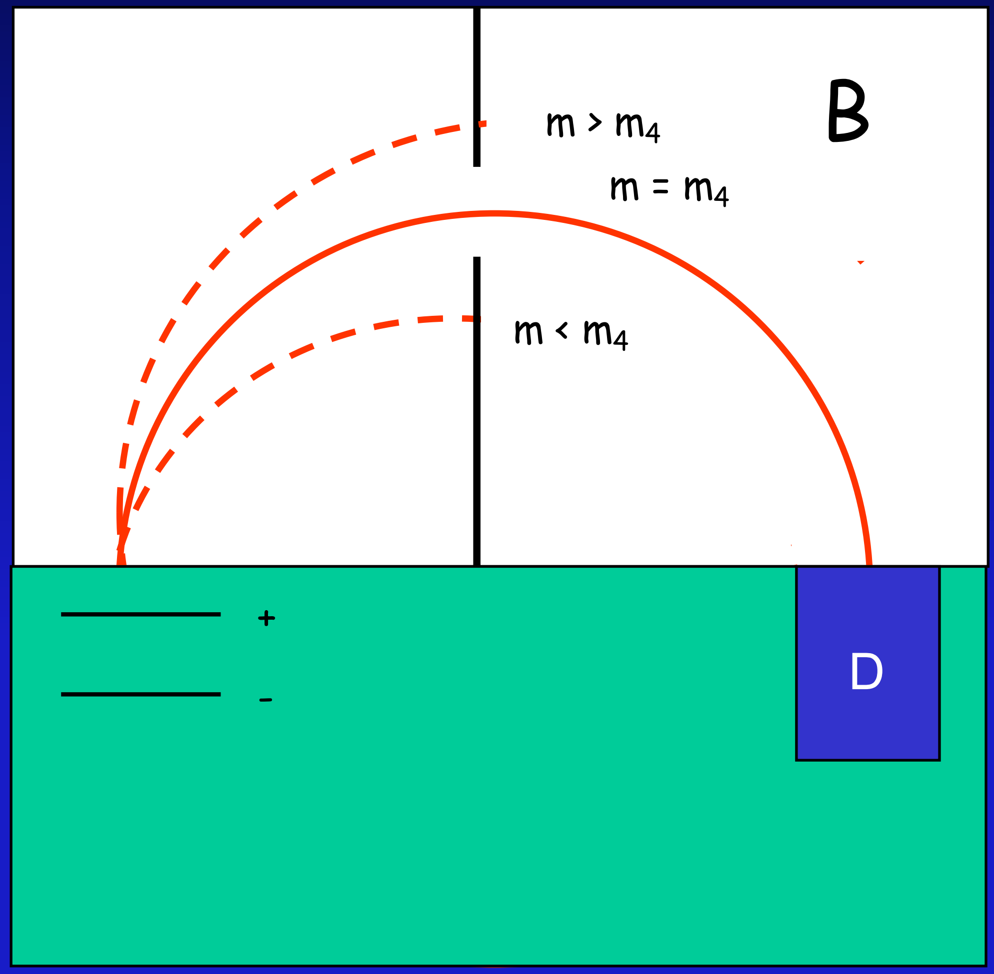
higher base temperature

bad vaccum

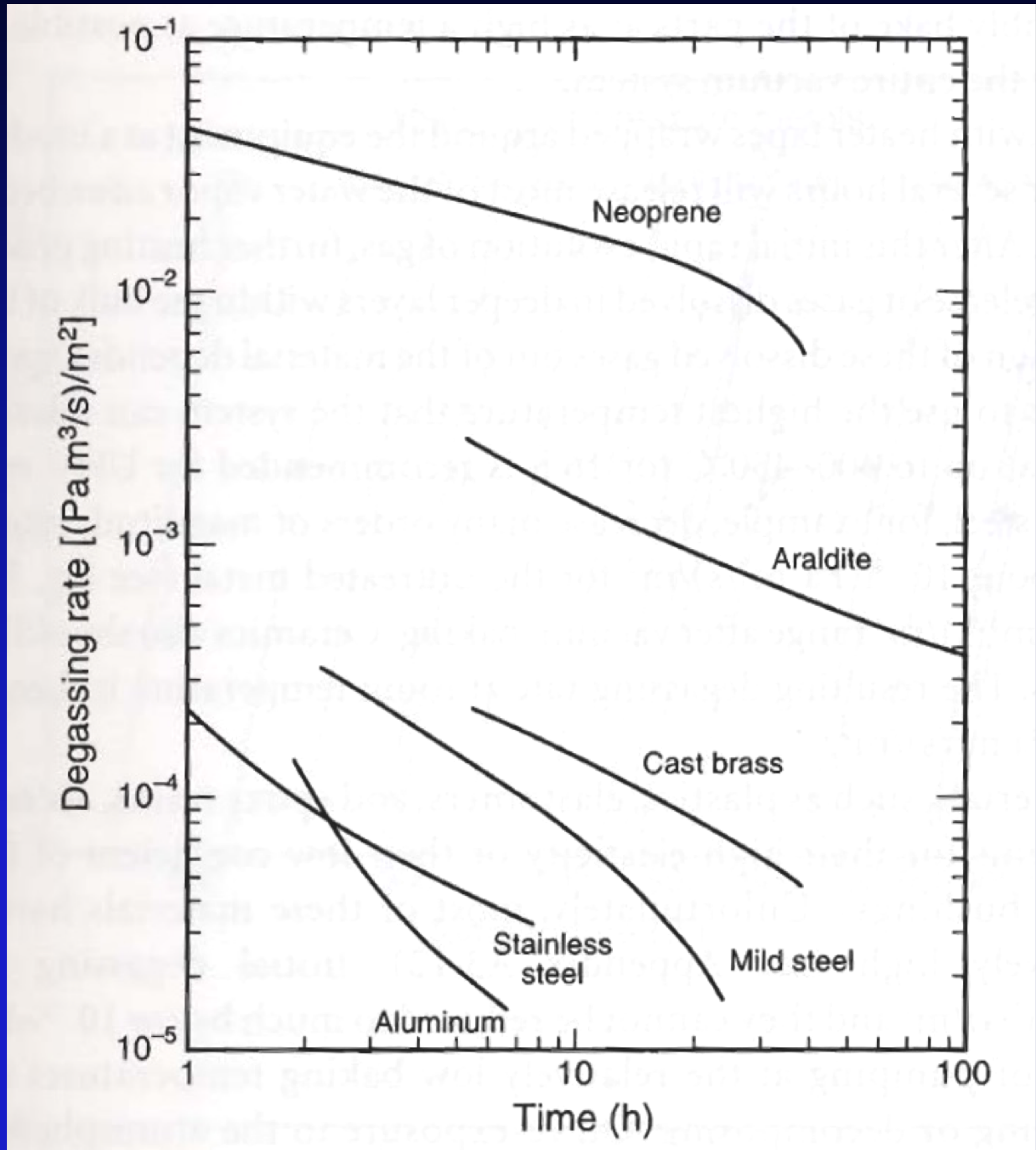
thermal short

Leaks and Leak Detection

mass spectrometer

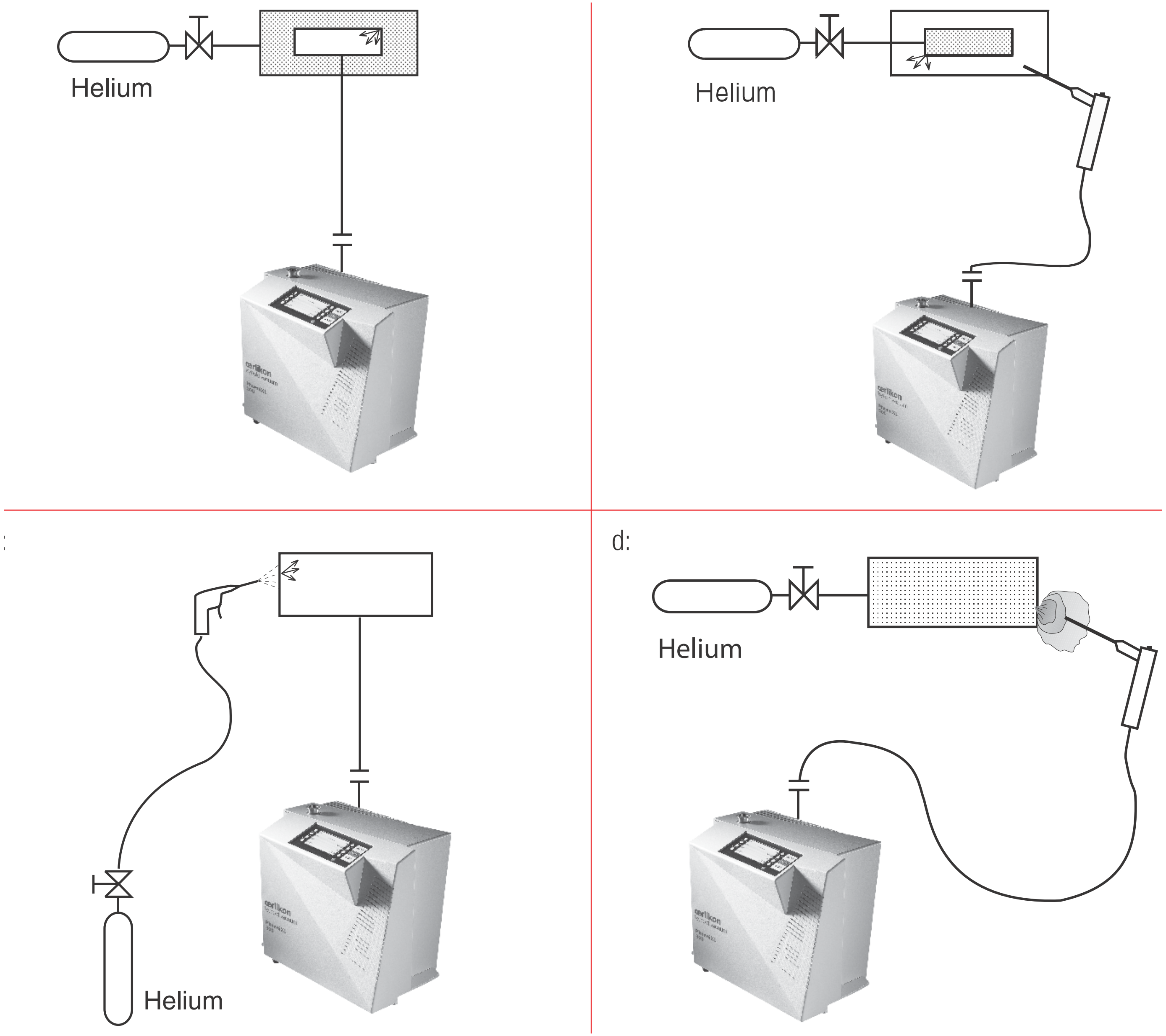


Outgasing

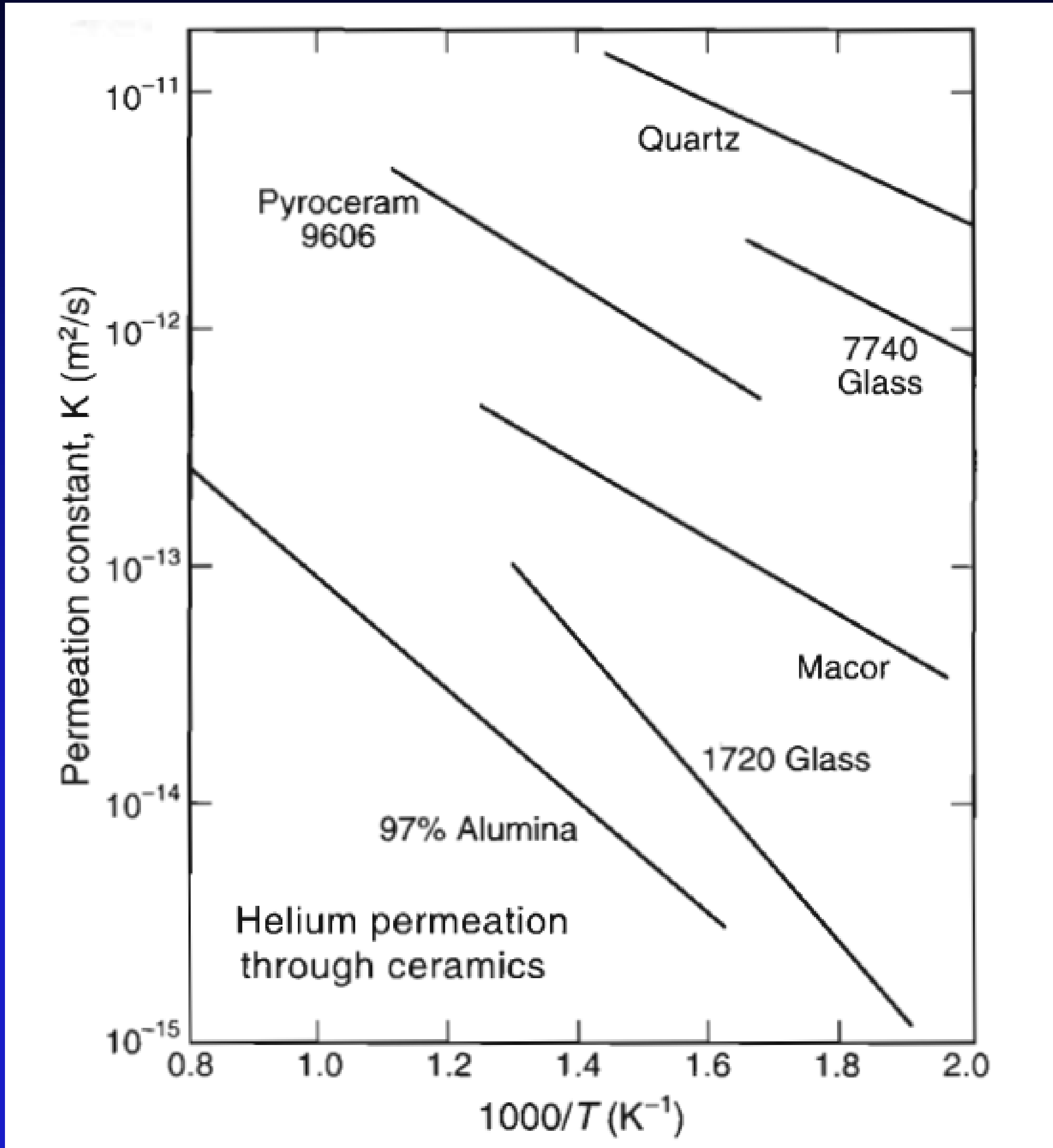


10^{-1}
10^{-2}
10^{-3}
10^{-4}
10^{-5}
Degassing rate [(Pa m³/s)/m²]
Neoprene
Araldite
Cast brass
Stainless steel
Mild steel
Aluminum
1
10
100
Time (h)

Leak Test Schemes



Permeation

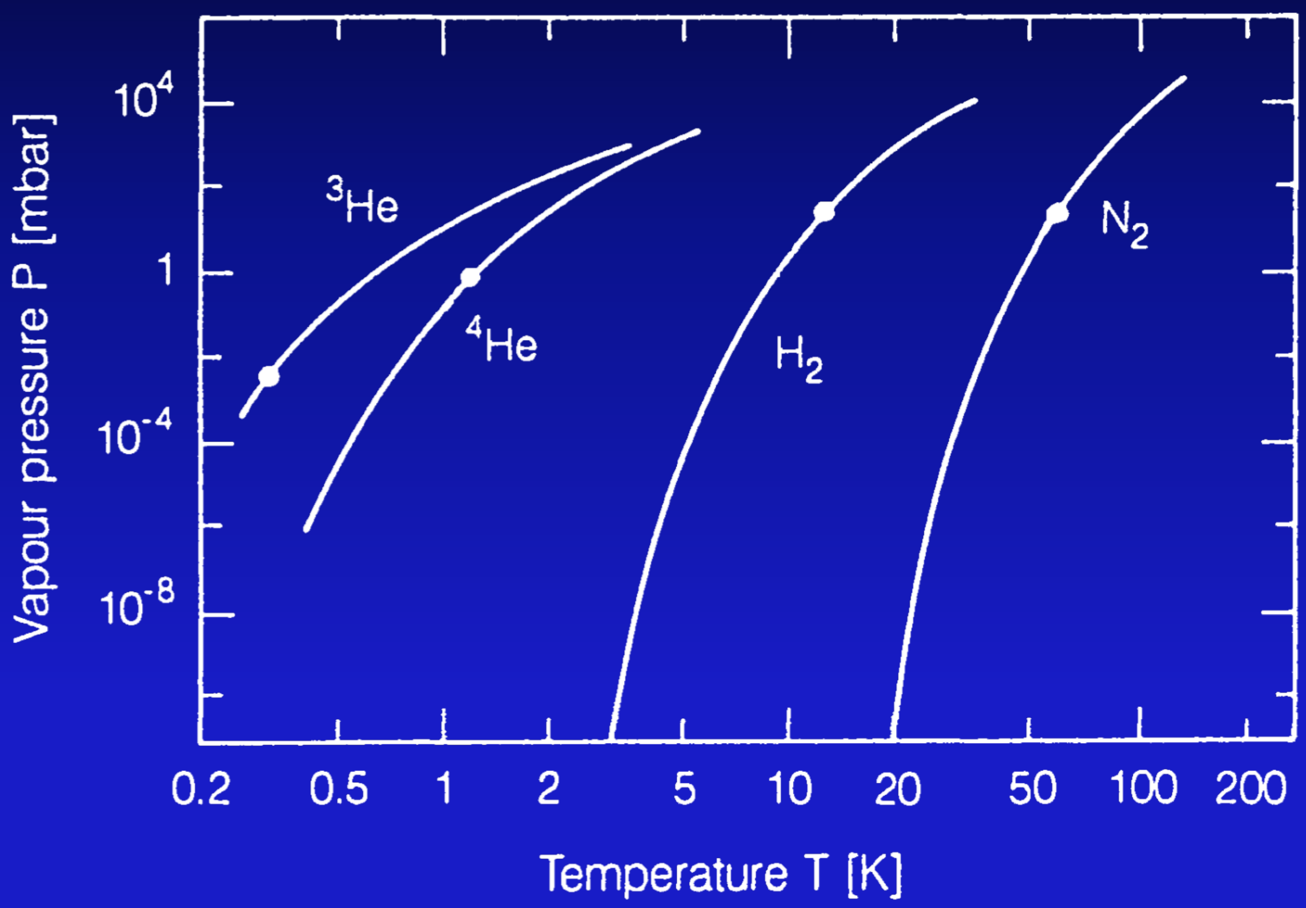


Quartz
Pyroceram 9606
7740 Glass
Macor
1720 Glass
97% Alumina
Helium permeation through ceramics
Permeation constant, K (m²/s)
10⁻¹¹
10⁻¹²
10⁻¹³
10⁻¹⁴
10⁻¹⁵
0.8
1.0
1.2
1.4
1.6
1.8
2.0
1000/T (K⁻¹)

Permeation

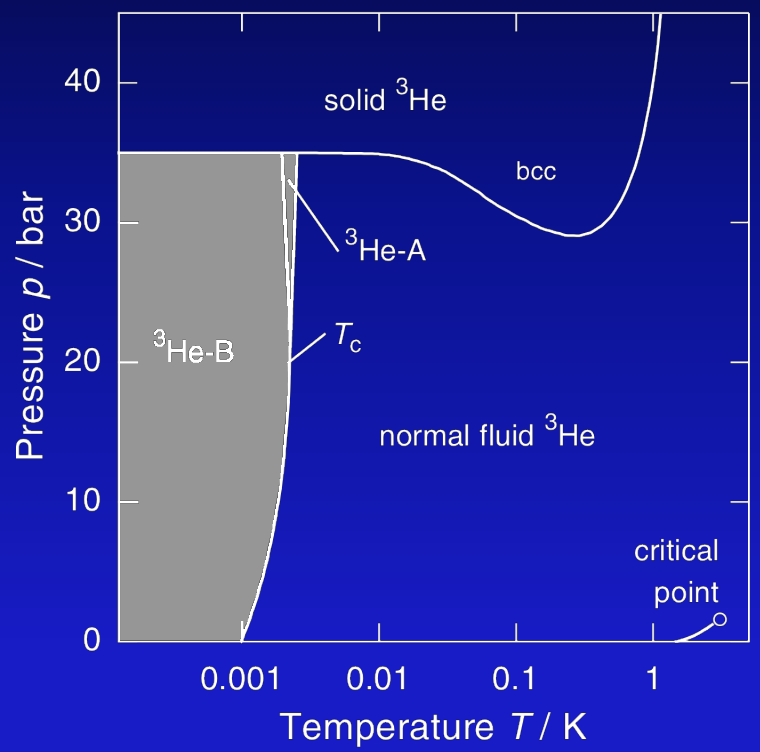
Cryoliquids

Substance	T_b [K]	T_m [K]	T_{tr} [K]	P_{tr} [bar]	T_c [K]	P_c [bar]	Latent heat L [kJ/ℓ]	Vol.% in air
H_2O	373.15	273.15	273.16	0.06	647.3	220	2252	--
Xe	165.1	161.3	161.4	0.82	289.8	58.9	303	$0.1\cdot10^{-4}$
Kr	119.9	115.8	114.9	0.73	209.4	54.9	279	$1.1\cdot10^{-4}$
O_2	90.2	54.4	54.36	0.016	154.3	50.4	245	20.9
Ar	87.3	83.8	83.81	0.67	150.9	48.7	224	0.93
N_2	77.4	63.3	63.15	0.12	126.0	33.9	160	78.1
Ne	27.1	24.5	24.56	0.43	44.5	27.2	110	$18\cdot10^{-4}$
D_2	23.7	18.7	18.72	0.17	38.3	16.6	50	--
H_2	20.3	14.0	13.80	0.07	33.3	13.0	31.8	$0.5\cdot10^{-4}$
^4He	4.21	--	--	--	5.20	2.28	2.56	$5.2\cdot10^{-4}$
^3He	3.19	--	--	--	3.32	1.16	0.48	--



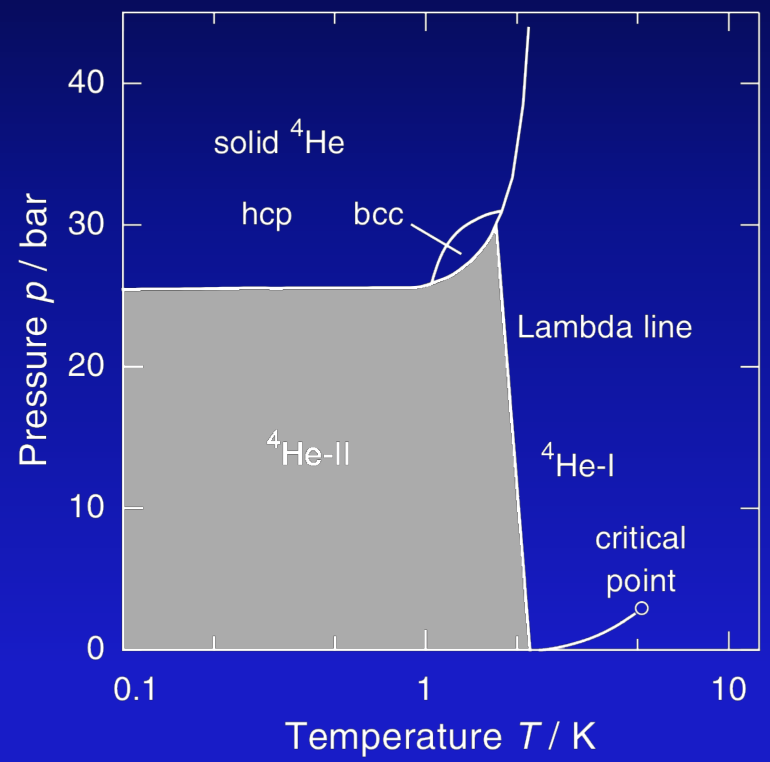
Vapour pressure P [mbar]
10^4
1
10^-4
10^-8
³He
⁴He
H₂
N₂
0.2
0.5
1
2
5
10
20
50
100
200
Temperature T [K]

^3He

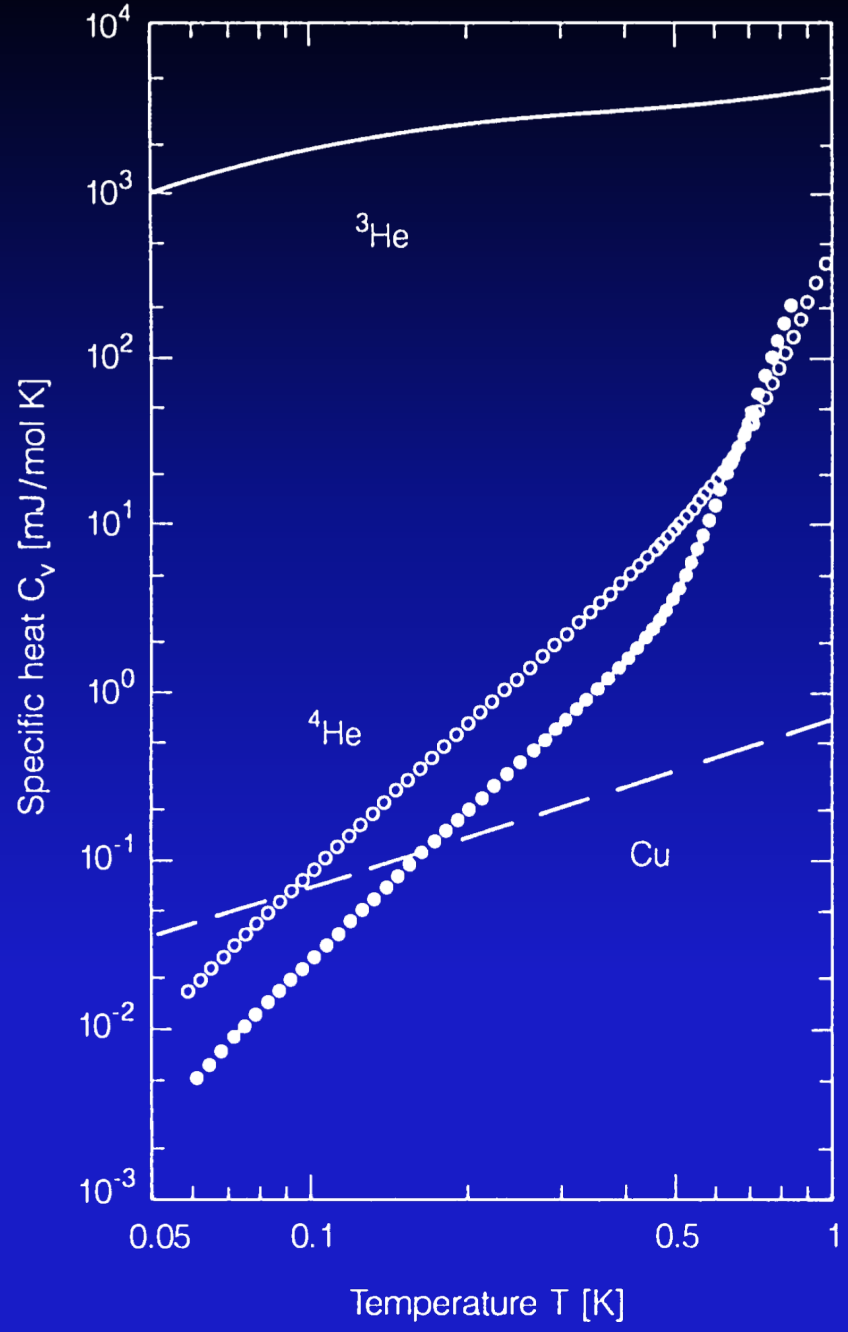


solid ^3He
bcc
^3He-A
^3He-B
T_c
normal fluid ^3He
critical point
Pressure p / bar
0
10
20
30
40
0.001
0.01
0.1
1
Temperature T / K

^4He



solid ^4He
hcp
bcc
Lambda line
^4He-II
^4He-I
critical point
Pressure p / bar
0
10
20
30
40
0.1
1
10
Temperature T / K



10^4
10^3
10^2
10^1
10^0
10^-1
10^-2
10^-3
Specific heat C_v [mJ/mol K]
3He
4He
Cu
0.05
0.1
0.5
1
Temperature T [K]

Thank you for your attention