Vacuum Technology

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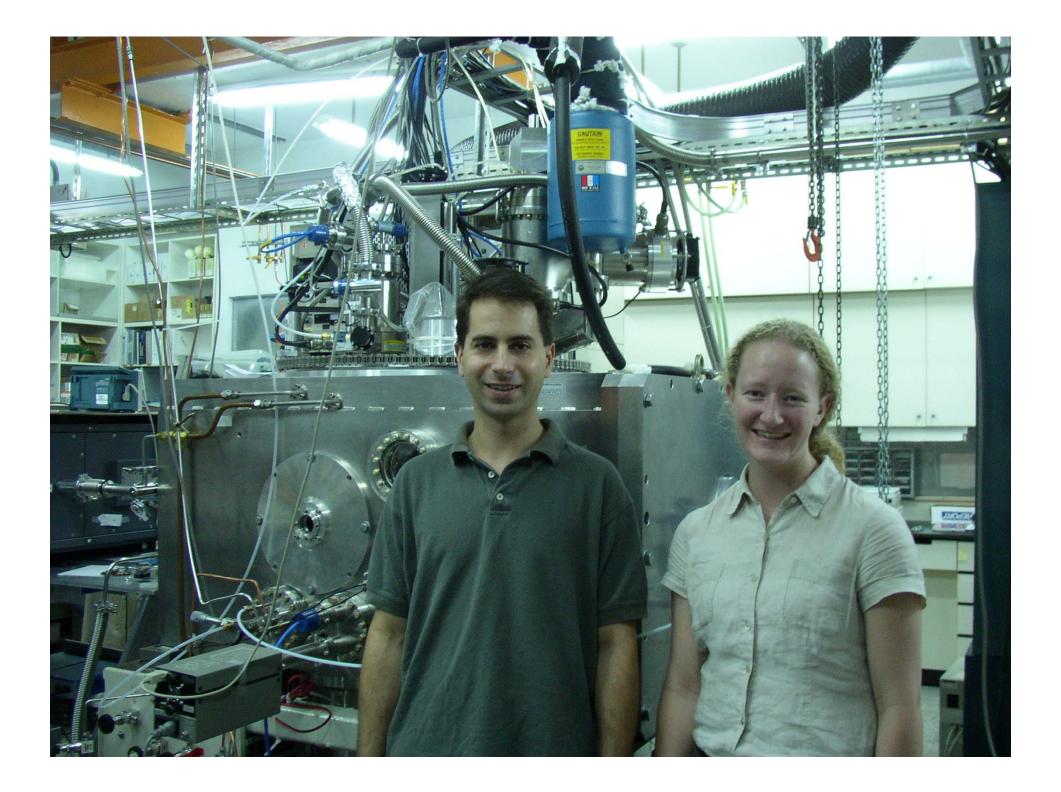
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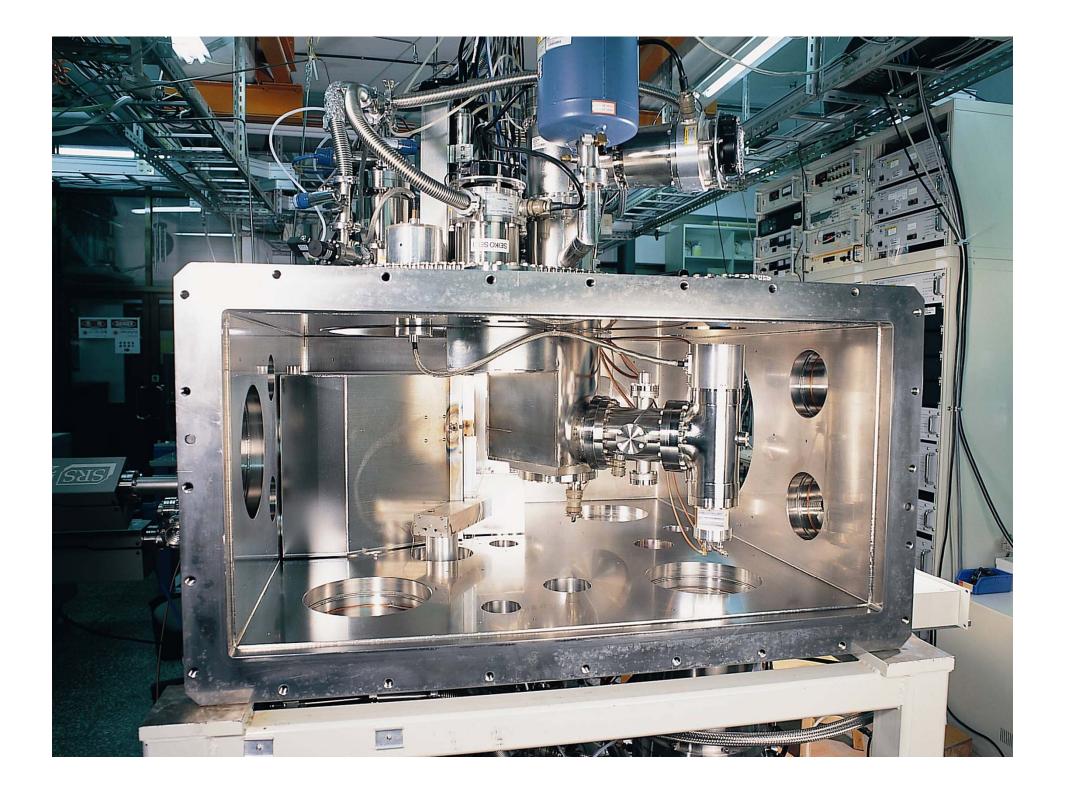
A very useful reference book:

Building Scientific Apparatus

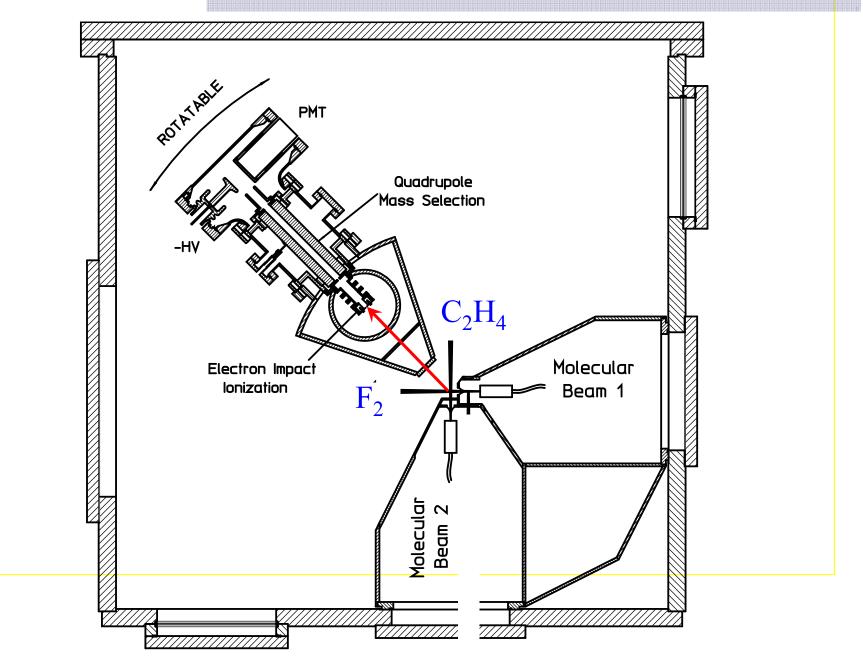
A practical guide to design and construction

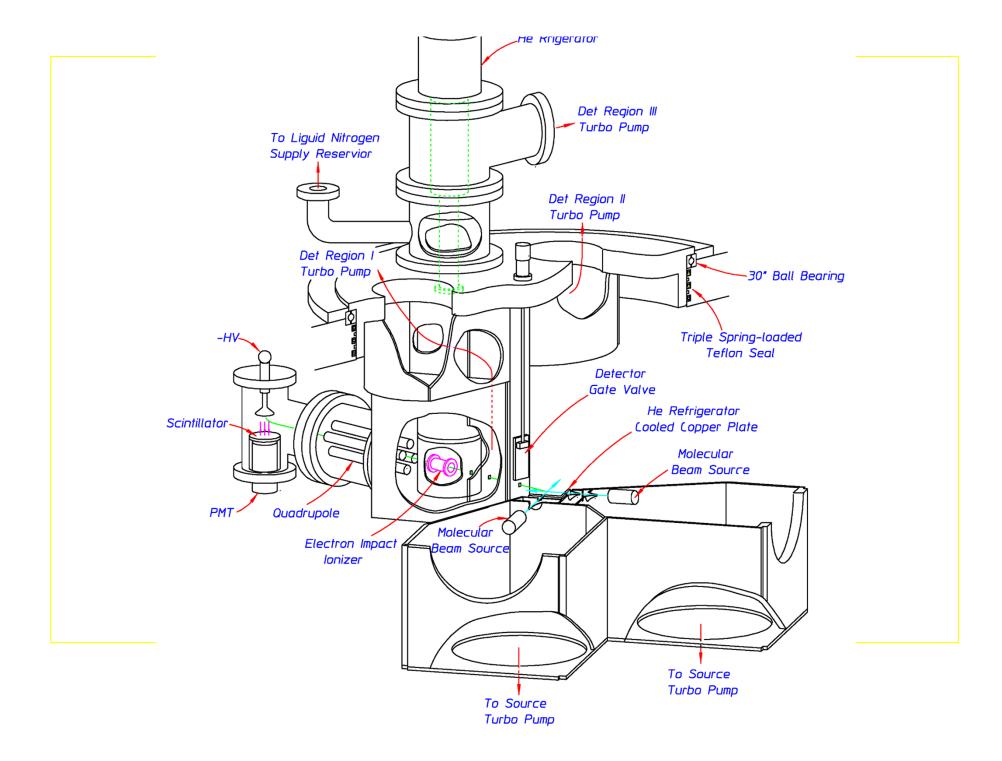
by John H. Moore, C. C. Davis and M. A. Coplan 2ed, 3rd or 4th edition





Horizontal Cut View of the Crossed Beam Chamber at R410, IAMS





Outlines:

Mean Free Path of gas molecules: Viscose flow Vs. Molecular flow Gas flow: Throughput, Conductance, Pumping speed, time **Pumps:** Mechanical pump, Roots, Turbo, Diffusion, Dry pumps **Gauges:** Mechanical, Thermal conductance, Ionization, **Chambers:** Joints (metal, elastomer), parts **Practical concerns:** Surface Material: SUS, Al alloy, ceramic, plastic, Sealing, Virtual leak **Baking** Leak test UHV

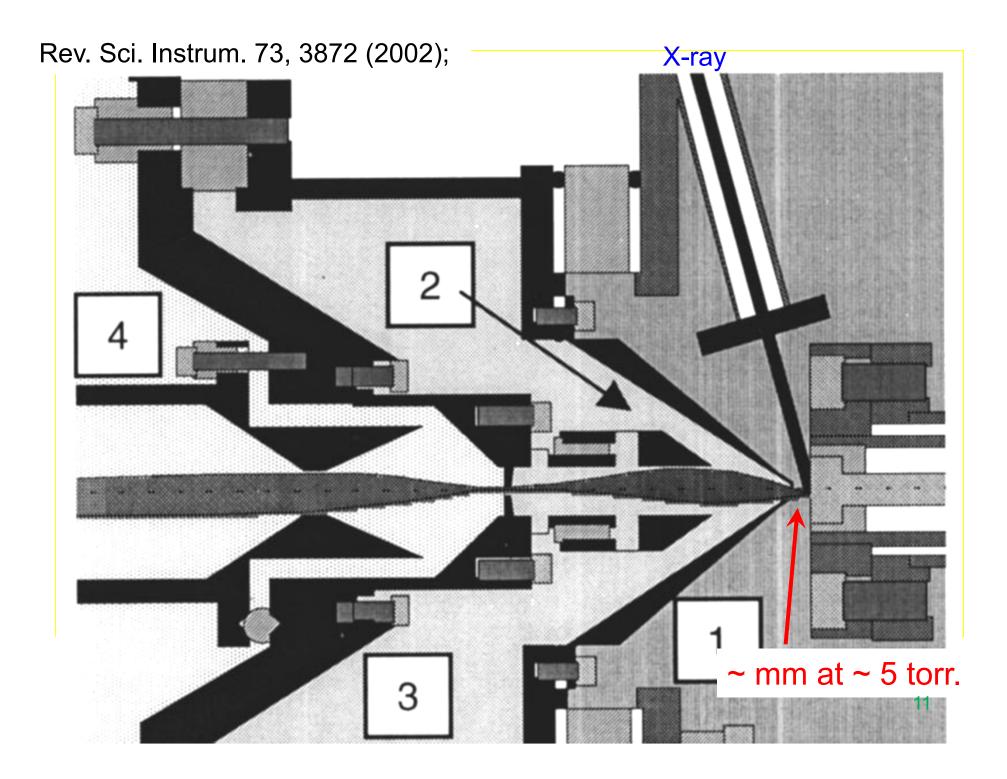
Mean Free Path: 平均自由徑

The average distance that *a molecule* travels between successive collisions.

Speed of gas molecules: for air (80% N₂ + 20% O₂) at 20 °C

If mean free path < container dimension ⇒ Viscous flow region Molecule motion is dependent on each other Molecule-molecule collisions dominate \Rightarrow *Flow dynamics* If mean free path > container dimension *⇒ Molecular flow* region Molecule motion is independent of each other Molecule surface collisions dominate \Rightarrow *Vacuum*

Rev. Sci. Instrum. 73, 3872 (2002) **Ambient Pressure Photoelectron Spectroscopy** For electrons at 500 eV, the mean free path is 2 mm at 1 torr. ELECTRON ANALYSER *The molecule mean free path* X-RAY SOURCE is much shorter! P = 760 torr $\lambda = 700 \text{ Å}$ T.RAYS $P = 1 \text{ torr} \quad \lambda = 50 \text{ } \mu\text{m}$ $P = 10^{-3}$ torr $\lambda = 5$ cm $P = 10^{-6}$ torr $\lambda = 50$ m SAMPLE for Molecules



Pumping speed:

$$PV = nRT$$
 $n \propto PV$ at $T = \text{const}$

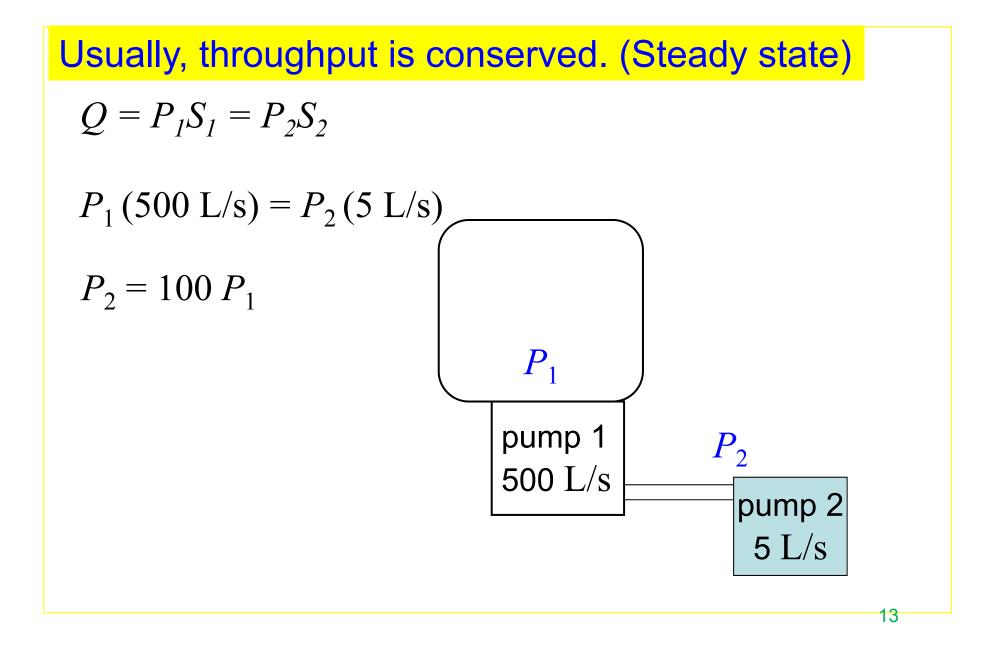
For a typical pump, the amount of gas being pumped out is proportional to *P*, such that the pumping speed *S* is defined as

$$\frac{\delta(PV)}{\delta t} = PS \quad S = \frac{1}{P} \frac{\delta(PV)}{\delta t}$$

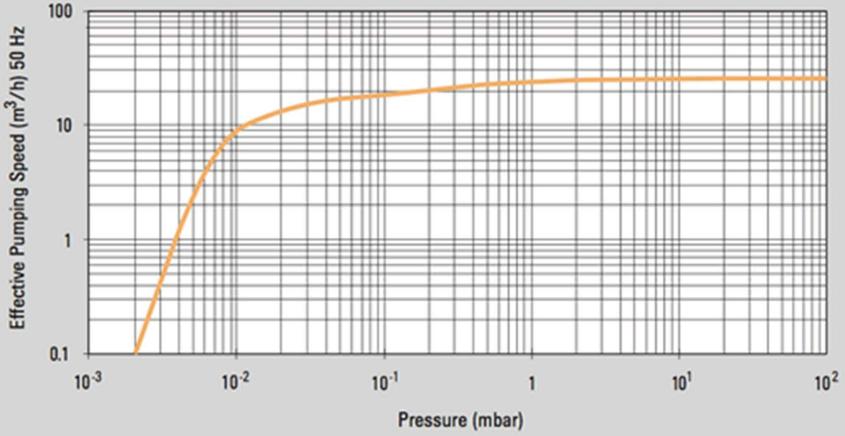
S in Liters per Second, L/s
P in torr·L/s

$$\frac{dn}{dt} \propto \frac{dPV}{dt} = Q = SP$$
at *T* = const

12

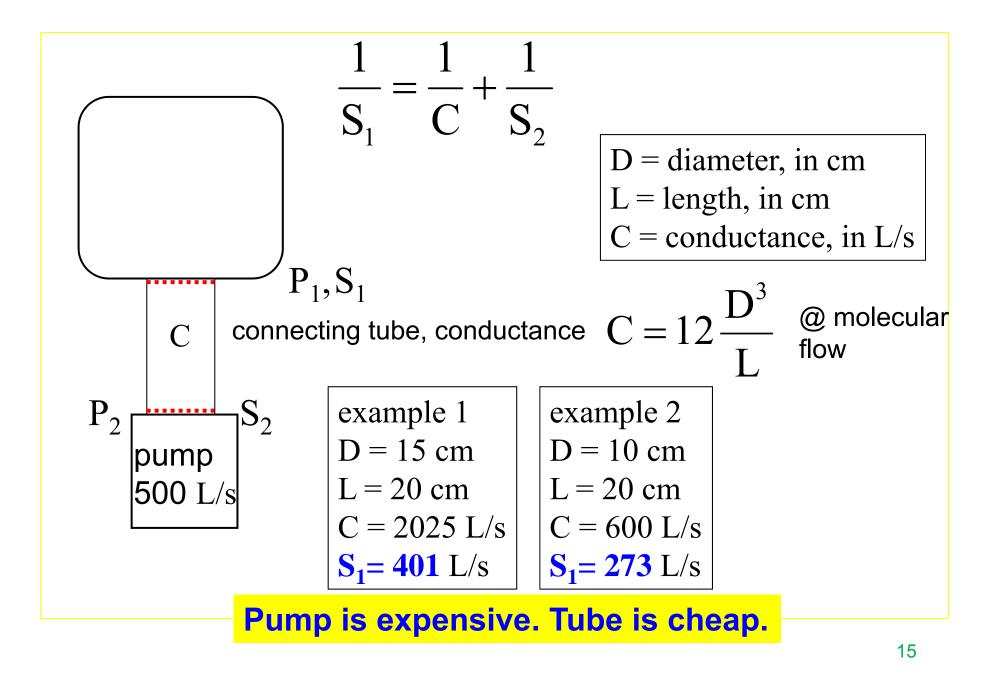


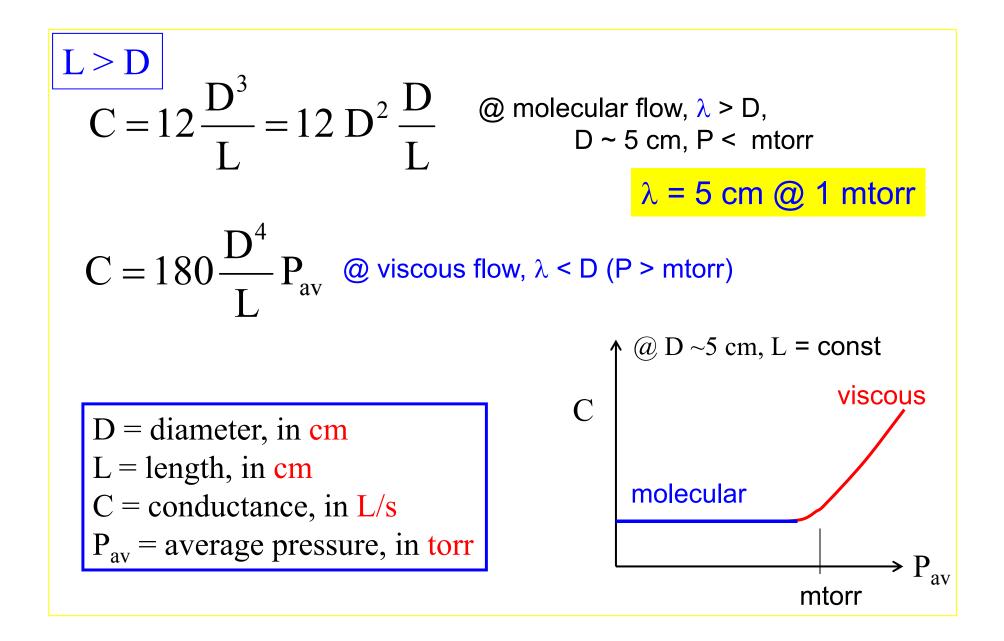
Pump Speed Curve





Agilent DS602 Dual Stage Rotary Van Pump



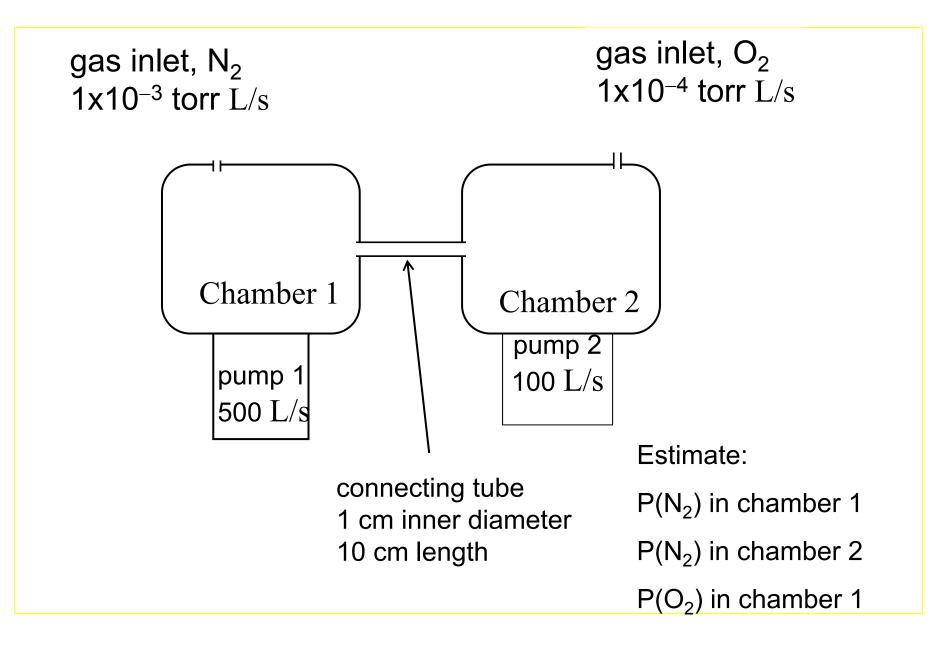


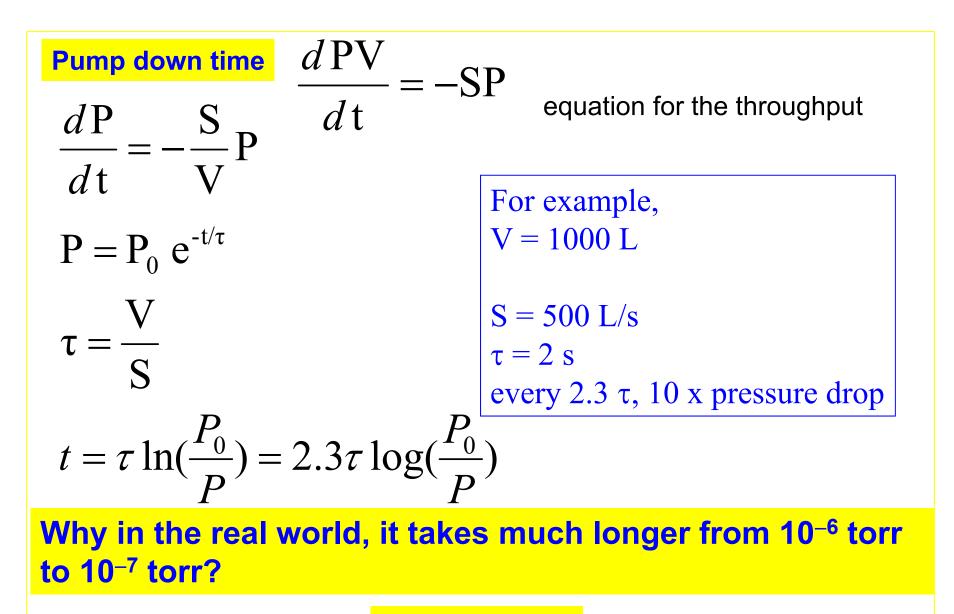
Serial Connection

$$\frac{1}{C} = \frac{1}{C_1} + \frac{1}{C_2}$$

Parallel Connection

$$\mathbf{C} = \mathbf{C}_1 + \mathbf{C}_2$$





Surface outgas

Pumps:

Mechanical: Oil rotary vane, Roots Dry vane, Scroll, Diaphragm

Diffusion

Turbo

Ion & Ti Sublimation

Cryo

Pumping Speed

Working pressure range

Compression Ratio

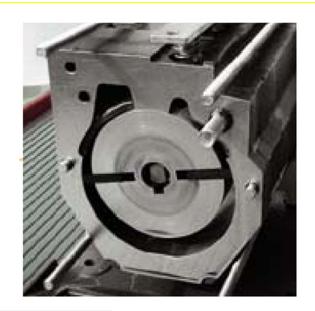
Gas species dependent

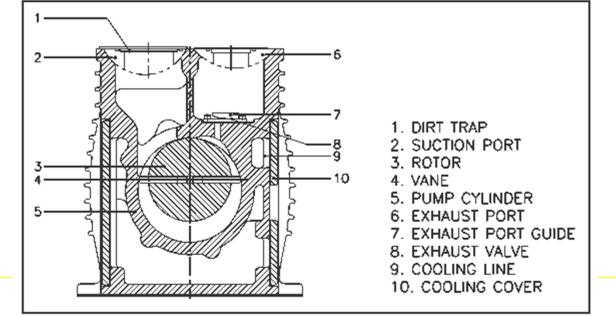


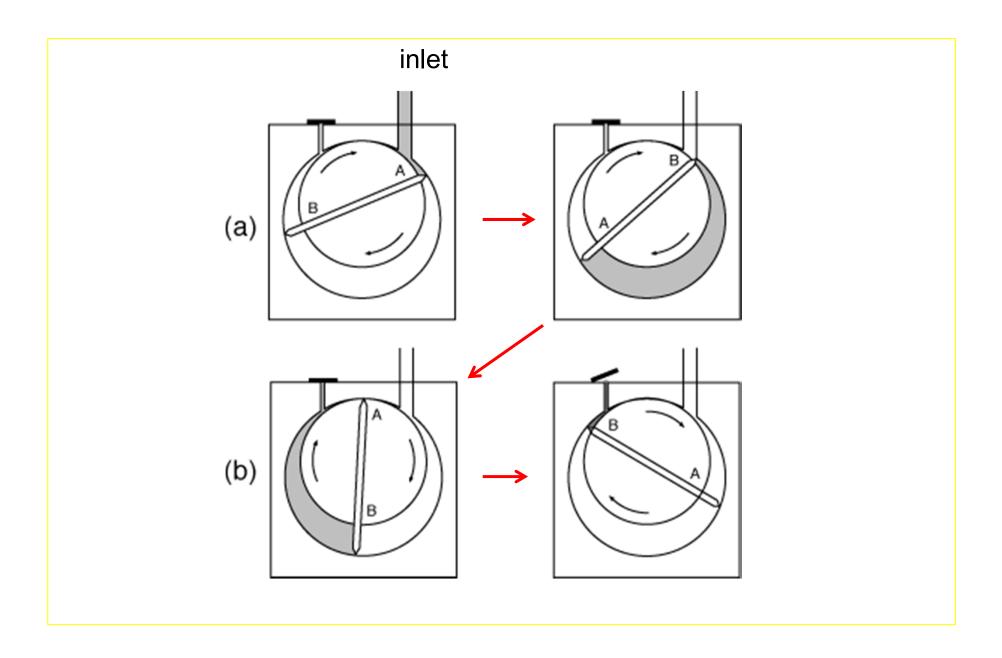
Rotary vane pump

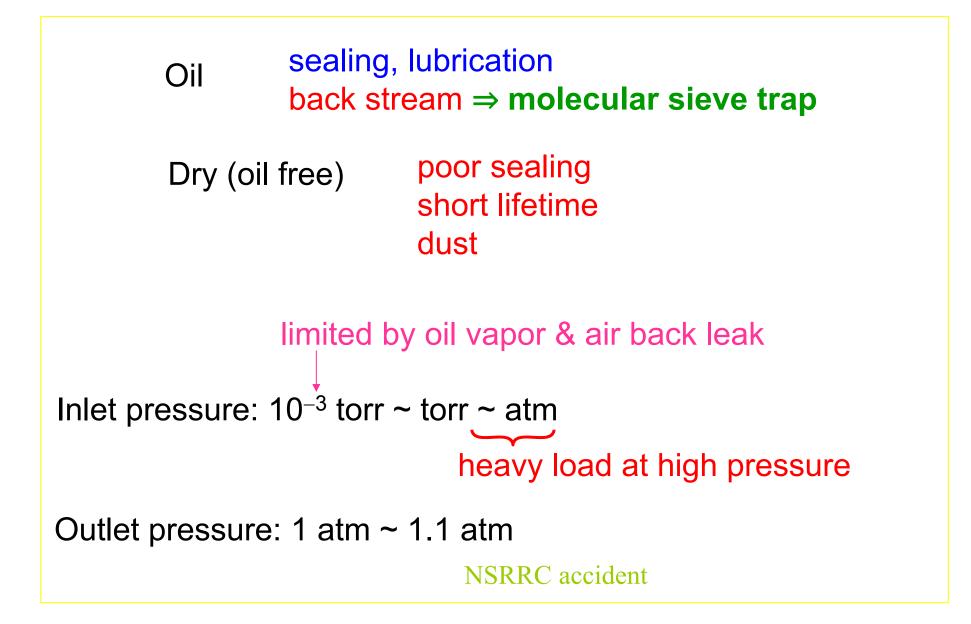
Mechanical Pump

single stage: down to 10^{-1} torr two stage: down to 10^{-3} torr

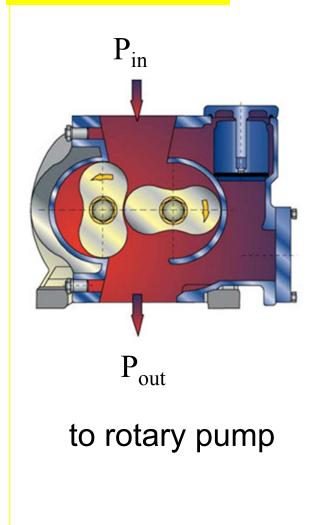








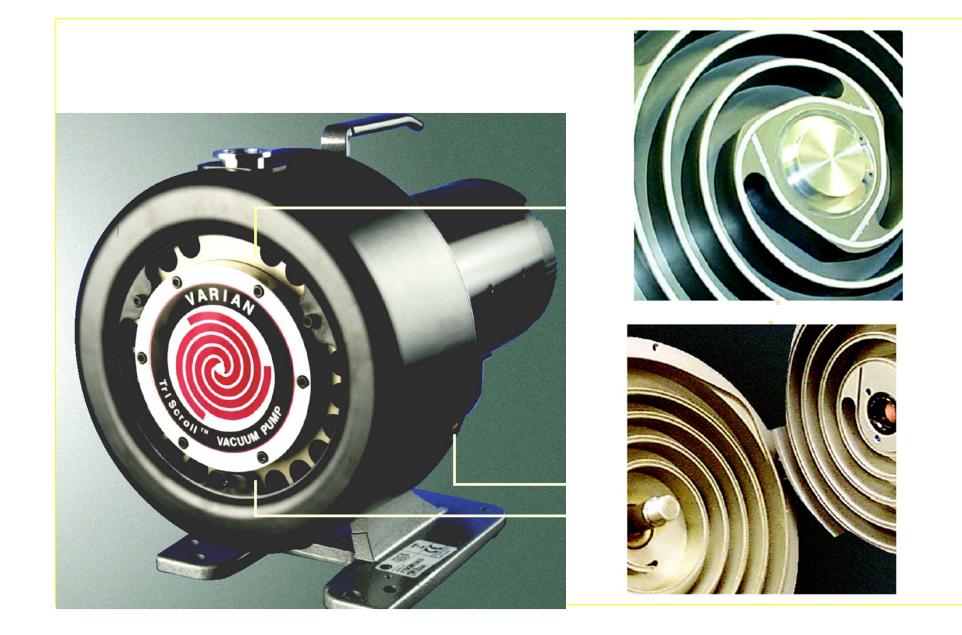
Roots blower



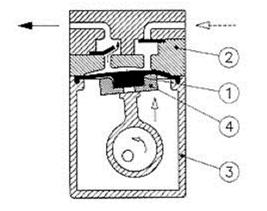
Large pumping speed Low compression ratio ~ 10 Compression ratio $K = \frac{P_{out}^{eq}}{P_{in}^{eq}}$ @Q_{net} =0Net throughput $Q_{net} = P_{in} S_{for} - P_{out} S_{back}$ If P_{out} is high, Q_{net} can be reduced to 0 Here P_{out}/P_{in} is at maximum and $K = P_{out}/P_{in} = S_{for}/S_{back}$

Scroll pump Dry (24hr/day)(365day/yr) = 8760 hr/yrShorter maintenance interval (5000 hrs) Poorer corrosive resistance Inlet pressure: 10^{-3} torr ~ torr ~ atm Outlet pressure: 1 atm ~ 1.1 atm Gas Inlet Compression **Orbiting scroll** Outlet pocket Suction process Exhaust process Fixed scroll Compression process (a) (Suction completed) (d) (c)

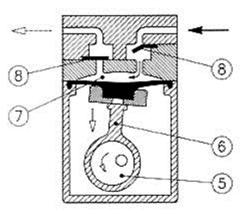
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EXHAUST STROKE



INLET STROKE



- Diaphragm
- Intermediate plate
- ③ Pump housing
- ④ Support
- ⑤ Eccentric
- ⑥ Connecting rod
- ⑦ Working chamber
- ⑧ Valve plate



Dry; Small; Low power consumption Low pumping speed

Low compression ratio

Poor ultimate pressure: ~ torr

Diaphragm outgas

Diaphragm crack

1–4 stages are available

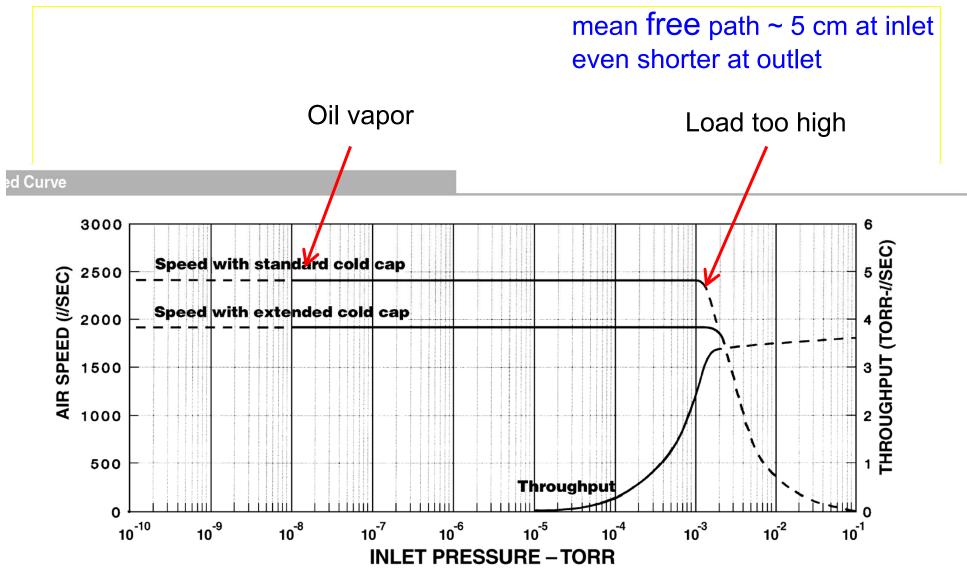


Oil diffusion pump

mechanism: momentum of oil molecules >> momentum of gas molecules pumped water cooling coil $10^{-3} \sim 10^{-1}$ torr Cheap, Robust Oil back stream Slow heat up (~0.5 hr) & cooling down (~2 hr) Large electric power consumption

 $10^{-4} \sim 10^{-7}$ torr

heater ~ 200 °C



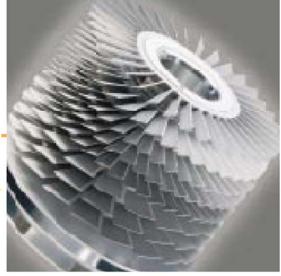
^r Refer to page 50 for a description of speed test.

Turbo Molecular Pump

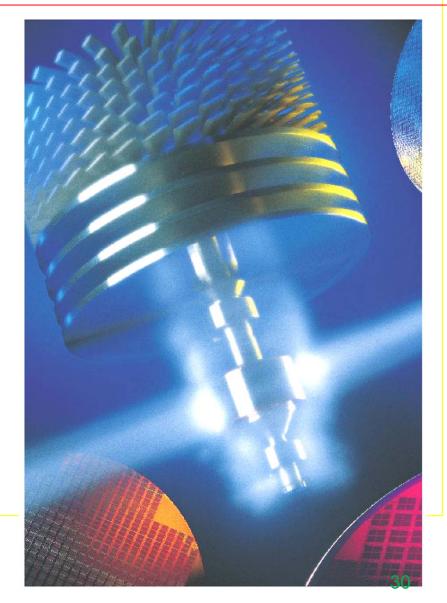
- High Speed Fan (rotor)
- momentum transfer
- blade speed ~ molecular speed

$$\overline{v} = \sqrt{\frac{8kT}{\pi m}} = 500 \, \frac{m}{s} \text{ for air}$$

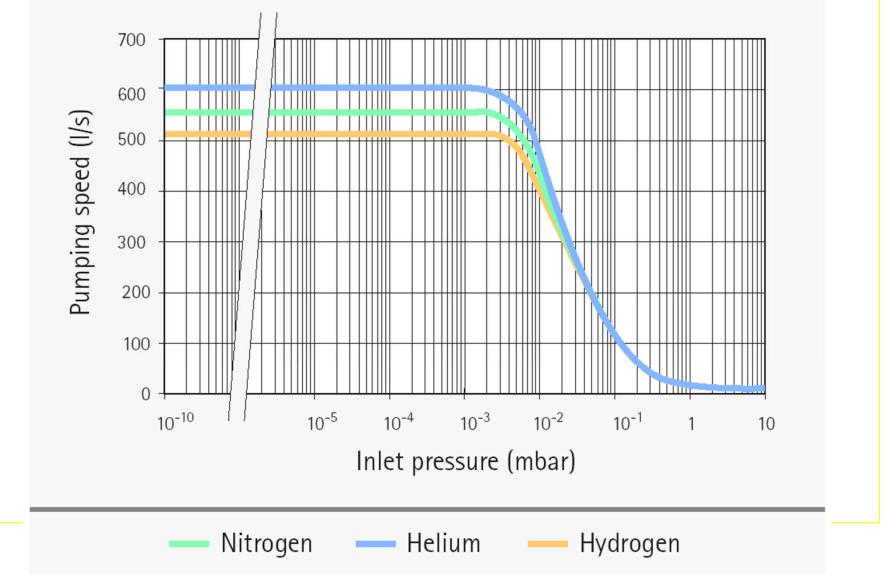


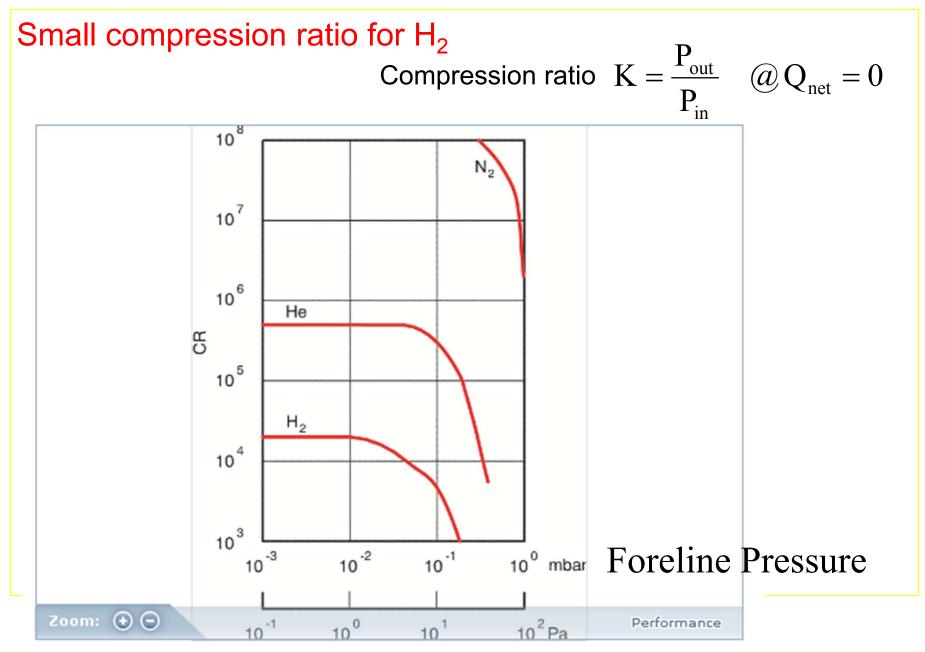


There are stators to block bouncing back (not shown)



Nitrogen Pumping Speed vs Inlet Pressure (DN 160 only)





TurboConvenient, reliable now, small sizeBakable to 125 °C

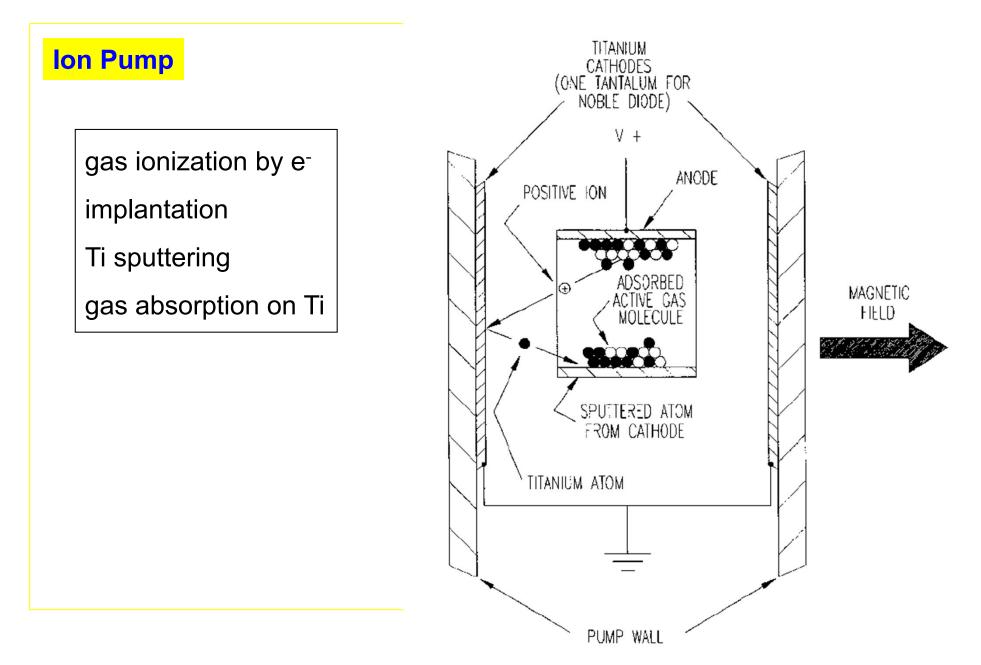
Low compression ratio for H_2 , $10^3 \sim 10^5$

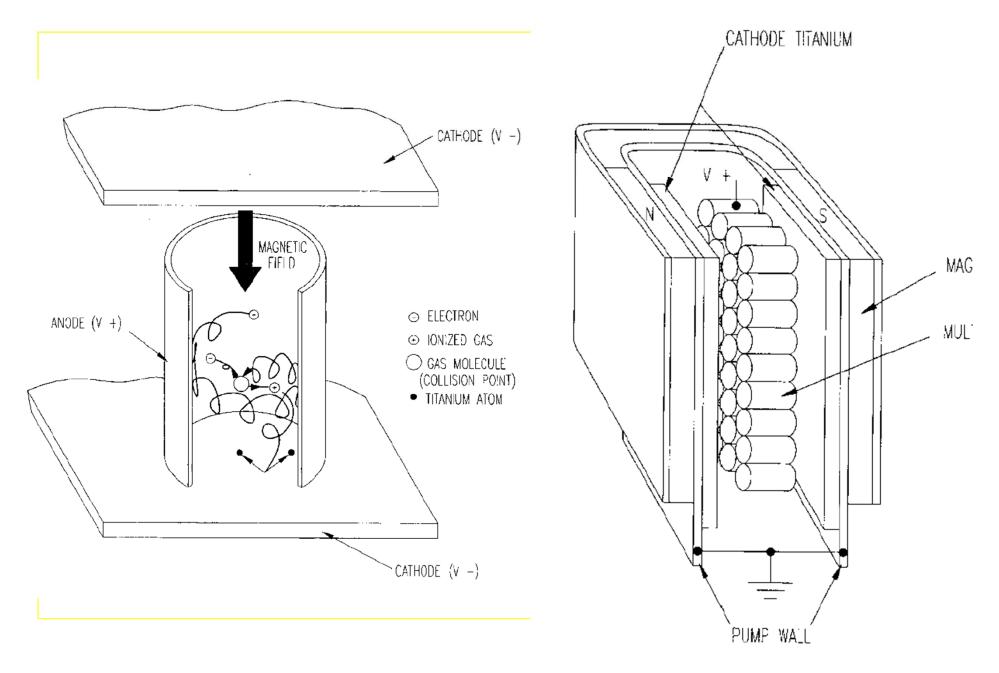
Outgas from lubricant and wires

magnetic bearing maintenance free

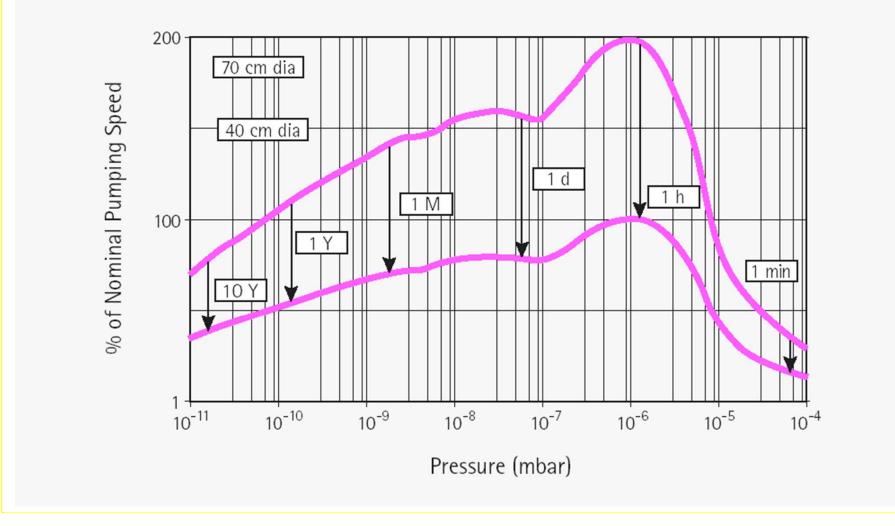
Corrosive resistant

Foreline Pressure determines its load.





Saturation Effect



Ion Pump and Ti Sublimation Pump No foreline needed Ti sorption pumping when electric power is off Robust Saturation effect, only for very small load Memory effect, outgas Gas species dependent

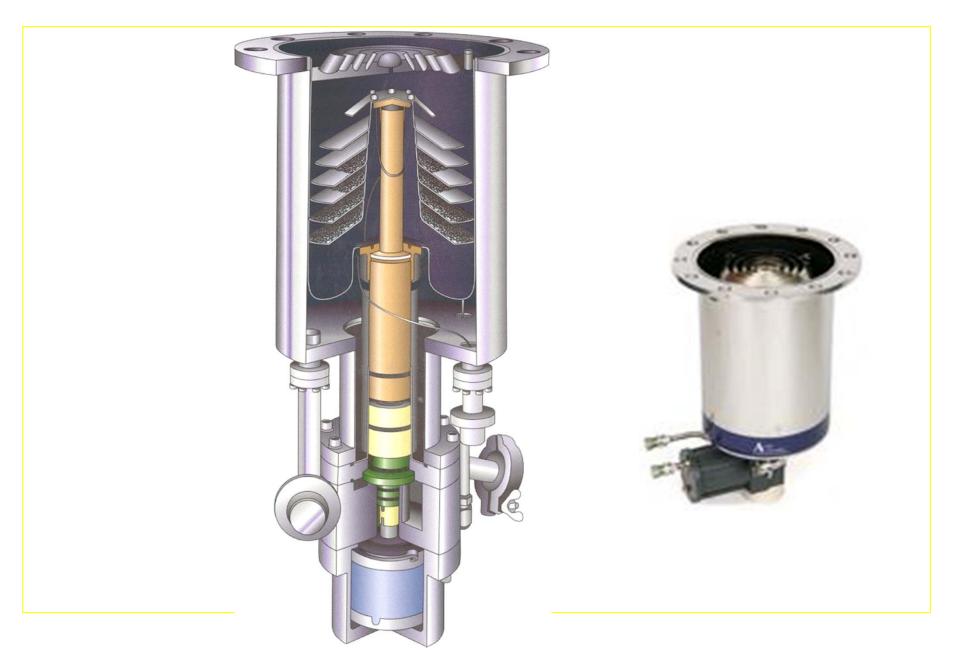
Cryopump

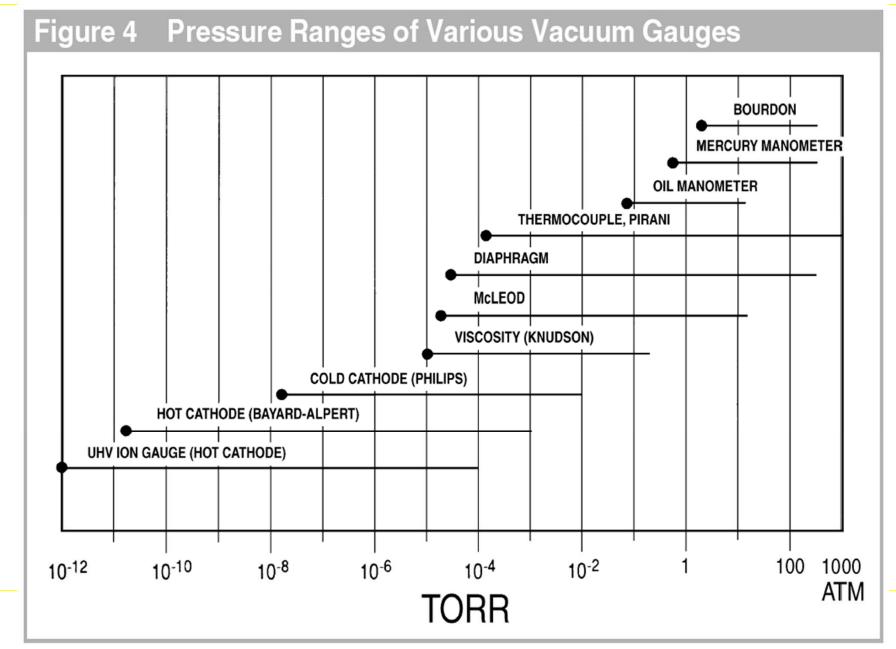
Gas condensation on ultracold surfaces < 20K activated carbon to absorb H_2 , Ar, N_2 , O_2 , etc < 80K metal surface to absorb H_2O , etc Large pumping speed for condensing gases No fore line needed Corrosive resist Gas species dependent: No pumping for He, Ne

Saturation effect, Memory effect

Huge outgas when warm

gate valve recommended





Gauge types

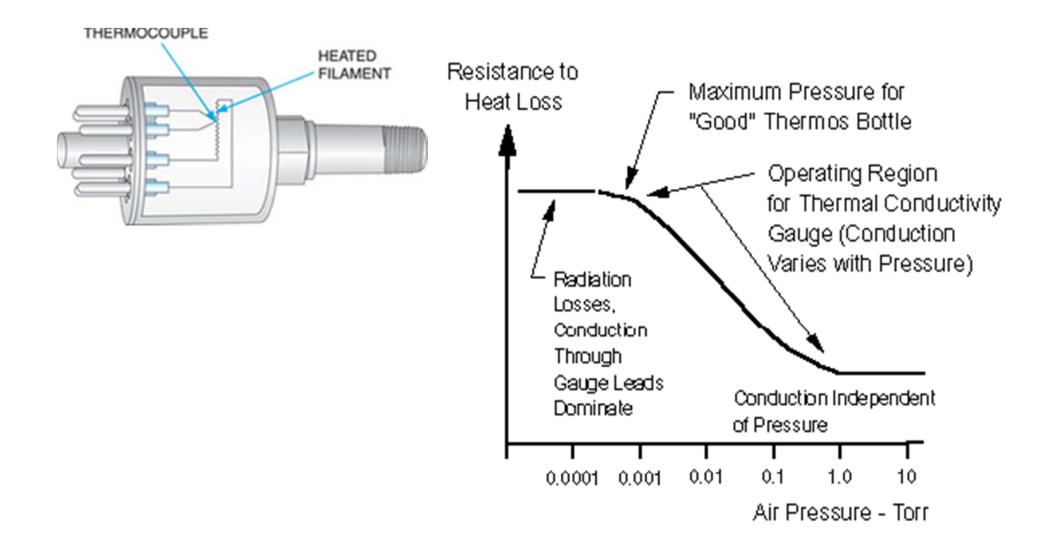
Mechanical: liquid column, diaphragm, etc

psig Vs. psia

Real pressure from force measurement

Thermal Conductance:Thermal Couple (TC)
thermo resister (Pirani)
convectioncheap, wide range 106
nonlinear sensitivity
zero point is not absolute
species dependentIonization: $e^- + M \rightarrow M^+ + 2e^-$ linear sensitivity
absolute zero point
species dependentIonization: $e^- + M \rightarrow M^+ + 2e^-$ linear sensitivity
absolute zero point
species dependent

Principle of the Thermal Conductivity Gauge



42 Copyright 1994-1996, záz Bell Jar

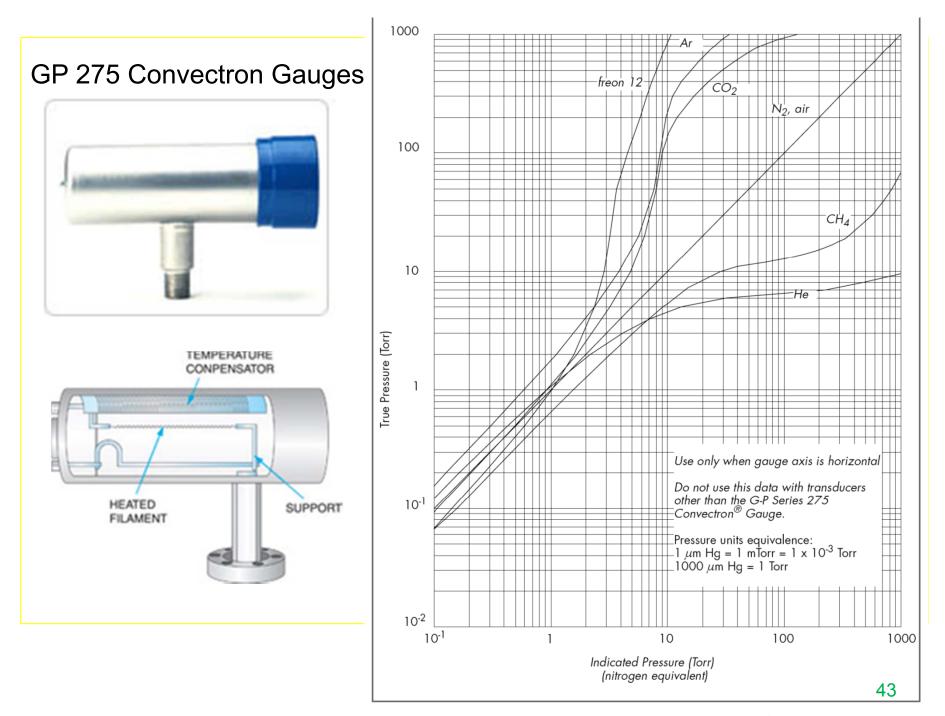
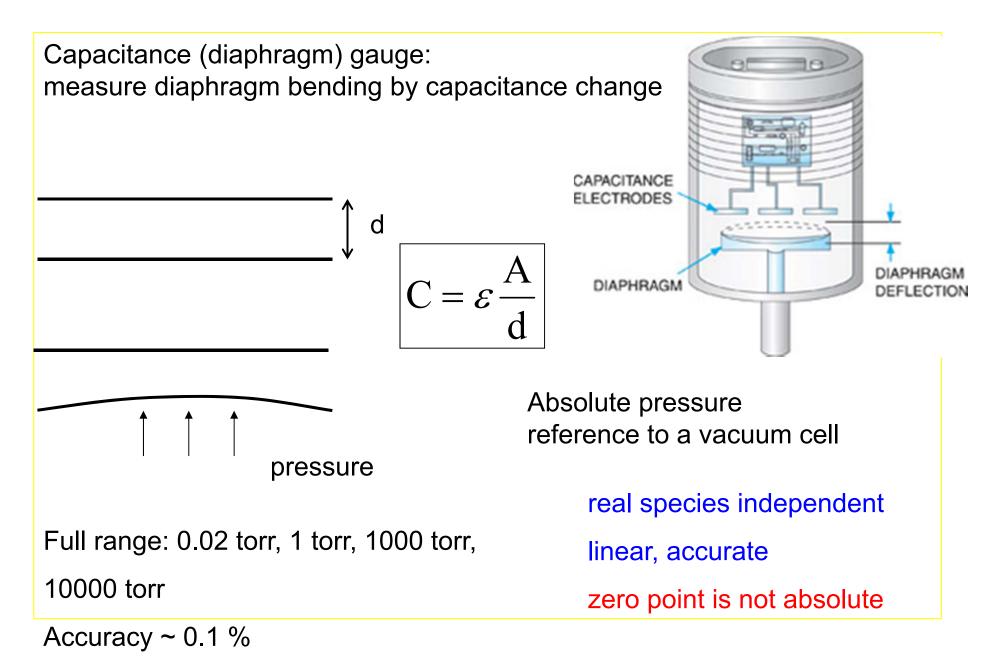
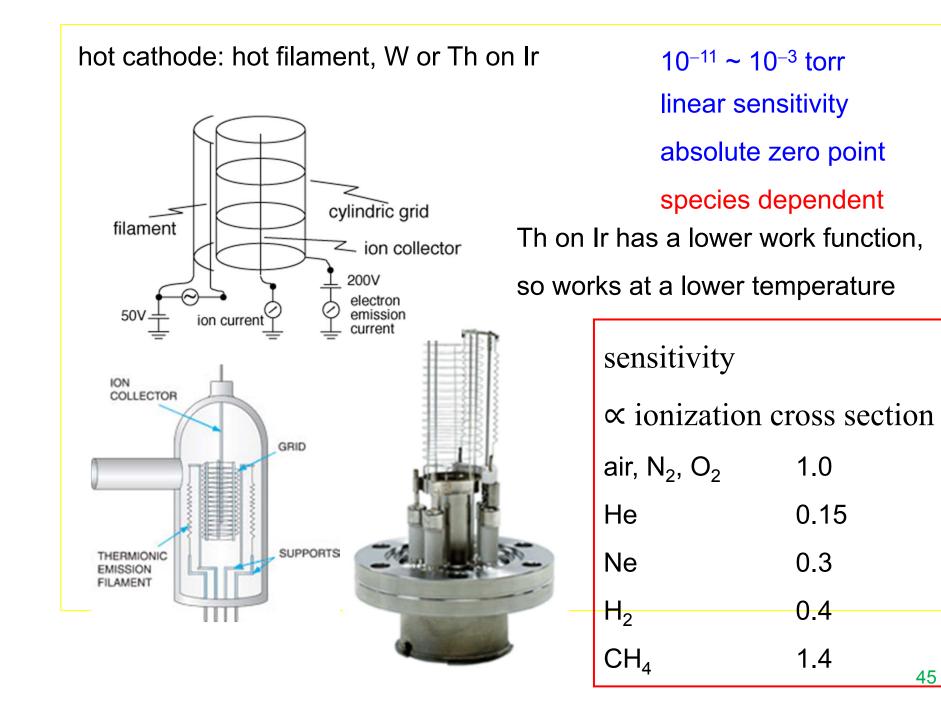
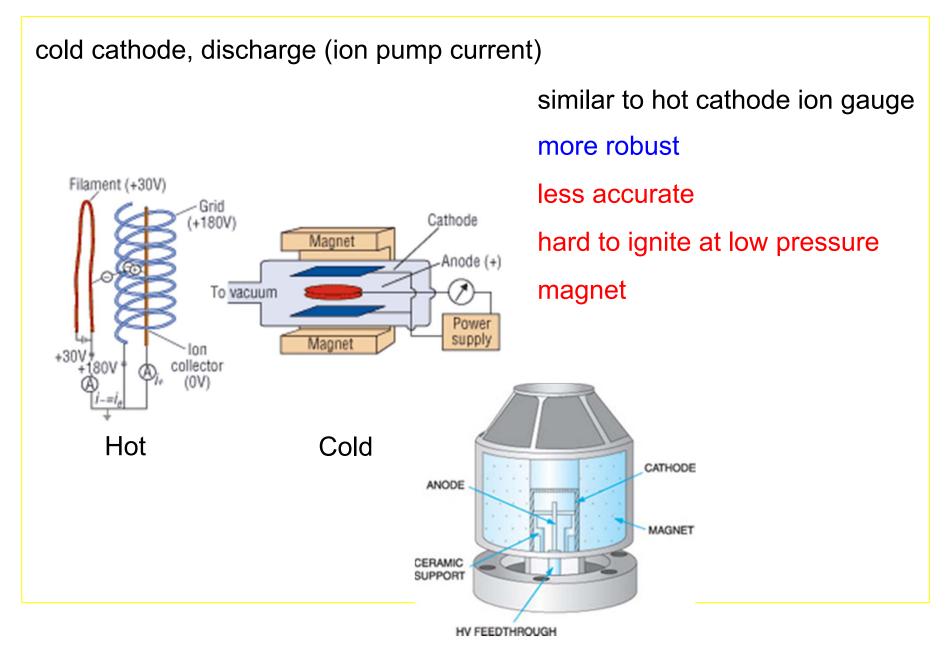


Figure 3-3 Convectron Gauge Indicated vs. True Pressure Curve; 10⁻¹ to 1000 Torr.

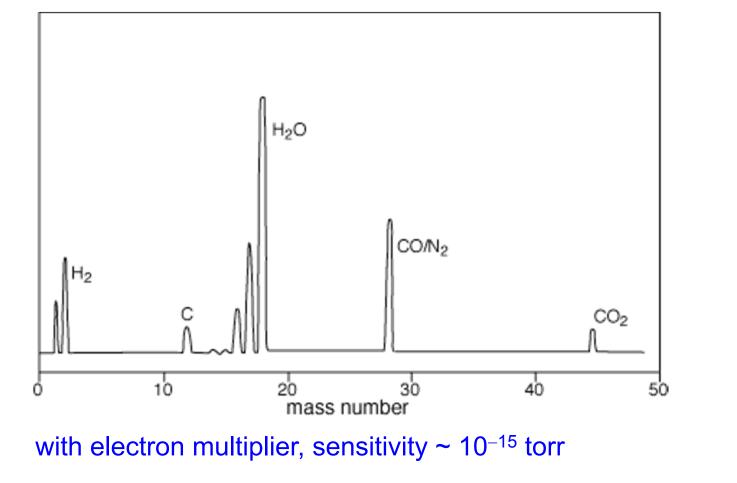






Residue Gas Analyzer (RGA)

A small quadrupole mass analyzer with an electron impact ionizer



A mixture of 10% C_3H_8 in He flows into a vacuum chamber which is pumped by a turbo pump. The Ion Gauge reading is 1×10^{-5} torr.

- (a) What is the true pressure?
- (b) What is the pressure reading for a 30% C₃H₈ in He mixture under the same flow rate?
- (c) What is the pressure reading for a 30% C_4H_{10} (butane, not on the table) in He mixture under the same flow rate?
- (d) What pressure gauge is independent on the gas species? How does it work?

Nominal Gas Correction Factors

Gas	R _g
He	.18
Ne	.30
D ₂	.35
H ₂	.46
N ₂	1.00
Air	1.0
O ₂	1.01
СО	1.05
H ₂ O	1.12
NO	1.15
NH ₃	1.23
Ar	1.29
CO ₂	1.42
CH ₄ (methane)	1.4
Kr	1.94
SF ₆	2.2
C ₂ H ₆ (ethane)	2.6
Xe	2.87
Hg	3.64
C ₃ H ₈ (Propane)	4.2

Where to get information? Wiki, Google, ... Manufacturer: catalog, manual, ... Experienced users: ... (salesman?)

Thinking: operation principle, ...

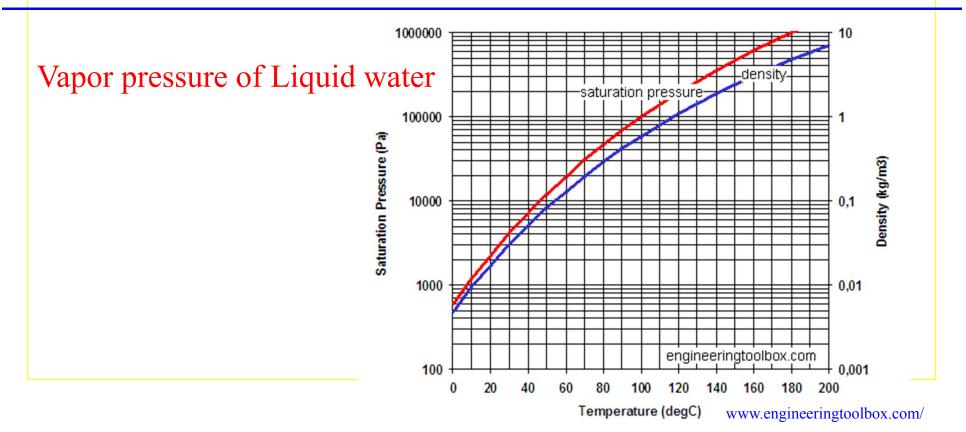
Chamber: 方 or 圓





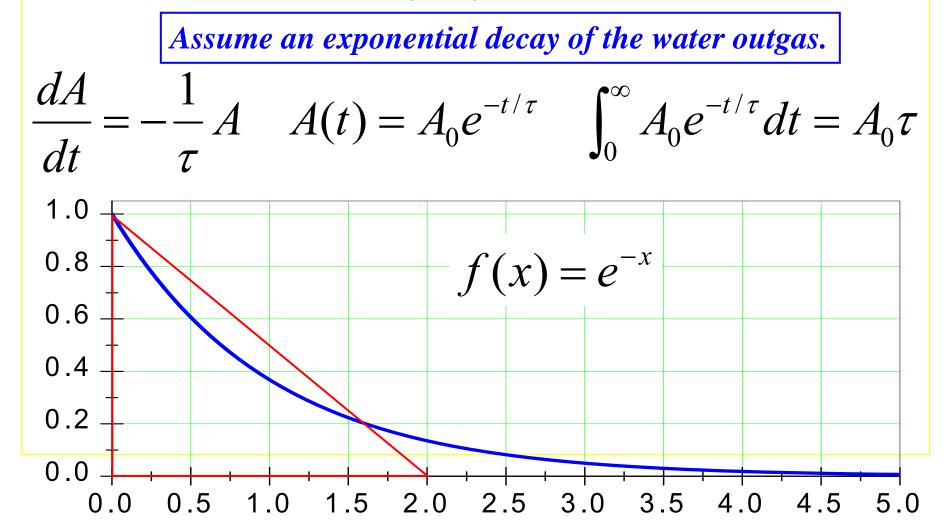
Surface Outgas Concern

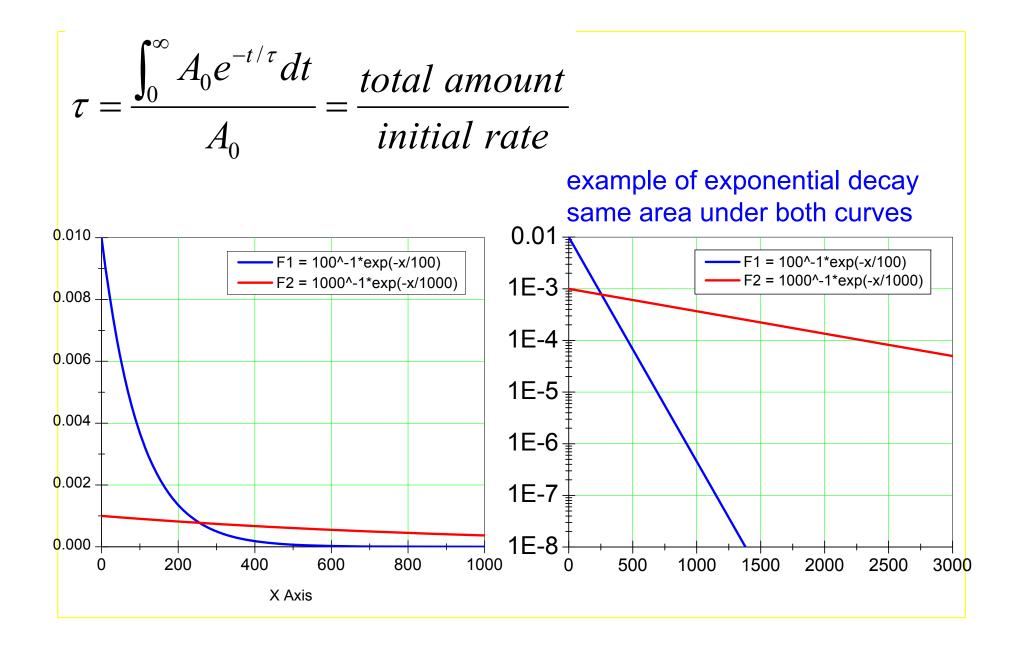
For a normal vacuum chamber that has no leak, it is known that water outgas from the surface is the major source of residue gases at a pressure range of $10^{-9} - 10^{-6}$ torr.



			20	<i></i>	20.0	
	Vapour	Vapour	27	3.6	27.0	on wikingdig org/
Temperature	pressure	pressure	28	3.8	28.5	en.wikipedia.org/
(°C)	(kPa)	(mmHg)	29	4.0	30.0	
0	0.6	4.5	30	4.2	31.5	
3	0.8	6.0	32	4.8	36.0	
5	0.9	6.8	35	5.6	42.0	
8	1.1	8.3	40	7.4	55.5	1
10	1.2	9.0	50	12.3	92.3	
12	1.4	10.5	60	19.9	149.3	
14	1.6	12.0	70	31.2	234.1	
16	1.8	13.5	80	47.3	354.9	
18	2.1	15.8	90	70.1	525.9	
19	2.2	16.5	100	101.3	760.0]
20	2.3	17.3				
21	2.5	18.8				
22	2.6	19.5				
23	2.8	21.0				
24	3.0	22.5				
25	3.2	24.0				
26	3.4	25.5				
27	3.6	27.0				
28	3.8	28.5				
29	4.0	30.0				
30	4.2	31.5				
32	4.8	36.0				

If the water outgas pressure is 1×10^{-7} torr and it takes <u>a week</u> to pump down to 1×10^{-8} torr when the pumping speed is 1000 L/s, how much water has been pumped out?





$$\frac{dP}{dt} = -\frac{1}{\tau}P \qquad P = P_0 e^{-t/\tau}$$

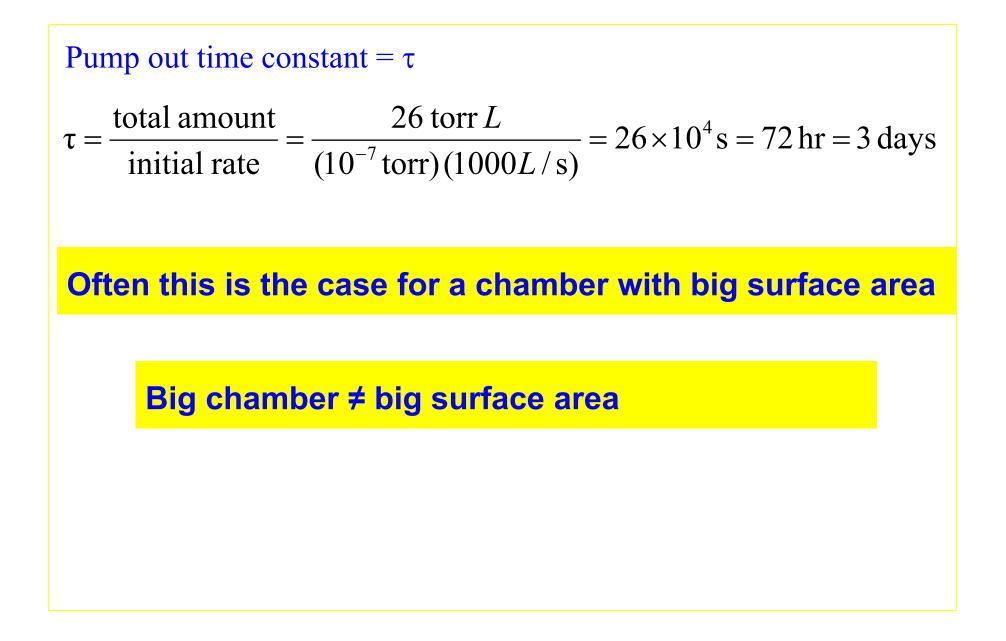
$$Q = PS; \quad \frac{dQ}{dt} = -\frac{1}{\tau}Q; \quad Q = Q_0 e^{-t/\tau}$$

$$\Delta Q = \int_{t_1}^{t_2} Q_0 e^{-t/\tau} dt = Q_0 \tau [e^{-t/\tau}]_{t_2}^{t_1} = \tau [Q_1 - Q_2]$$

$$= \tau S[P_1 - P_2]$$
If $P_1 = 10P_2, \tau = \Delta t/\ln 10 = (7 \times 24 \times 3600 \text{ s})/2.3 = 2.6 \times 10^5 \text{ s}$

$$\Delta Q = \tau S[P_1 - 0.1P_1] \approx 26 \text{ torr } L = 1.4 \times 10^{-3} \text{ mol} = 0.02 \text{ g}$$

A story about a rainy evening.



How to minimize surface area

Oxide could be porous

Remove thick surface oxide:

electro polish SUS chamber and parts basic wash (NaOH solution) Al alloy acid wash copper/brass parts sand blast

Dirty surface is thicker

Cleaning

Strong detergent is much more efficient than solvent

Estimate the effect of Baking

$$k(T) = Ae^{-E_a/RT}$$

When temperature rises to 100 °C, outgas rate rises by roughly two orders of magnitude, i.e., 10^{-5} torr instead of 10^{-7} torr

Initial pumping throughput is 10^{-5} torr * 1000 L/s = 10^{-2} torr L/s

$$\tau = \frac{2.6 \times 10^1 \operatorname{torr} L}{10^{-2} \operatorname{torr} L/s} = 2.6 \times 10^3 \operatorname{s} = 0.72 \operatorname{hr}$$

to P = 10⁻¹⁰ torr, P₀/P = 10⁵
$$\Delta t = \tau \ln \frac{P_0}{P} = 11.5 \tau = 8.3 \text{ hr}$$

Practically, it takes a little bit longer (≲100 hr)

∵ Single exponential delay is only an approximation

Deeper water has smaller outgas rate, thus longer $\boldsymbol{\tau}$

bake uniformly is important to avoid distortion

Aluminum foil on SUS chamber, heating tape on the aluminum

foil, another layer of aluminum foil to reduce heat loss

Degas ion gauge during baking

Clean ion gauge and its surrounding by excess heating

Don't bake oily surface.

 $oil \rightarrow tar$

Estimate the effect of using plastic parts

plastic may absorb H_2O to 1~2 % w/w

Assume 100 g plastic can absorb ~1.8 g $H_2O = 0.1$ mol

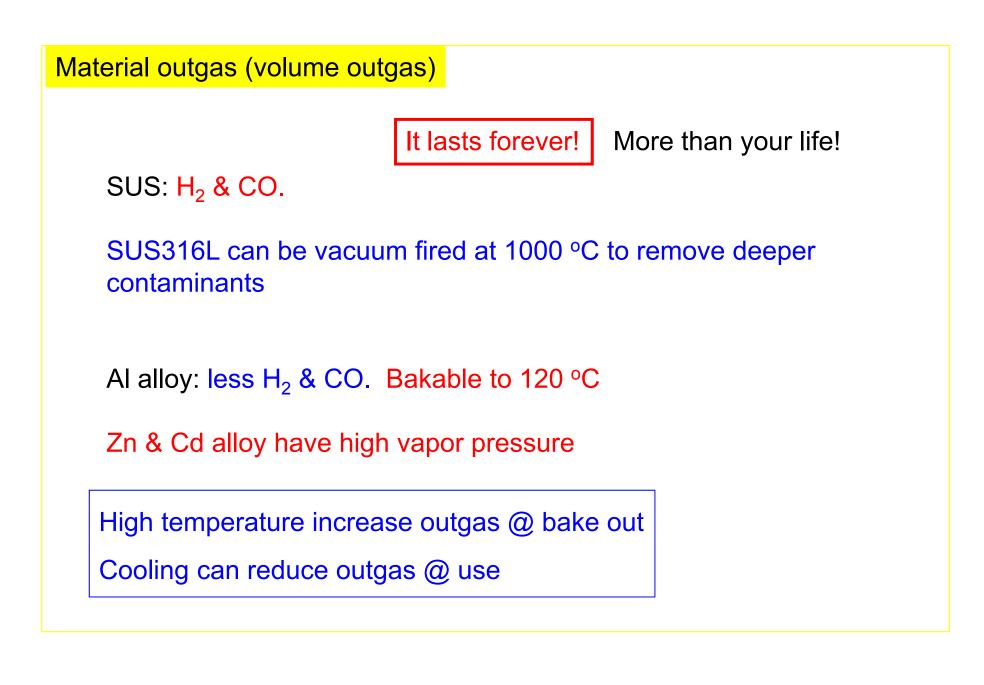
If the initial outgas pressure is 10^{-7} torr, $\tau = 5000$ hr

If the initial outgas pressure is 10^{-6} torr, $\tau = 500$ hr

more troublesome is that most plastics cannot be baked

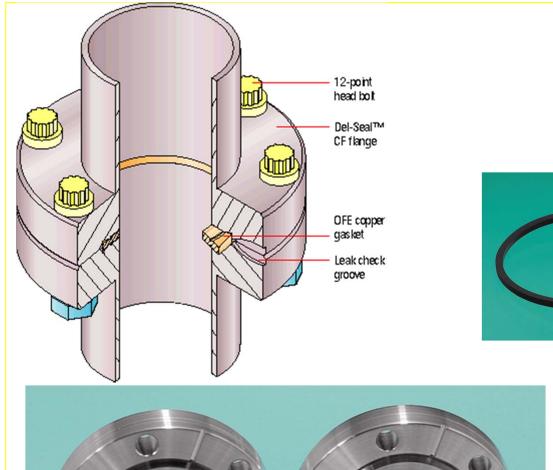
```
Use only:Less absorptionInert material: Teflon, PE, PP, Kel-F, Viton,<br/>Teflon insulated wire< \approx \frac{1}{10} normal plasticHigh temperature material : polyimide (Vespel, Kapton),<br/>Kalrez perfluoro elastomer (O-ring)Bakable to<br/>200°C
```

@1000 L/s



Sealing Concern:

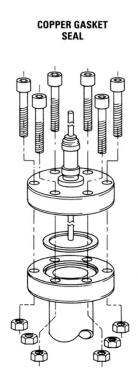
Metal seal: copper gasket & ConFlat flange are preferred 100% seal low outgas two surfaces may fuse together bakable use silver plated screws in SUS taps convenient O-ring seal: Viton O-ring bakable to 100 °C non-consuming 15 ~ 18 % compression to seal sealing surface polish is important volume compression is not allowed small leak is possible (*Hard* to find small leaks) Careful to use viton gasket on conflat flanges very easy to leak for size larger than 4.5" O.D. not cheap

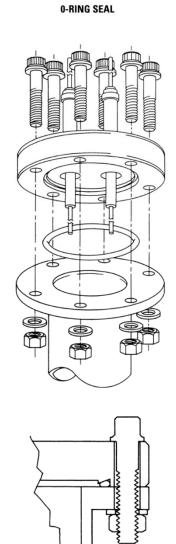






CONFLAT FLANGE



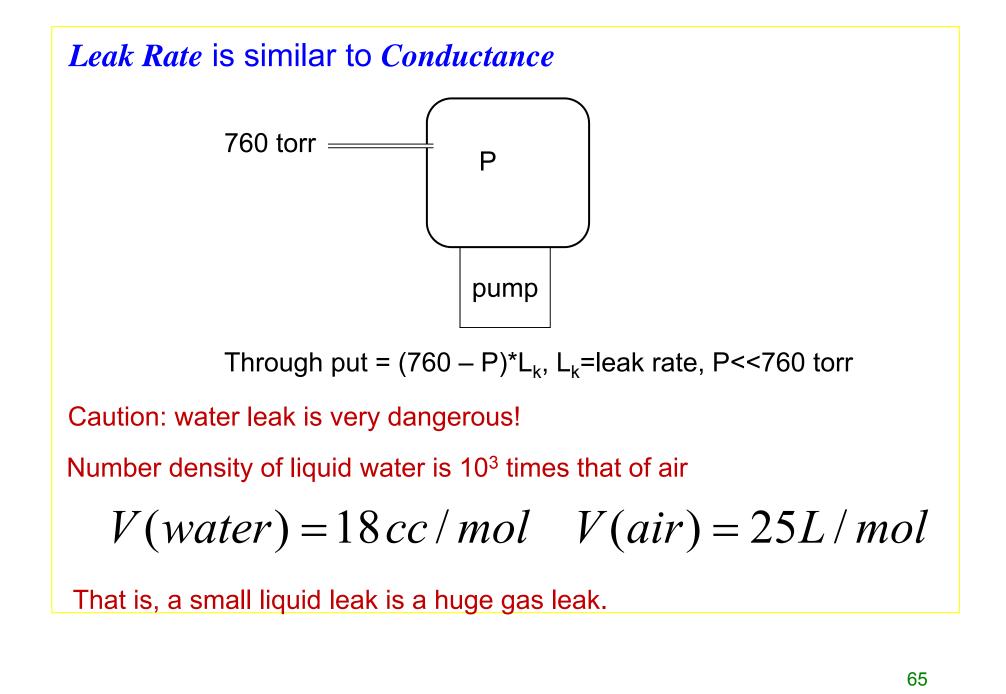


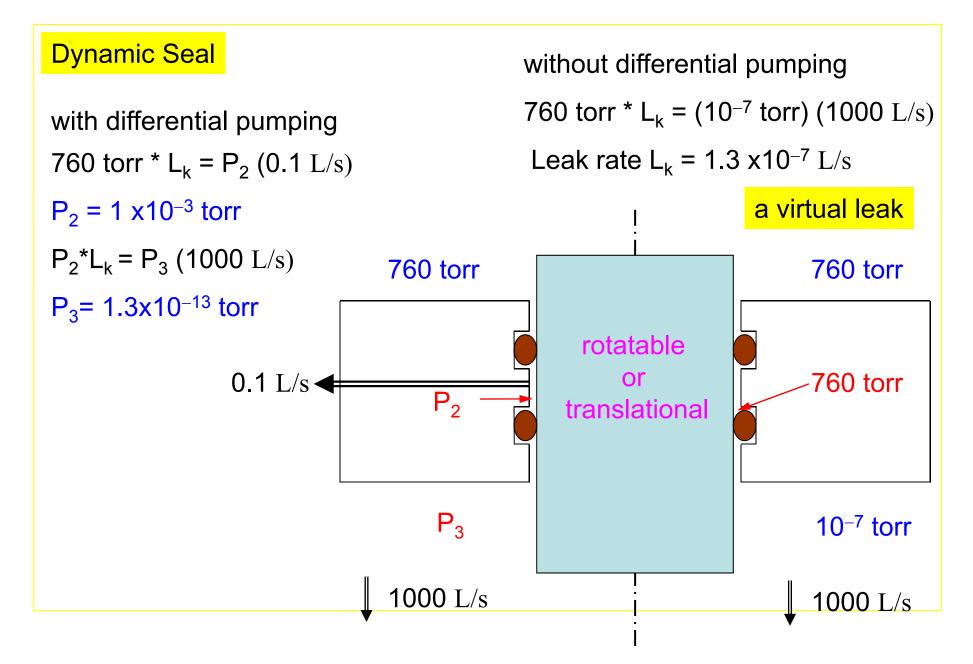
Flange to Flange			
Flange Dia.	O-Ring Cross Section	Parker O-Ring (美規 Cat. No.)
1.33	.103 Dia.	2-114	
2.75	.139 Dia.	2-223	
3.38	.139 Dia.	2-227	
4.50	.139 Dia.	2-234	
6.00	.139 Dia.	2-246	
8.00	.139 Dia.	2-260	
10.00	.139 Dia.	2-268	

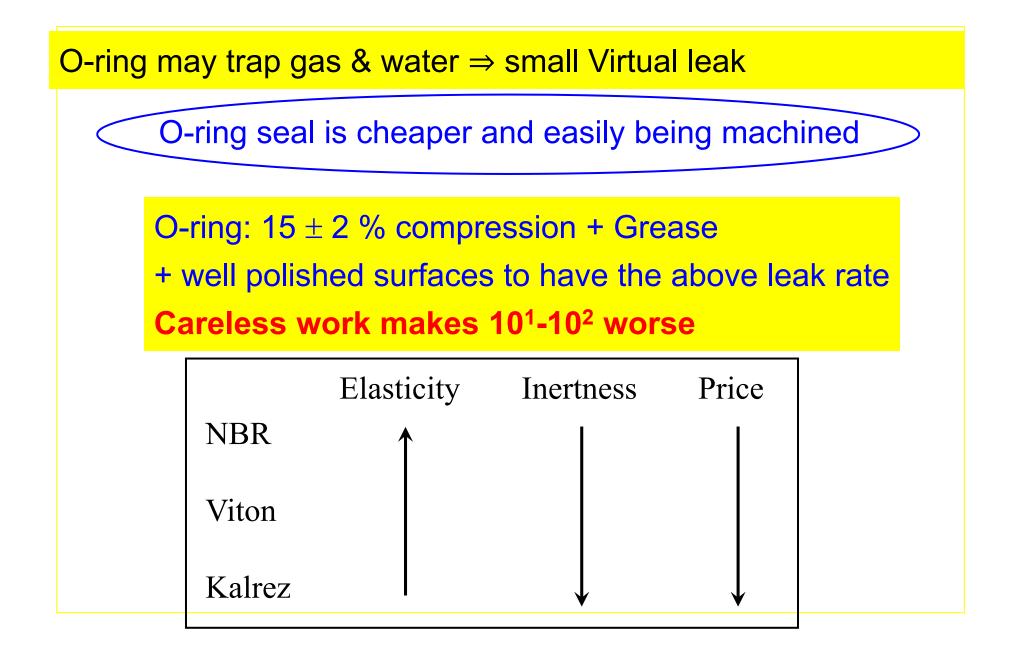
Viton O-Ring Seal

Viton O-Ring Seal **Flange to Flat Plate**

Flange Dia.	O-Ring Cross Section	Parker O-Ring Cat. No.
1.33	.070 Dia.	2-17
2.75	.103 Dia.	2-130
3.38	.103 Dia.	2-139
4.50	.103 Dia.	2-151
6.00	.103 Dia.	2-157
8.00	.103 Dia.	2-165
10.00	.103 Dia.	2-173







Conductance of an Aperture or short tube

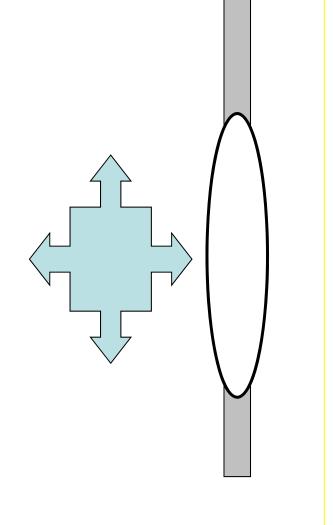
where A is the area in cm^2 . In the molecular-flow region, the conductance of an aperture for a gas of molecular weight M is:

$$C = 3.7 \left(\frac{T}{M}\right)^{1/2} A \ \text{L s}^{-1} \tag{3.25}$$

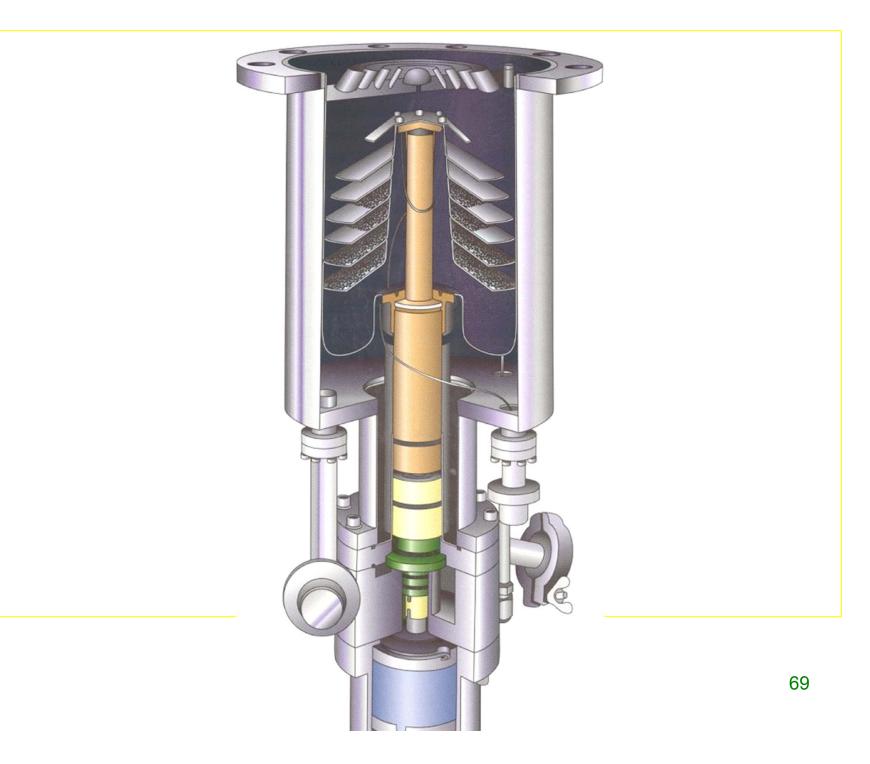
where A is the area in cm². These equations are useful for determining the rate of gas flow into a vacuum chamber through an aperture in a gas-filled collision chamber or ion source.

If there is a "black-hole" pump,

$$\frac{S_{\rm lim}}{L/s} = 3.7 \sqrt{\frac{T/K}{M/amu}} \frac{A}{cm^2}$$



For pumping port ϕ =20 cm, A=314 cm², S_{lim}(air)=3750 L/s



Specification		CP-8	S _{lim}
	Water	4000	4743
Gas Pumping speeds	Air	1500	3750
(liters/second)	Hydrogen	2200	14229
	Argon	1200	3182
Gas capacities	Hydrogen	12	
(standard liters @ 5x10 ⁻⁶ Torr)	Argon	1000	
Throughput	Argon	700 sccm	
Crossover	Torr-liters	150	
Cool down time	60 Hertz	90 min	
	Height	7.03"	
Cryopump measurements	Length	22.6"	
	Weight	42 lbs	
Inlet flange		ANSI/ISO/CF	

SlimS/Slim74384% ~ 175040% ~ 1/2422915% < 1/2</td>818238% ~ 1/2

Leak Check, not a trivial Job!

Spread CH_3OH or C_2H_5OH on a possible leak to see if pressure rises Acetone is OK for metal, bad for O-ring, bad for health Response rise time \sim few seconds, Don't move too fast. It takes very long to dry out the solvent. Very long fall time !! From lower spots to higher spots. few hours is possible 2.0 Average Chamber Pressure 1.5 Liquid 1.0 Gas 0.5 -

Time,

450

500

400

550

0.0

350

Helium leak check:

Spread He to see if P_{He} rises

MASS is required. RGA (Residue Gas Analyzer) or He leak detector

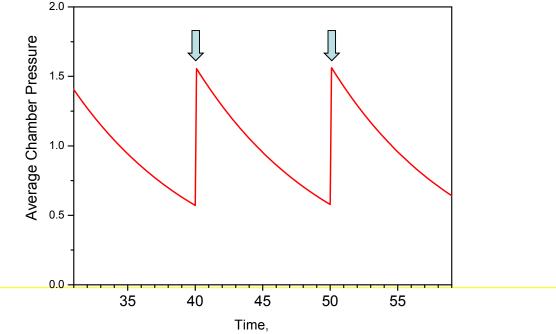
fast \lesssim 1 sec \therefore light mass \therefore fast speed

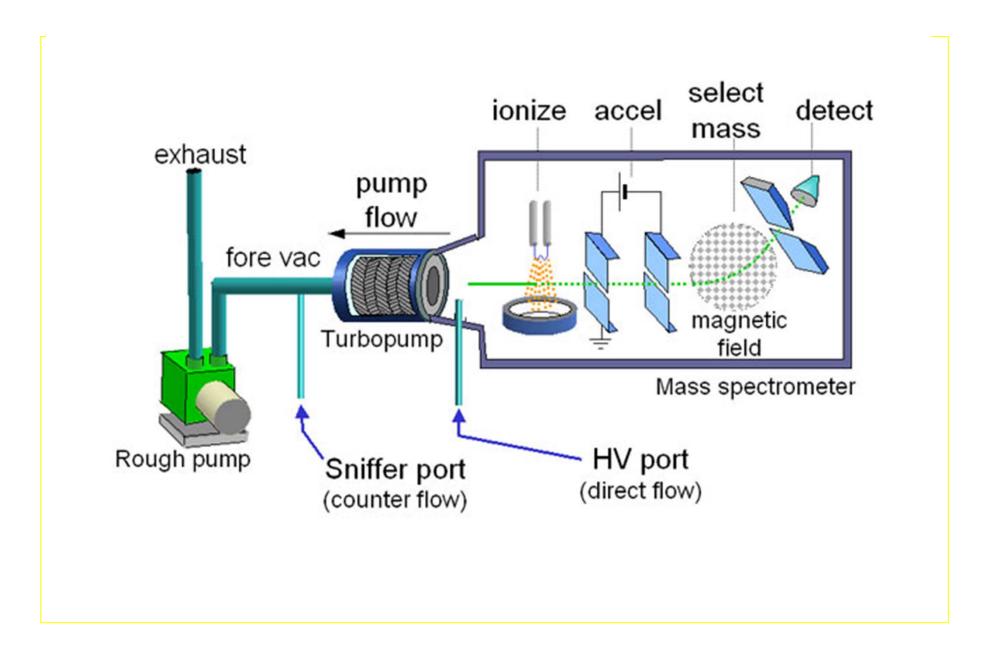
He is fast to escape, fast to pump down

low background, inert

from higher spots to lower spots

He is easy to reach a nearby (even 1 m away) spot. \Rightarrow isolation





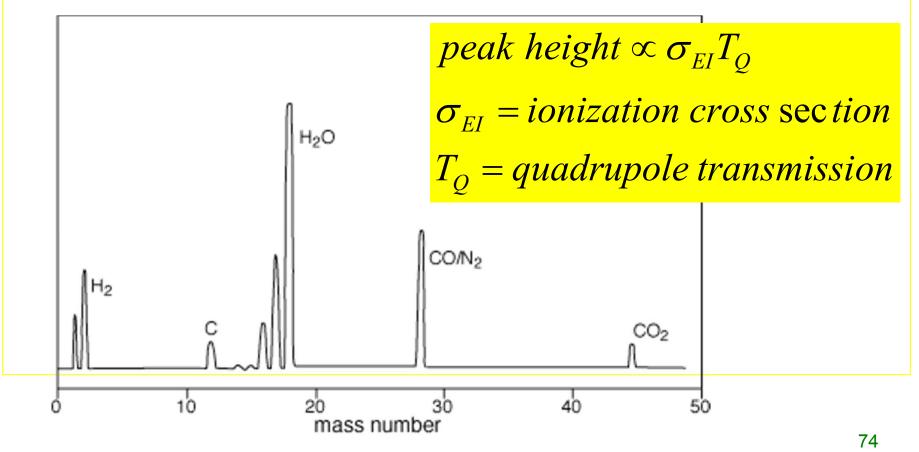
Residue Gas Analyzer (RGA) provide very important information

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high m28/m32 (4:1) indicate air leak
```

daughter ion is useful. CO+ / C+ , O+ Vs. N_2^+ / N+

H₂O is very common

Don't make vacuum chamber wet, especially at a rainy day



Good vacuum practices

No leak

Clean: traps for oil pumps: molecular sieve, LN2

Metal & non-porous ceramic is excellent

Plastic and grease: as less as possible

Confident sealing. Finding a leak is very labor-consuming.

Bakable for 10⁻¹⁰ torr or better

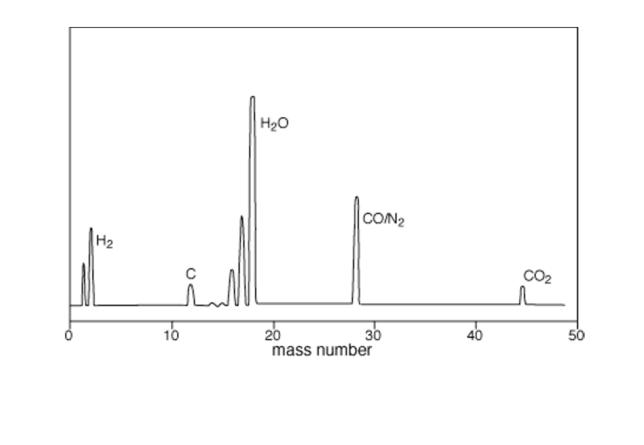
Good Local Conductance for pumping speed concern.

Gas composition (partial pressure) is often more important than the total pressure, as most vacuum parameters are species dependent. e.g. surface laser burn, background masses

RGA is very nice to have (it is cheap now)

Take home message:

Partial Pressures is more important than the total pressure.

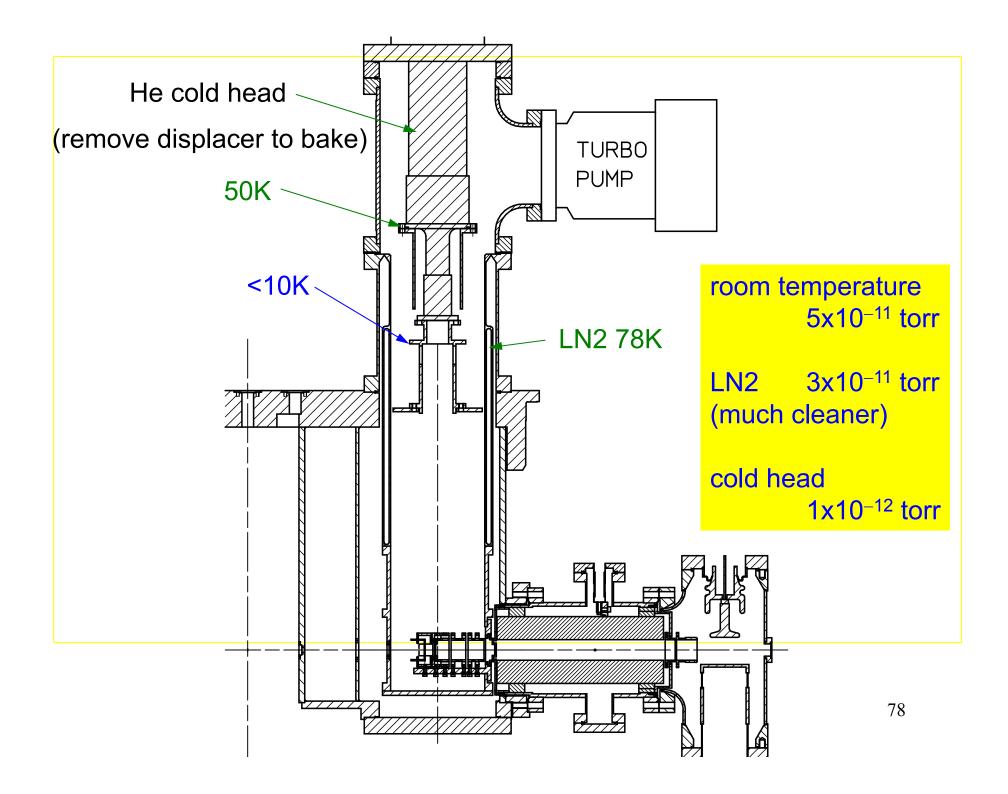


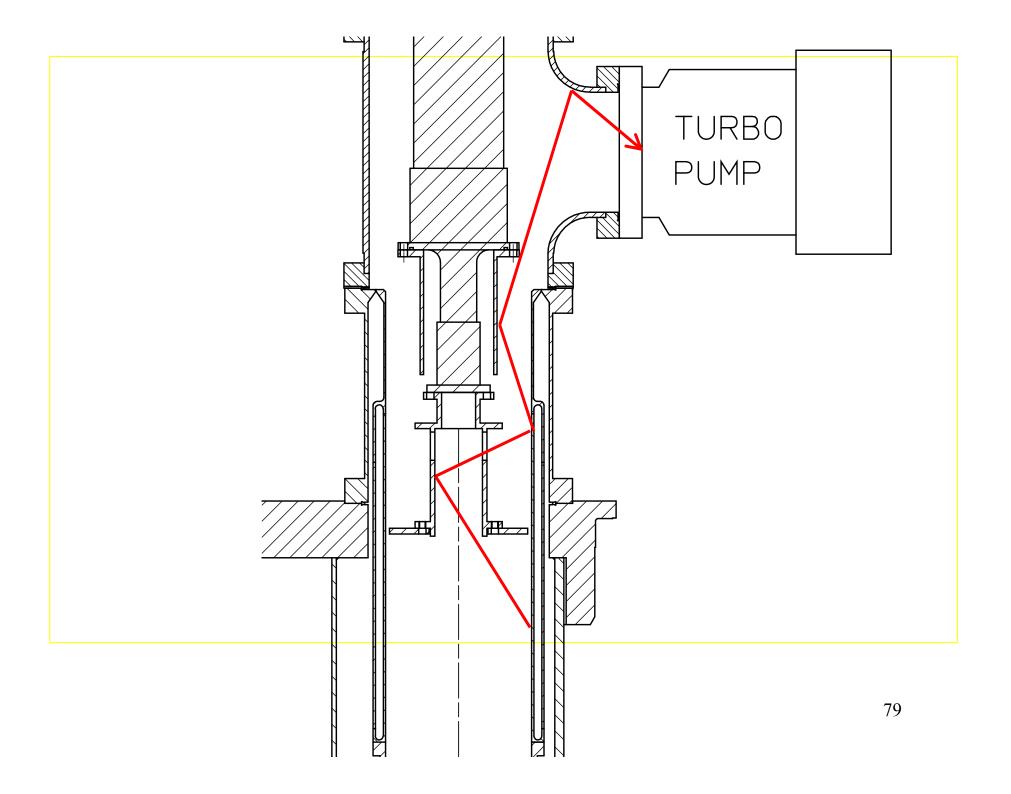
Practically

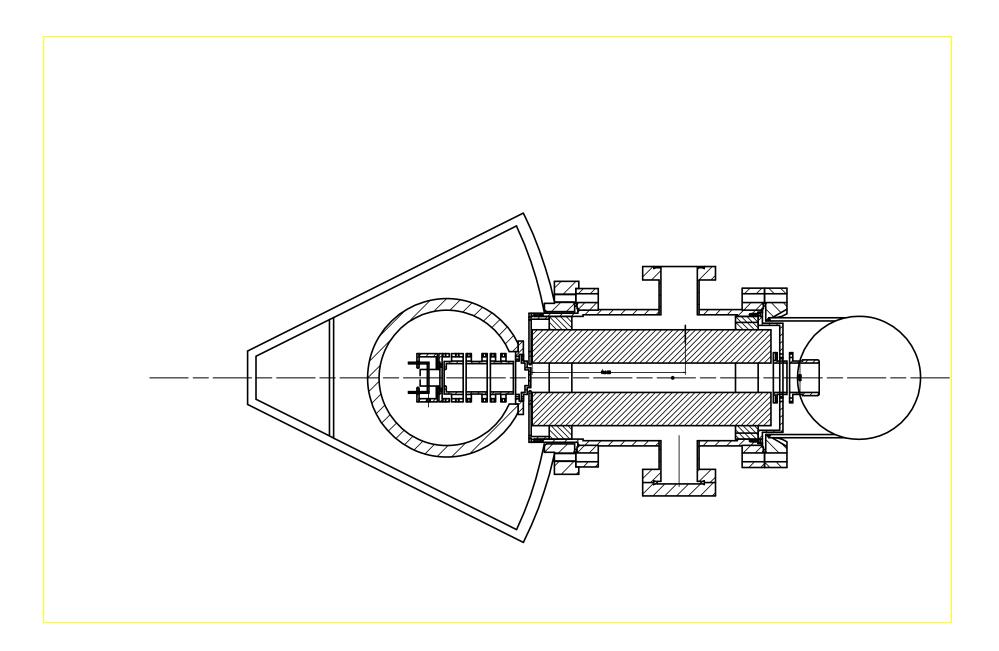
clean chamber, turbo pump, not baked, 10^{-9} torr clean chamber, 2 serial turbo pumps, baked, 10^{-11} torr (compression ratio for H₂)

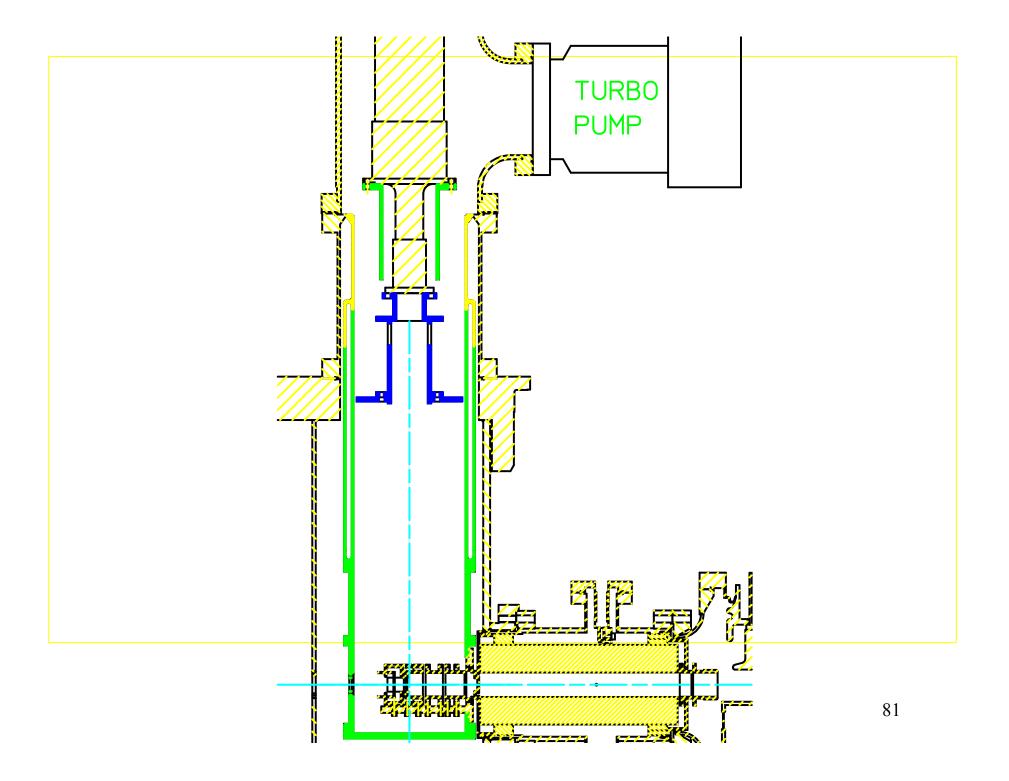
Untra High Vacuum < $1x10^{-10}$ torr

Example of 1 x 10⁻¹² torr









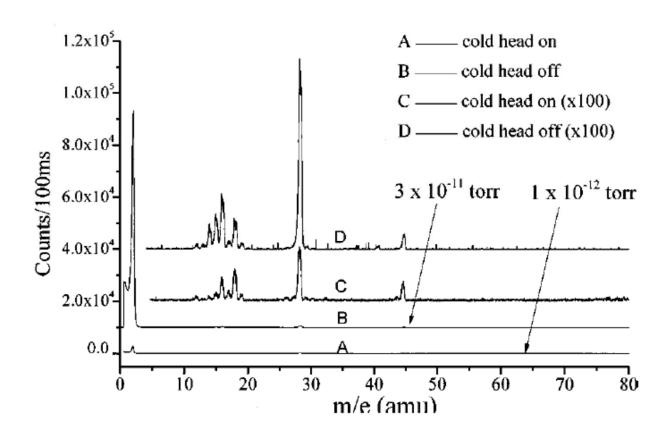
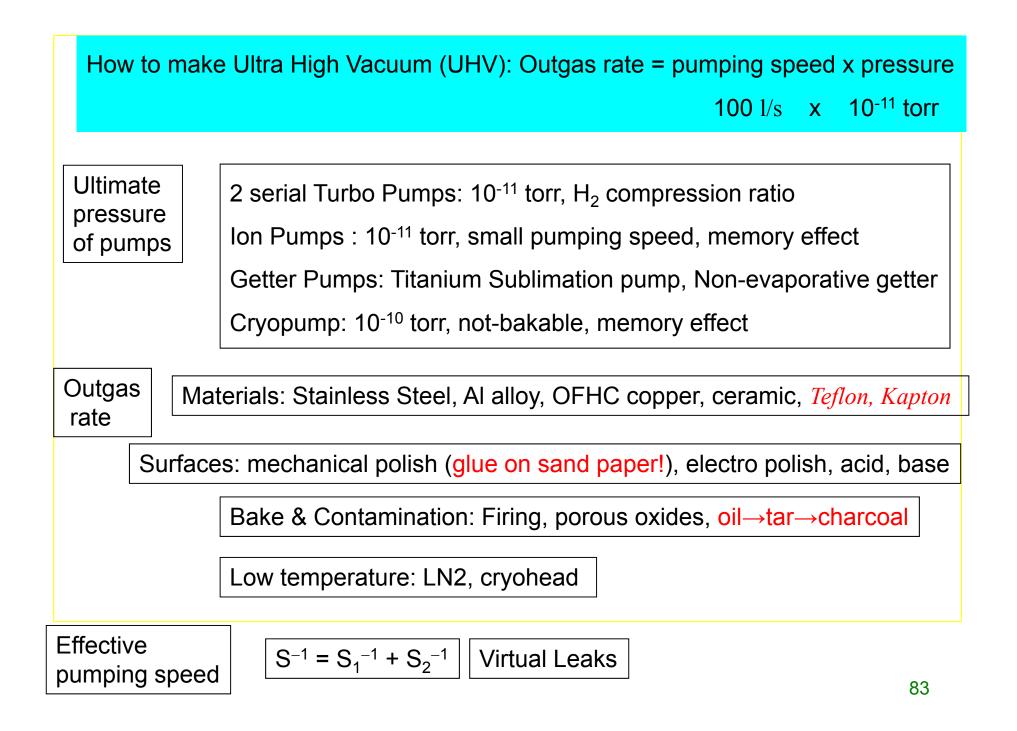


FIG. 3. Detector residual gas mass spectra before and after the cryogenic He cold head cryopump starts to pump.



```
Turbo pumps: 400 L/s, 600 L/s
compression ratio: 10<sup>2</sup>-10<sup>4</sup> for H<sub>2</sub>, 10<sup>6</sup> for He,10<sup>9</sup> for N<sub>2</sub>
<125°C
Oil, grease, or magnetic bearing and insulated wires
Fore line back stream when Electric shutdown
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Getter pumps: Ion, Ti, NEG No foreline needed, no continuous electricity needed 250°C, 400°C Not for every gas, memory effect Low maintenances for low load systems Cryopumps: >1500 ℓ/s,

bake to 70°C only,

Not for every gas, memory effect

Outgas due to activated carbon absorber

Electric shutdown

Cryopump + turbo pump: Very high pumping speed even for H_2 at 10⁻⁹ torr

Bakable Cryohead without absorber: high pumping speed for H_2 at <10⁻¹¹ torr Low outgas \Rightarrow 10⁻¹² torr SUS304, SUS304L: Cr 18%, Ni 10%, Fe, C<0.2 % or Low carbon < 0.08% SUS316, SUS316L: Cr 18%, Ni 10%, Mo 2-3%, std and Low carbon

Sand ballasting, basic detergent, Acid dip, Electro polish, DI water Easy to be welded,

Bake to 250°C, SUS316L: Fire at 1000°C at 10⁻⁸ torr

Major outgas: H₂, CO

Al alloy 6061-T6, and others

Low H₂, CO outgas

Welding at outside, must clean before welding

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NaOH(aq), HNO<sub>3</sub>(aq),
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 Al_2O_3 is porous. Mirror finish parts is available (Japan)

120°C, high temperature will change tempering condition

Oxygen Free High Conductance copper, Beryllium copper

Brass and bronze could be dirty (zinc, phosphor)

Special (strong) acid brightening

Plastic: gas/water permittivity is high

Teflon absorb water 10 times less than usual plastics, but still too much for UHV

Teflon, PE, PP, might be OK for 10⁻⁸ torr, others are only good to 10⁻⁶ torr. No PVC

polyimide (Kapton) is bakable, 10⁻¹⁰ torr

Ceramic could be porous

Vacuum firing

Al₂O₃ (alumina) (thermal conductance better than SUS)

