Ultrahigh vacuum is an ion's best friend

Tips and tricks for the experimenter

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Physics with Trapped Charged Particles

Very few and minor modifications

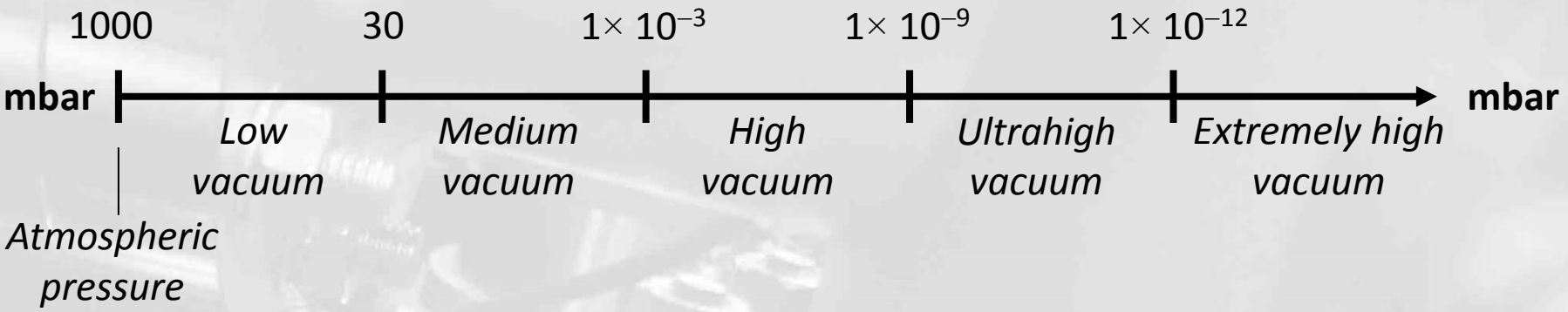
by Martina Knoop

Outline

- Ultrahigh vacuum: what and why
  Cryogenic UHV→ See talk by J. Crespo
- Producing UHV
  - Pumps
  - Outgassing, leaks and backflow
  - Bakeout
- Building UHV apparatus
  - Design rules
  - Leak detection
  - UHV compatible materials
- Keeping UHV
  - Pressure monitoring
  - Venting UHV without spoiling

UHV: What and Why

Vacuum pressure ranges



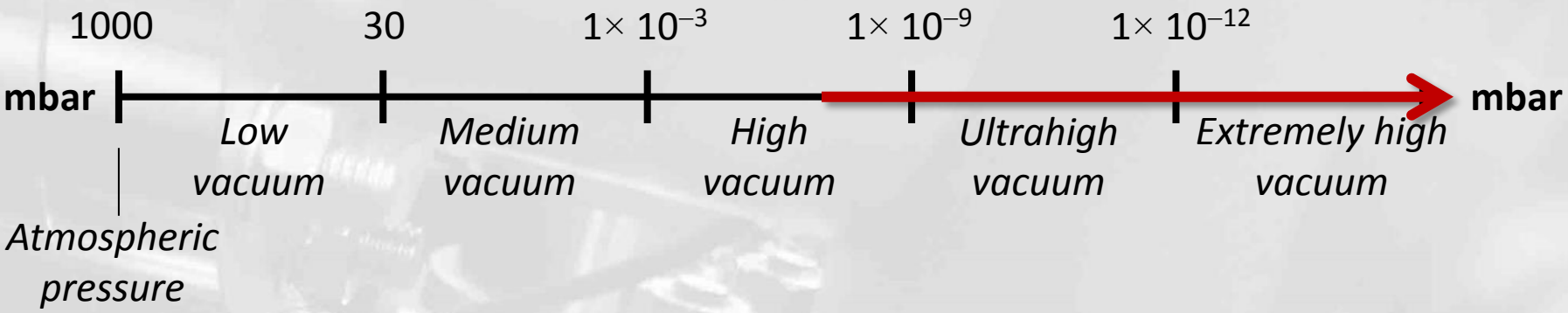
Commonly used pressure units:

SI unit: 1 N/m² ≡ 1 Pa (use this for ideal gas law, $p = n\,k_B T$)

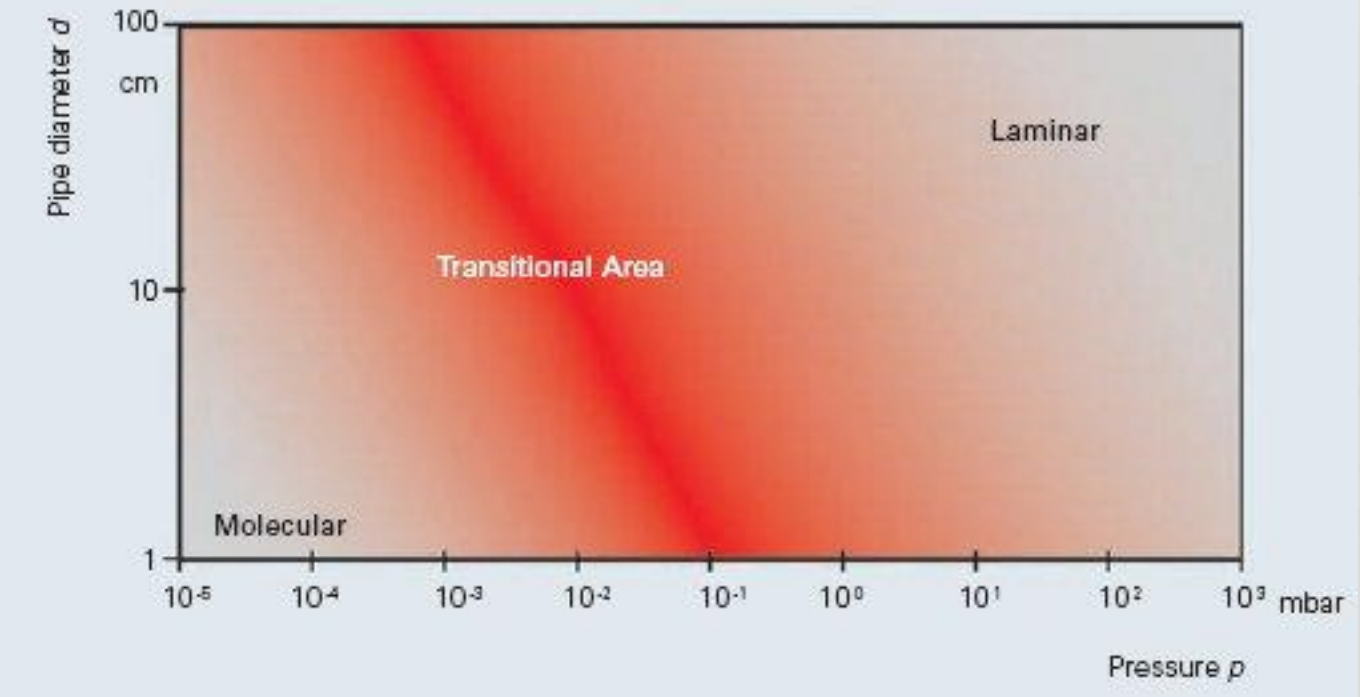
1 mbar = 100 Pa (1 bar ≈ atmospheric pressure at sea level)

1 Torr = 133.3224 Pa (mm_{Hg})

Vacuum pressure ranges



Molecular flow regime: mean free path > apparatus dimensions



Collisions: the good, the bad and the ugly

- **Collisions: trapped ion + neutral atom/molecule**

- Good collisions: when buffer-gas cooling, collisions with cold He (few Kelvin)

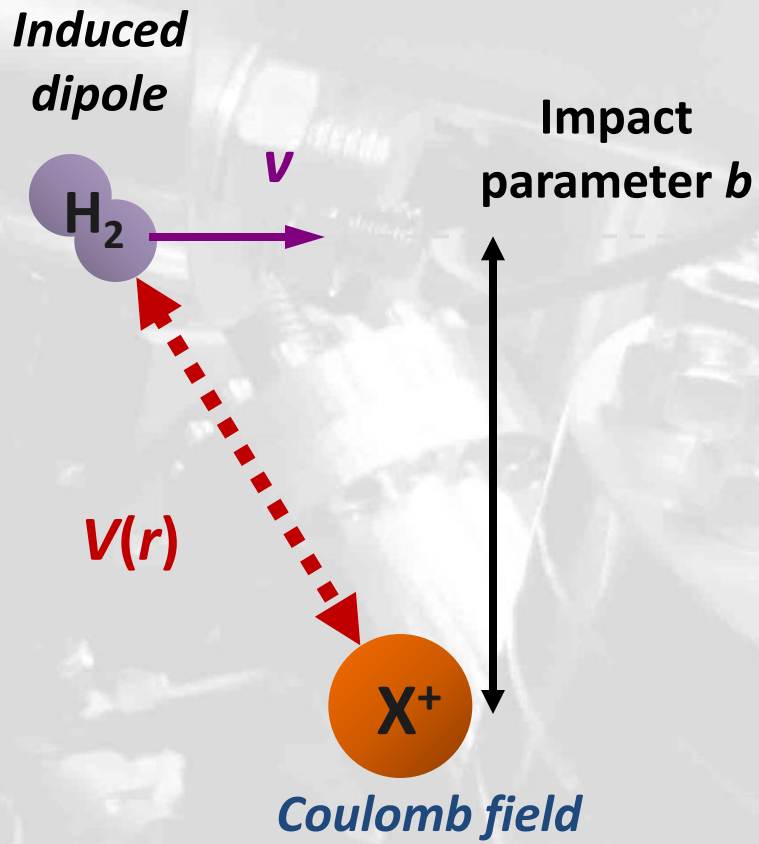
- Bad collisions: elastic collisions with room-T gas

  e.g. Be^+ ion (1 mK) + H_2 (300 K) $\rightarrow$ Be^+ (121 K) + H_2 (179 K)

  - Can lead to decoherence (QIP)
  - Can lead to frequency shifts (ion optical clocks)

- Ugly collisions: (unwanted) chemical reactions

Example: Langevin collisions



Long range: $V(r) \sim -1/r^4$

***b* ⇔ centrifugal barrier**

Critical b_c such that:

- $b > b_c$ glancing collision
- $b < b_c$ spiralling of neutral towards ion ("Langevin collision")
- At short range, energy transfer or chemical reaction may occur

Collision rate & outcome

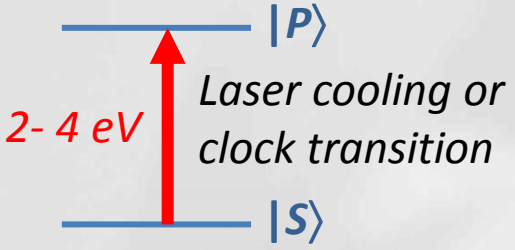
- Elastic collisions: trap heating
- Inelastic collisions: $X^+ + H_2 \rightarrow XH^+ + H$

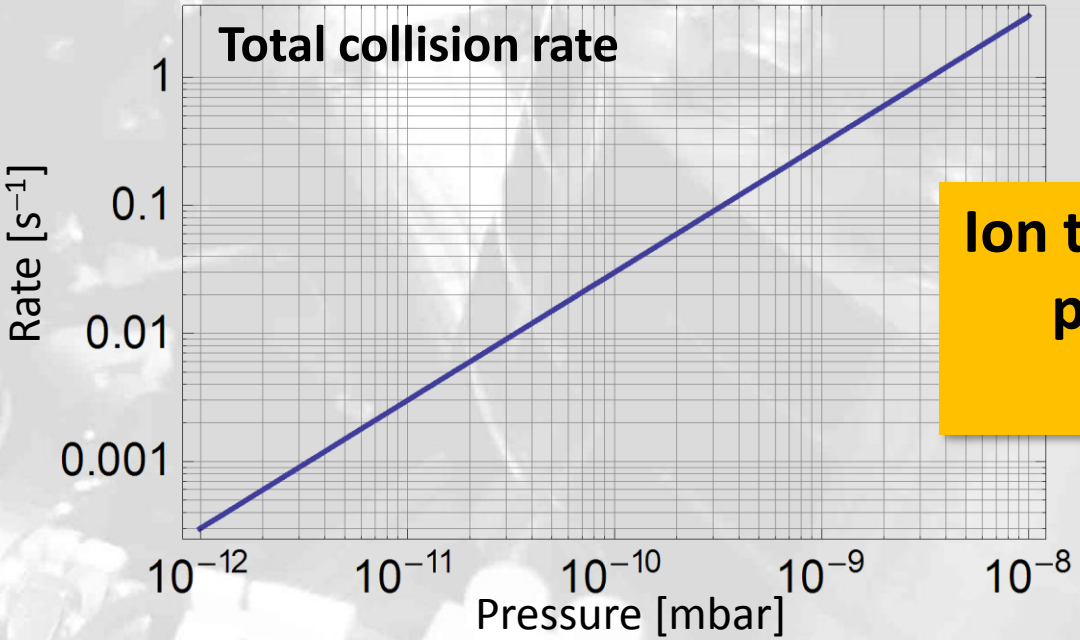
  Requires 4.5 eV to break H_2 bond

  Releases ~2 eV when forming XH^+

  $\Rightarrow$ Reaction endothermic by ~ 2 eV

  $\Rightarrow$ Ion in excited *P* state can provide energy to form XH^+ …

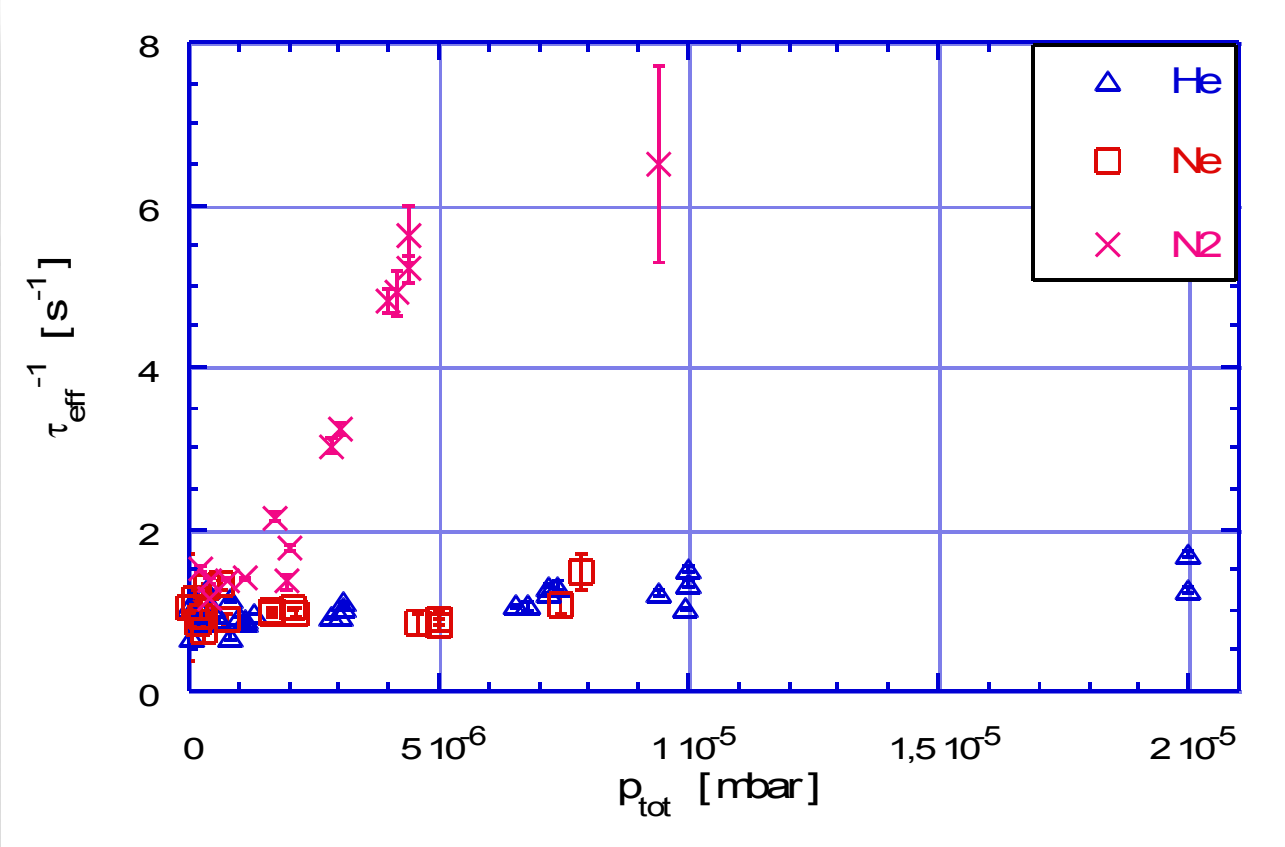




Ion trap experiments require pressure << 10^{-9} mbar (UHV)

*See *e.g.* thesis B.E. King (NIST/CU, 1999)

Quenching collisions



Stern-Vollmer-plot

for Ca+, $3D_{3/2}$

$$\frac{1}{\tau_Q} = \frac{1}{\tau_{nat}} + \sum_i n_B^i \Gamma_Q^i$$

with *i* : H_2, He, CH_4 , Ne, N_2, O_2, Ar, and CO_2

Collisional quenching and j-mixing rate constants for the 3D levels of Ca+, M. Knoop, M. Vedel, F. Vedel, Phys. Rev. A **58,** 264 (1998)

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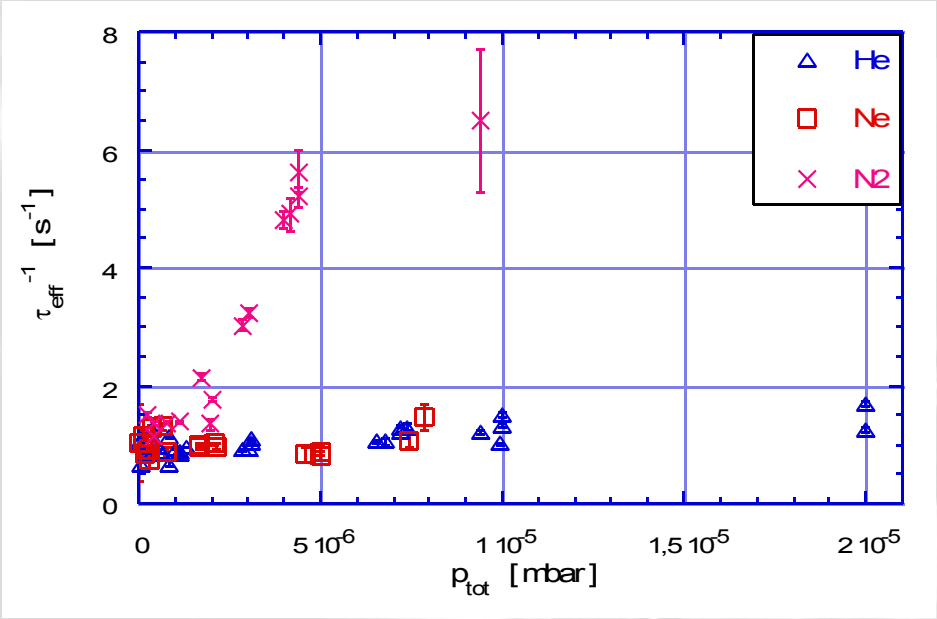

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TABLE I. Quenching rate constants for the $3D$ levels of Ca^+.

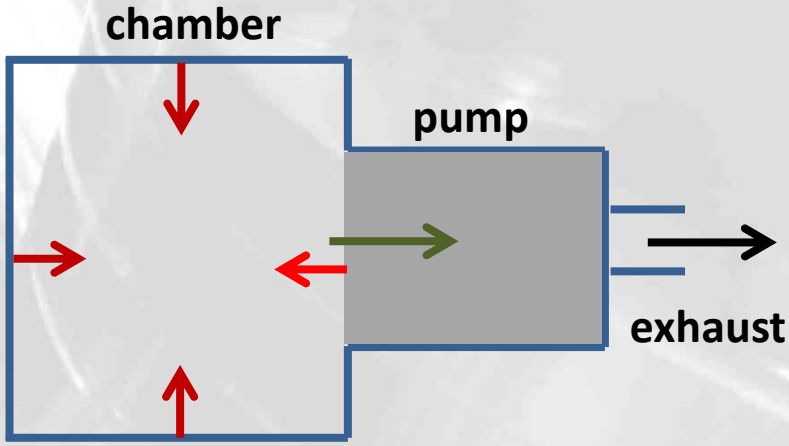
Gas	m (amu)	α (10^{-24} cm^3)	Γ_Q (10^{-12} cm^3 s^{-1})	k_L (10^{-10} cm^3 s^{-1})	k_L/Γ_Q
H_2	2	0.804	37 ± 14	15.2	41 ± 15.5
He	4	0.205	1.05 ± 0.40	5.56	529 ± 201
CH_4	16	2.593	54^{+91}_{-17}	11.15	$21^{+35}_{-6.5}$
Ne	20	0.396	0.9 ± 0.7	4.03	448 ± 348
N_2	28	1.74	170 ± 20	7.61	4.5 ± 0.5
Ar	40	1.64	29.5 ± 17.0	6.70	23 ± 13

Producing UHV

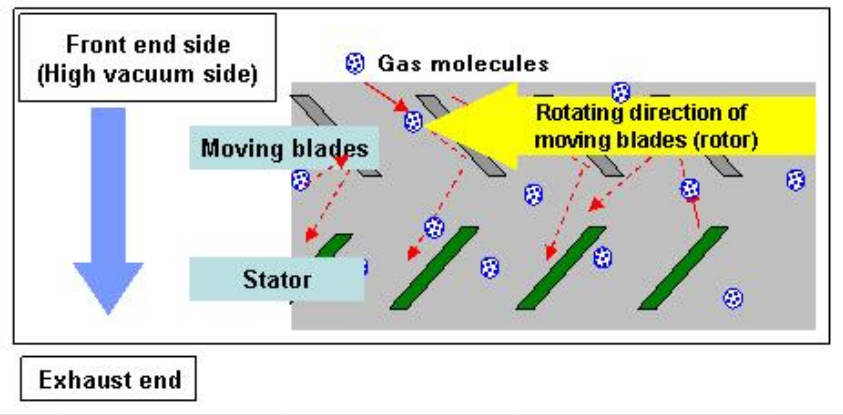
Pressure and gas flows

- Vacuum chamber pressure is determined by equilibrium between:
  - **Pump speed/throughput**
  - **Backflow (through pump and leaks)**
  - **Outgassing from walls and *in vacuo* parts**

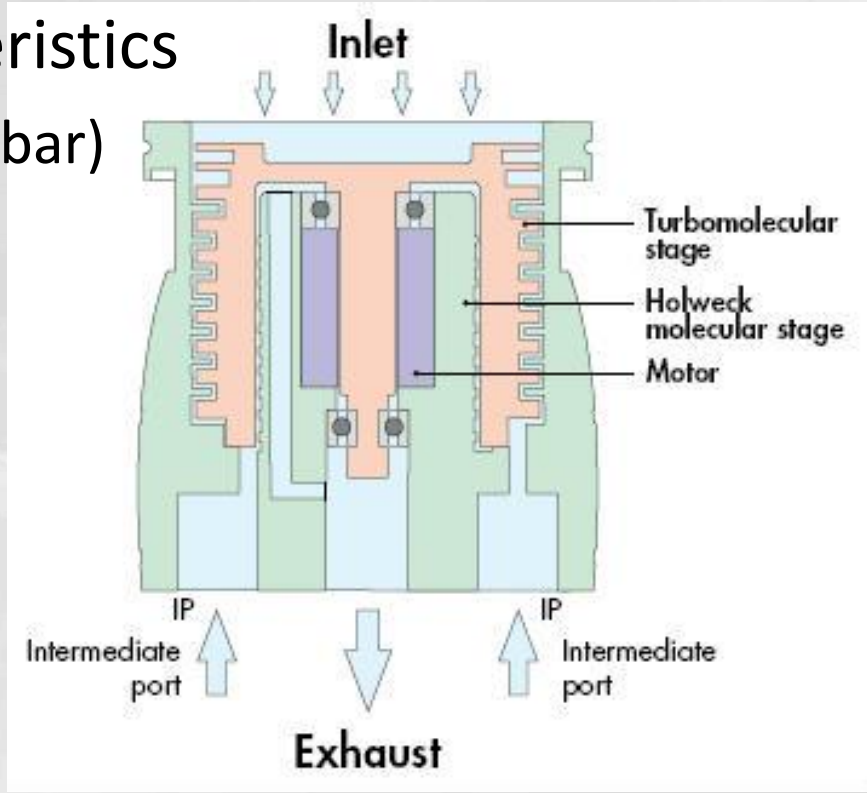
Note that outgassing can occur from inside the walls (the 'bulk')



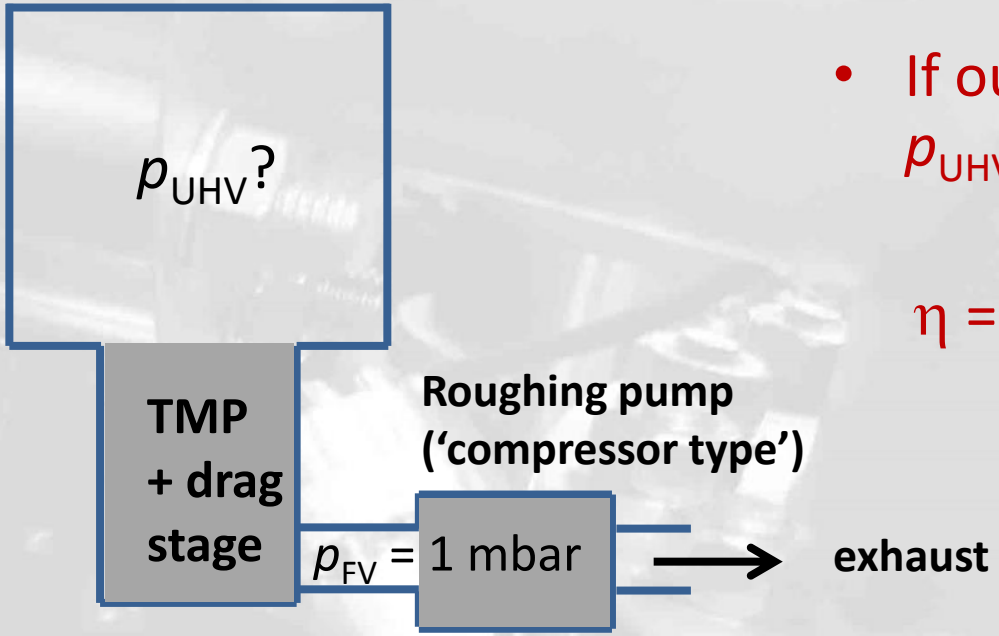
UHV pump types



- Turbomolecular pump
  - Fast-spinning blades ‘knock’ incoming molecules out of main chamber
- Important features/characteristics
  - Requires roughing pump (1 mbar)
  - Pumping speed
  - Mesh to protect blades?
  - Compression ratio
  - Use turbo with Holweck (‘drag’) section!



Example: TMP UHV chamber

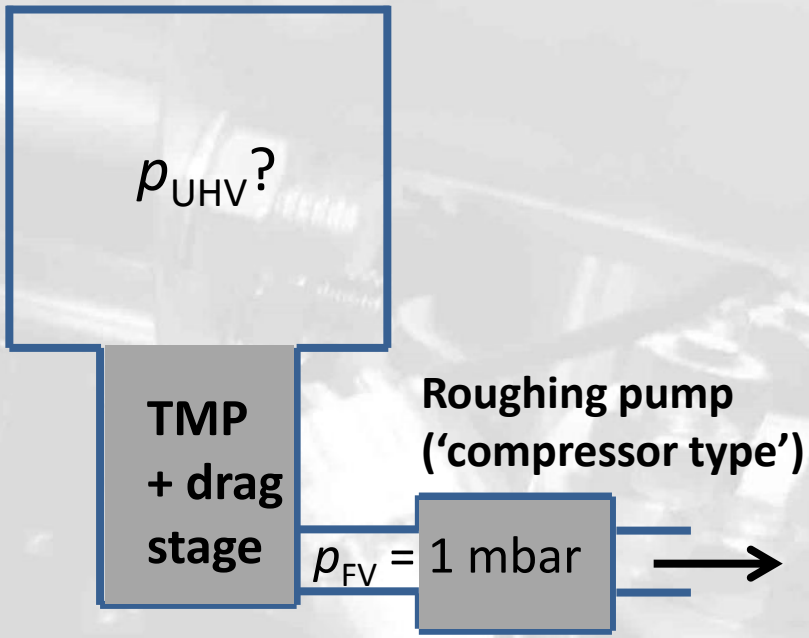


- If outgassing negligible:
  $p_{UHV} = p_{FV} / \eta$

η = **TMP compression ratio**

Gas species	η	Partial p_{UHV} (p_{FV} = 1 mbar, atmospheric composition)	
N_2	$> 10^{11}$	$< 8\times10^{-12}$ mbar	78 %
Ar	$> 10^{11}$	$< 10^{-13}$ mbar	0.93 %
He	3×10^{7}	2×10^{-13} mbar	0.00052 %
H_2	4×10^{5}	1.4×10^{-12} mbar	0.000055%

Example: TMP UHV chamber



- ~~If outgassing negligible:~~
  ~~$p_{UHV} = p_{FV} / \eta$~~

- **Typically, H_2 outgassing dominates UHV**
- **H_2 accumulates in 1 mbar forevacuum, backflow to UHV at sub-10^{-10} mbar level**

Solution: use better roughing pump:
***e.g.* scroll pump, or small intermediate TMP**
($p_{FV} < 10^{-2}$ mbar should do)

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When to use a TMP?

- High gas loads (*e.g.* He buffer gas cooling)
- High noble gas loads

When not to use a TMP?

- If vibrations cannot be tolerated
- If setup needs be transportable under UHV without current supply

Titanium sublimation pump (TSP)

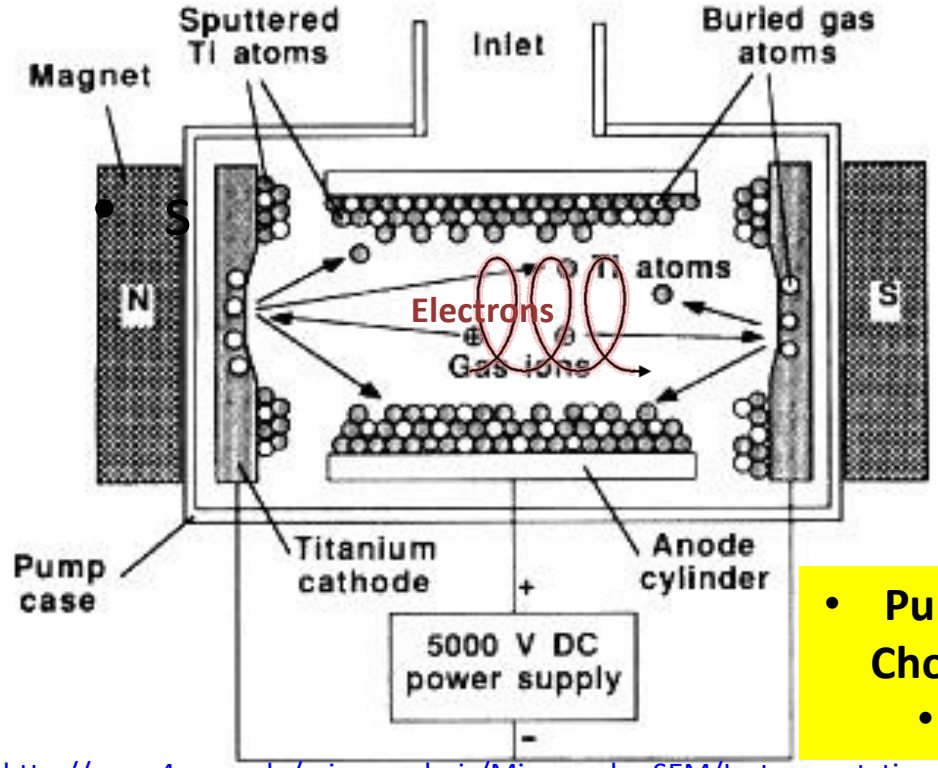
- Filaments plated with titanium
- In vacuum, fire filaments (ohmic heating)
- Ti sublimates and covers walls
- Ti layer acts as getter material

- **Improve pump speed by cooling**

  (use cryoshield)

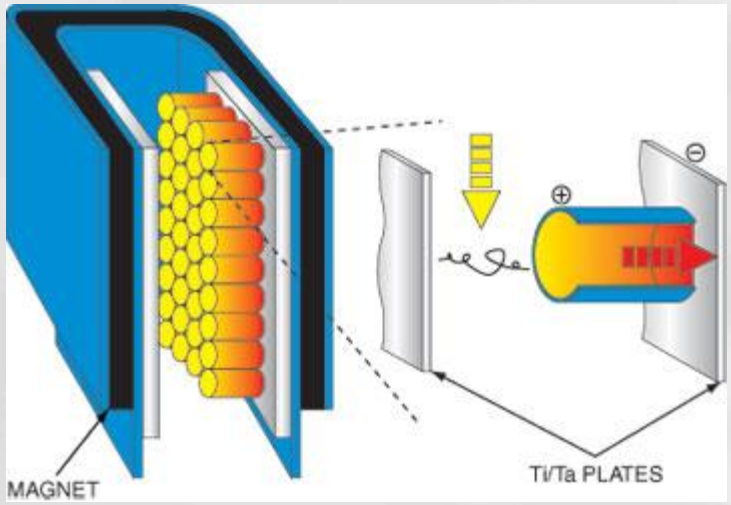
Pumping Speed in $ls^{-1}cm^{-2}$							
Gas Species	H_2	N_2	O_2	CO	CO_2	H_20	CH_2
+20°C	3	4	9	9	8	3	0
-196°C	10	10	11	11	9	14	0

Ion getter pump



http://www4.nau.edu/microanalysis/Microprobe-SEM/Instrumentation.html

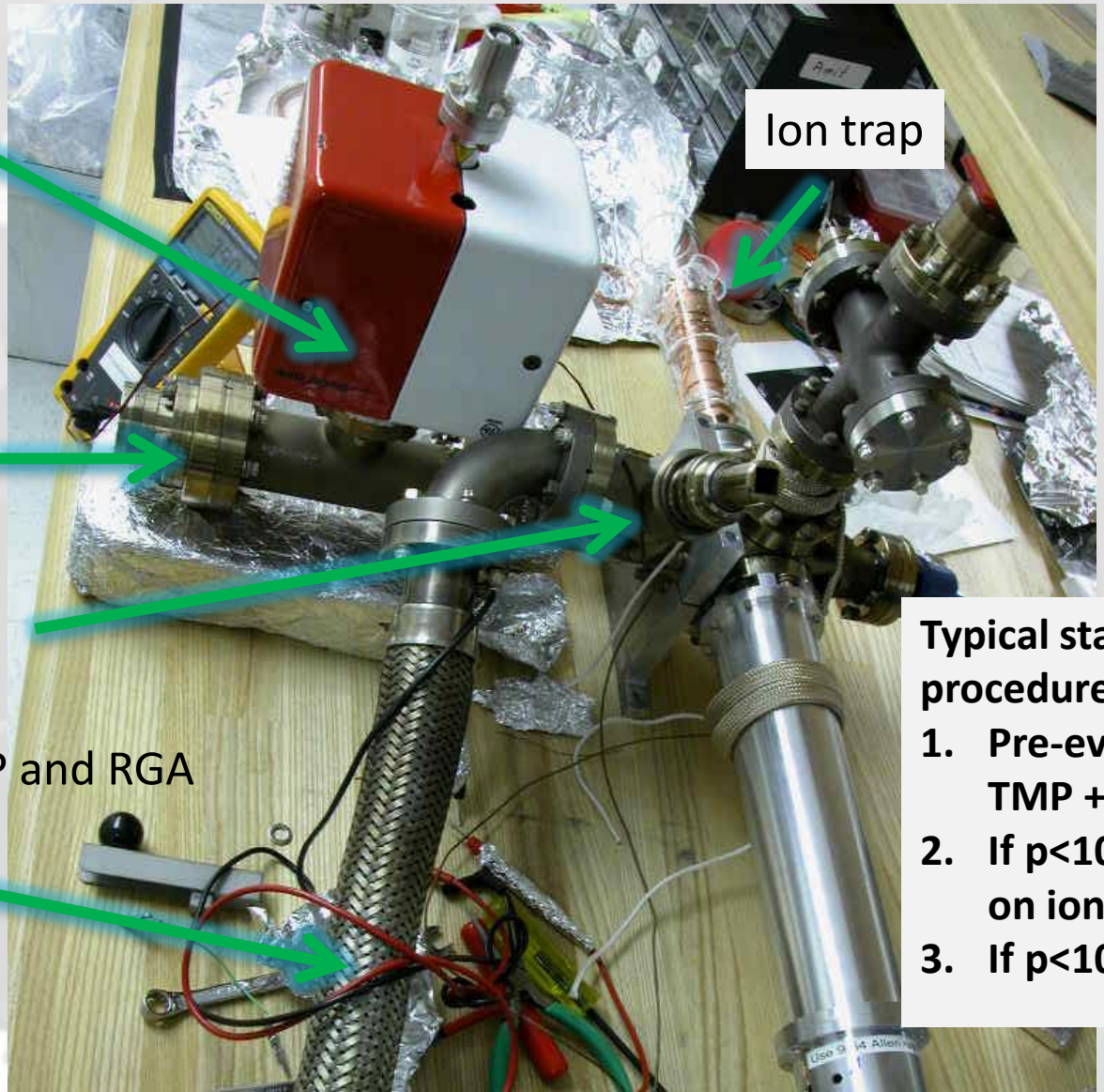
Use multiple 'cells' to increase area



- **Pumps with different getter material (Ti, Ta) exist, Choice depends on background gas composition**
  - **'All-round' pump: triode (StarCell) pump**

- Electrons in a Penning trap, fed by HV discharge
- Electrons ionize background gas; fragments are attracted to and strike cathode to:
  - Get stuck forever
  - Sputter cathode 'getter' material (often Ti) on walls of pump, which adsorbs neutral gas constituents

Typical pump arrangement (2nd Al^{+} trap NIST 2005)



Typical start-up procedure:

1. **Pre-evacuate using TMP + roughing pump**
2. **If $p<10^{-6}$ mbar, switch on ion pump**
3. **If $p<10^{-8}$ mbar, fire TSP**

Outgassing and bakeout

- You close and evacuate the vacuum chamber for the first time, and the pressure doesn't drop below …
  … **10^{-5} mbar** (*did you clean any of those parts at all??*)

Outgassing and bakeout

- You close and evacuate the vacuum chamber for the first time, and the pressure doesn't drop below …
  - … **10^{-5} mbar** (*did you clean any of those parts at all??*)
  - … **10^{-7} mbar** (*don't worry, this is normal – it's just water, it sticks to the walls because it is polar*)

- Desorption rate of water adsorbed to the walls*:

$$\Gamma \propto A\, \nu \exp(-E_a/k_B\, T)$$

where A = wall surface area

ν = vibrational frequency of 'bound' H_2O molecule

E_a = activation energy needed for desorption

At 300 K/10^{-7} mbar, virtually no change in abundance of H_2O

$\Rightarrow$ *no change in pressure…*

*Somorjai, Gabor A.; Li, Yimin (2010).
Introduction to Surface Chemistry and Catalysis. John Wiley and Sons.

Bakeout

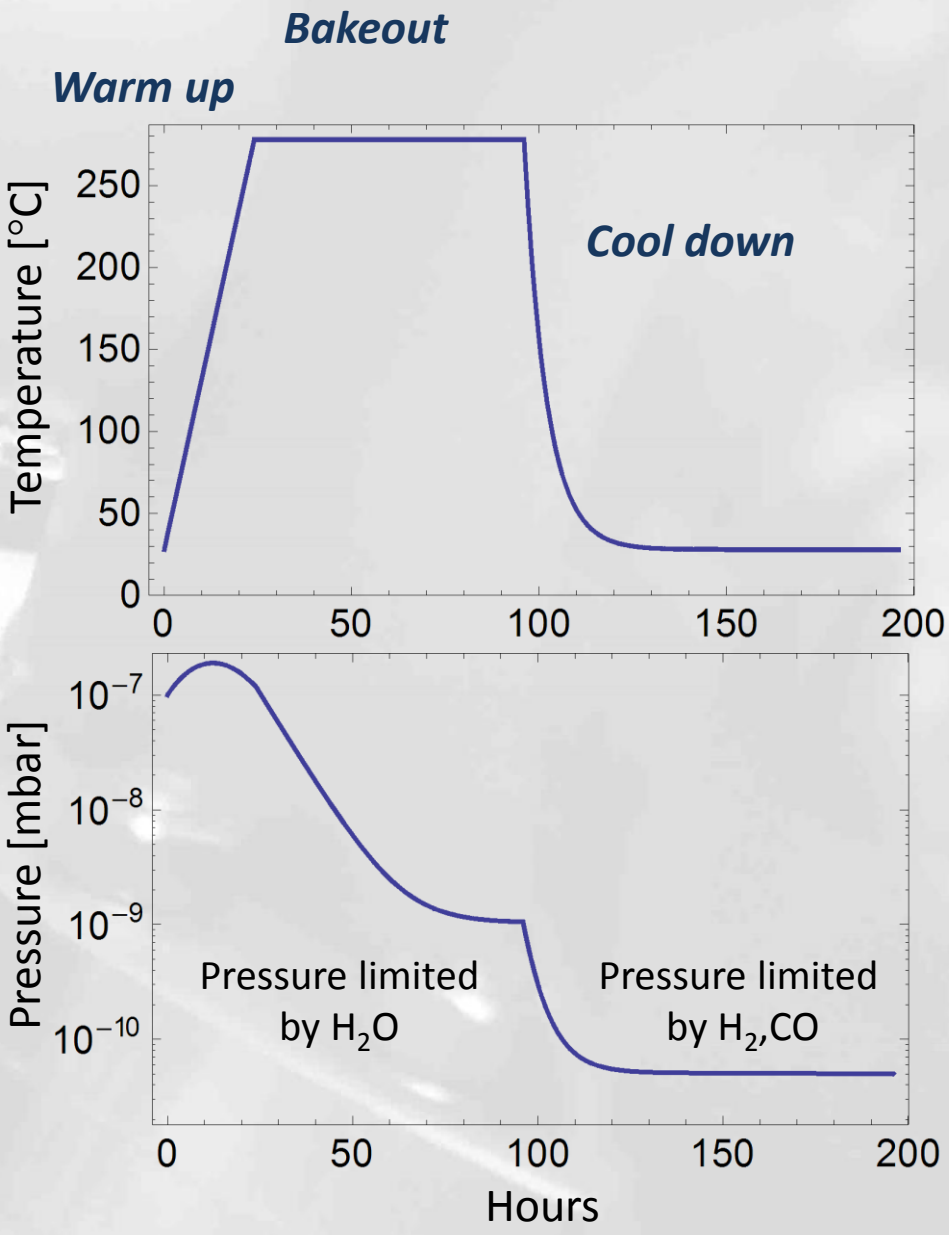
… but at elevated temperatures, desorption rate increase exponentially!

Bakeout: increase *T* to 200 – 400 °C, remove H_2O

After cooling down:

H_2 , CO, … remain (larger E_a)

Note: above 400 °C, H_2 diffuses from atmosphere through steel walls faster than you can desorb



Additonal strategies

- Air bake chamber at T = 200 - 400°C (steel will oxidize, forming an additional barrier for H_2 in bulk), followed by vacuum bakeout

  $\Rightarrow$*Reduce p from 10^{-11} to <10^{-12} mbar*

- Use in-vacuum IR heaters, or UV lamps (photodesorption)

Table I. N_2-equivalent outgassing rates of stainless steel and aluminium obtained after a vacuum bakeout at 100 and 400 °C, and after a 2 h bakeout in air at atmospheric pressure followed by a 18 h vacuum bakeout at the same temperature (bakeout time = 20 h).

	Outgassing rates in units of 10^{-13} Torr liters/sec cm² (N_2-equiv.)			
	Vacuum bakeout		Air bakeout	
	100 °C	400 °C	100 °C	400 °C
Stainless steel	10	6	1	0.4
Aluminium	0.4	—	0.28	—

G. Moraw & R. Dobrozemsky, Proc. 6th Intl Vac. Congr. (1974)

- Allegedly the following should work as well (no experience myself):

  With chamber at T ≈ 100°C, fill with oxygen

  $\Rightarrow$ $O_2 + H_2$ (bulk) $\rightarrow H_2O$

  (switch off pressure gauges!!)

  Remove O_2, increase T >200°C, remove H_2O

Building UHV apparatus

Sealing

- Connecting nipples, chambers etc. without leaks requires **sealing**, for example:
  - KF (quick flange): rubber/Viton O rings; use only for medium and high vacuum

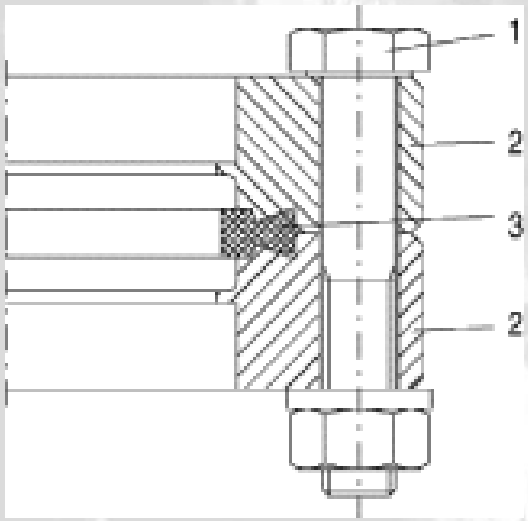
    (*e.g.* forevacuum)
  - Viton: bakeable to 140°C, rubber not…

Hammer test:
How to distinguish between Viton & rubber?

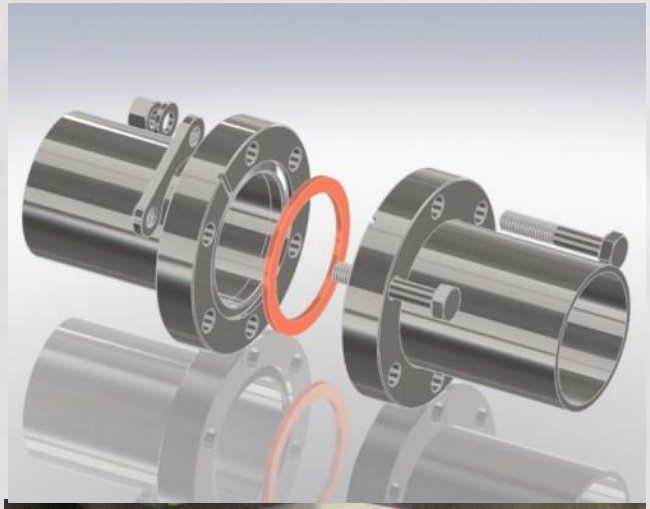
Land hammer on ring
Bounce back? ⇒ Rubber
Impact absorbed by ring? ⇒ Viton

UHV sealing

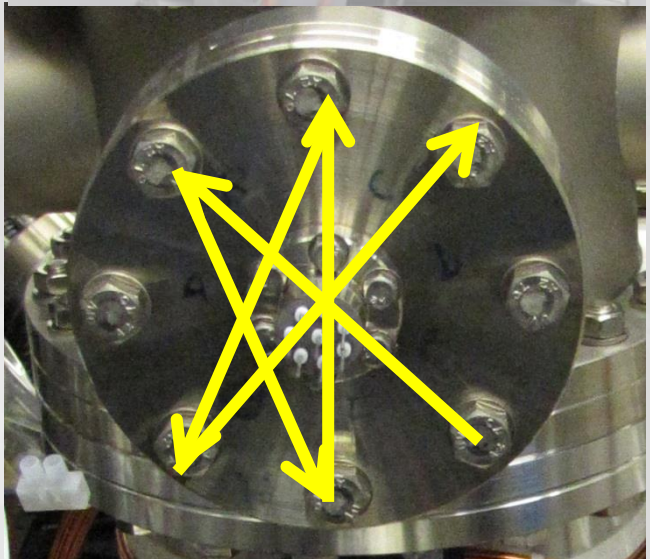
- CF (ConFlat): copper gasket clamped between knive edges, UHV seal



1. Flange bolt
2. CF flange
3. Copper gasket



Tighten screws in a zig-zag star pattern:

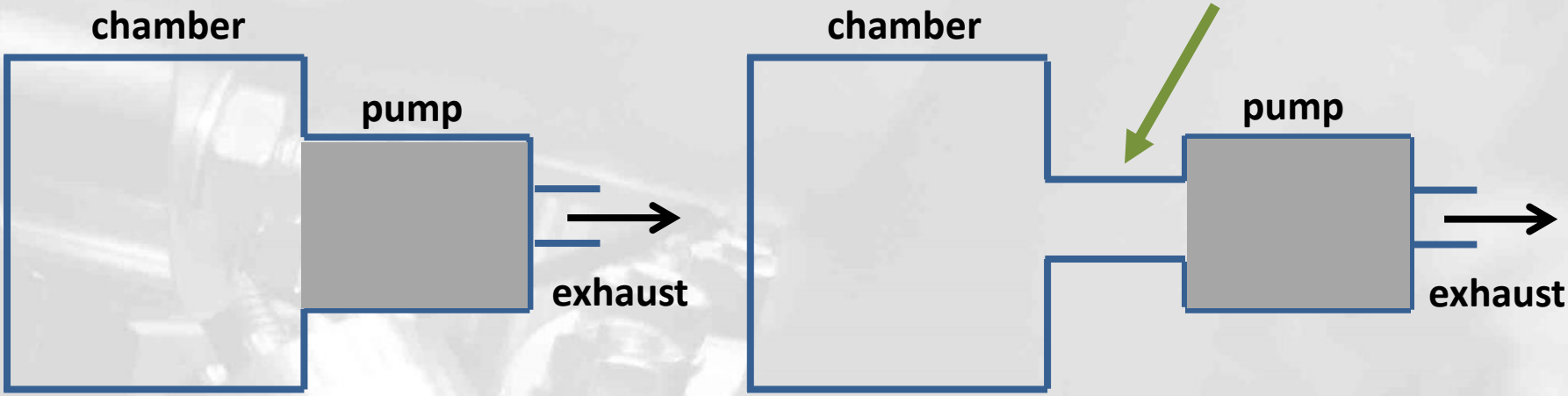


Use of vacuum grease

- very low vapour pressure (a few 10^{-12} mbar @ 25°C)
- use for lubrication (vacuum seals)
- do not bake-out
- do not use in uhv
- creep?

UHV chamber design

- Pump speed should not be reduced by **flow resistance**



- Molecular flow regime: gas flow (throughput) and pressure are essentially governed by Kirchhoff's laws for electrical circuits:

$$(V_i - V_f) = I\,R$$

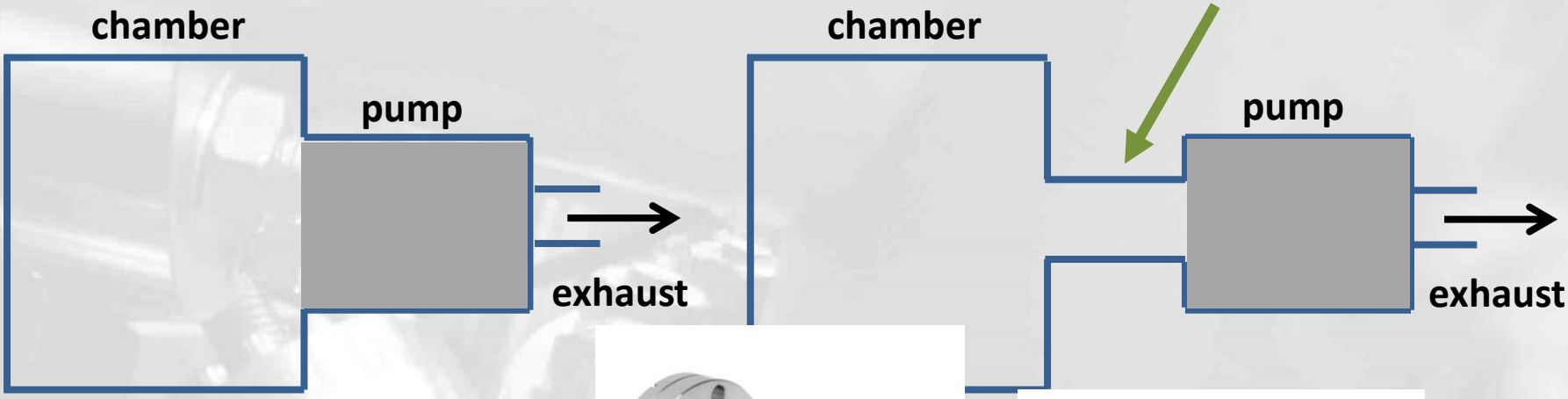
$$(p_i - p_f) = q\,W = q \times (1/L)$$

[mbar l/s] [l/s]

- Conductivity L scales intuitively, and standard expressions readily available

UHV chamber design

- Pump speed should not be reduced by **flow resistance**



- Molecular flow regime: ghp are esse by s fo s:

= *l*

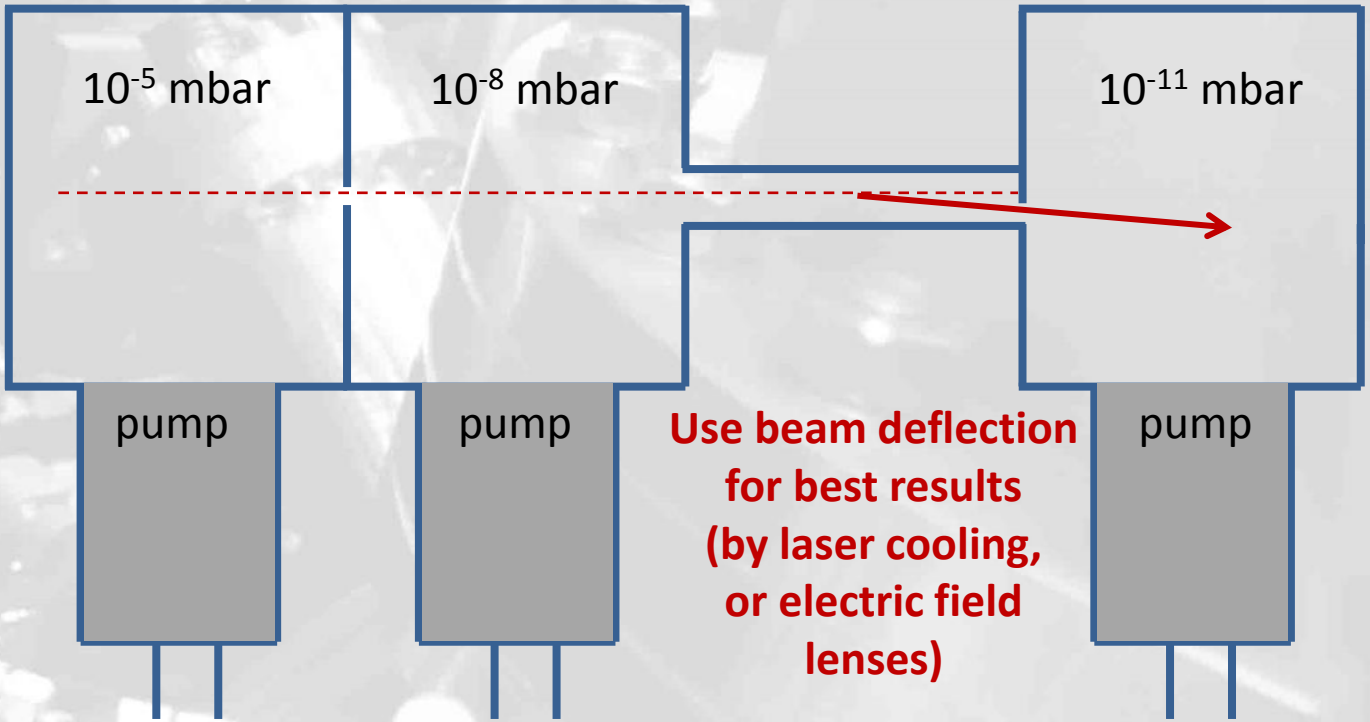
= *q*

bar l/s] [l/s]

- Conductivity *L* scales intuitively, and standard expressions readily available

Differential pumping

- Some vacuum setups necessarily deal with large pressure differences
  - E.g. ion production in atomic/molecular beam, transfer to UHV
- Use differentially pumped vacuum stages



Leak detection

- Occur most often at seals and glass-metal transitions
- Acetone test: spray aceteone and look for pressure changes in either direction
  - Local thermal shock (due to evaporating acetone); leak can either get better or worse (lower or higher *p*)
  - Acetone can 'seal' the leak, or creep through it (lower or higher *p*)

- leak detection with soap water --> look for bubbles

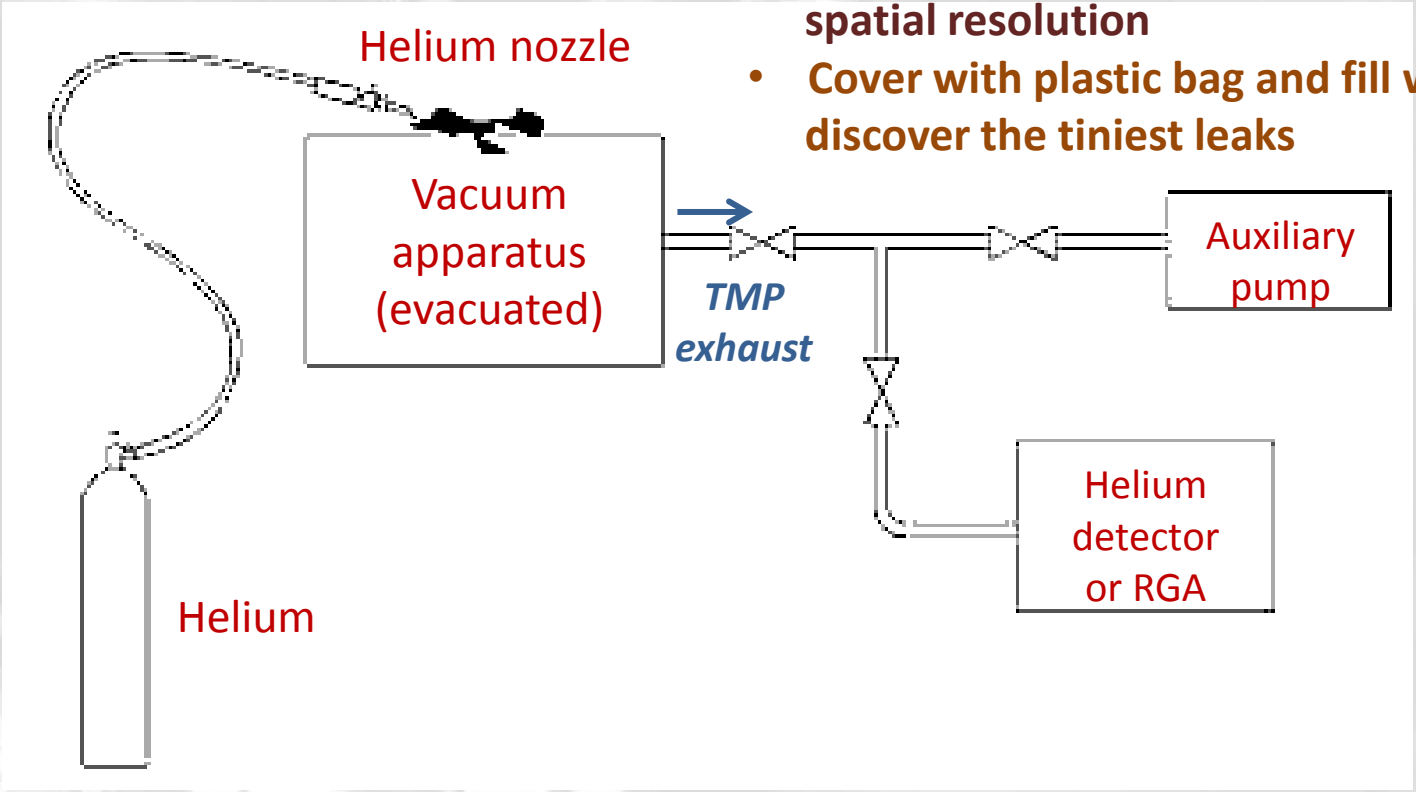
Leak detection (2)

Helium leak detection: high sensitivity, down to 10^{-11} mbar l/s

Example: 5×10^{-11} mbar l/s leak combined with 100 l/s pump

$\Rightarrow p = 5\times10^{-13}$ mbar

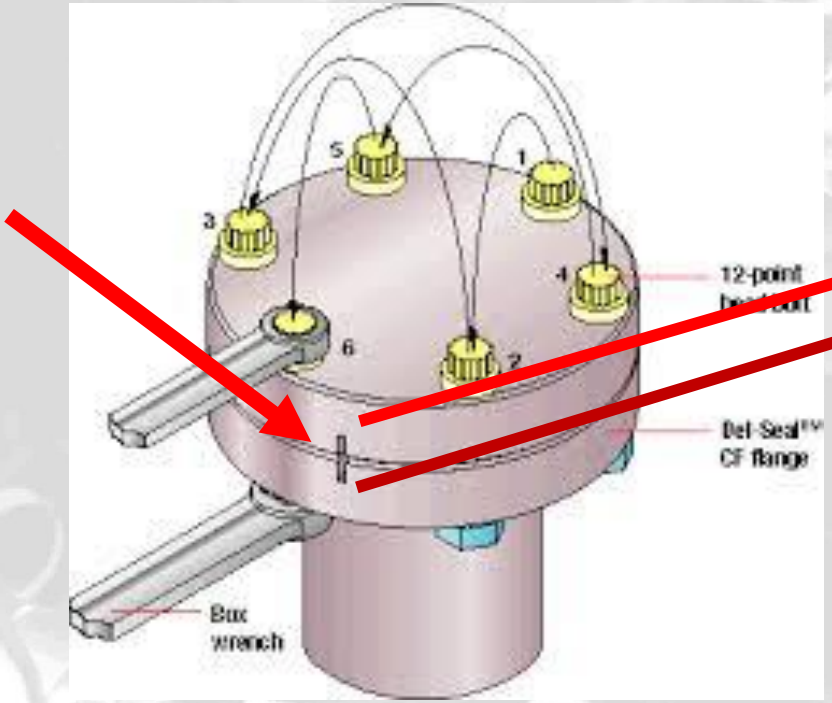
- **Start at the top of your setup (He floats upward!)**
- **Locally spray small amounts of He for best spatial resolution**
- **Cover with plastic bag and fill with He to discover the tiniest leaks**

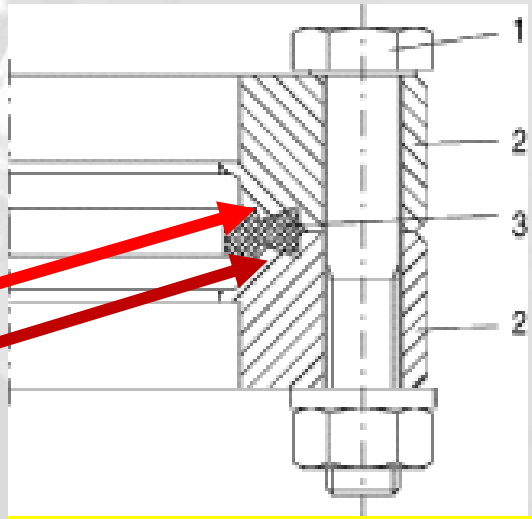


Oh yeah, about connecting CF flanges…

- It seems correct to mount CF flanges with the slots on the side facing each other

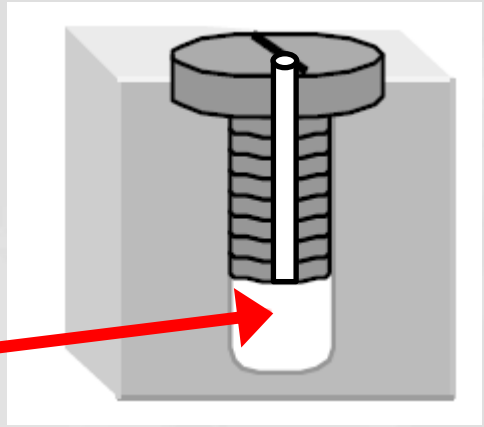
… but it is not





Better to rotate flange so that slots are far apart – allows detecting leaks on individual sides of copper gasket!

Vacuum parts



- Avoid virtual leaks due to trapped air (*e.g.* in blind tapped holes); use vented screws or through holes
- Same applies for welding
- Clean parts thoroughly! For example:
  - **Ultrasonic bath in water and dish-washing detergent or Alconox**
  - **Rinse with demi water and ultrasonic bath**
  - **Rinse with ethanol and ultrasonic bath (ethanol dissolves water)**
  - **Rinse with pure (HPLC-grade) acetone and ultrasonic bath**
  - **Rinse with pure (HPLC-grade) isopropanol and ultrasonic bath**
  - **Use cleanroom grade tin/alu foil, NOT the supermarket stuff (contaminated with organic grease)**
  - **Use cleanroom lint-free paper or towels**
  - **Use clean, powder-free gloves throughout**
  - **Use clean tools, and wrap hand grips in clean alu foil**
  - **Keep dust away, don't breathe/cough/spit on your parts**

UHV feedthroughs

- Mechanical (motional) feedthroughs exist
- Fiber-optic feedthroughs exist
- Electrical feedthroughs: pay attention to
  - Electrode material: max. current rating (steel vs. copper), max. voltage rating, magnetic properties (nickel)
  - Trap RF feedthrough: max. voltage rating is specified for DC, this can differ greatly from performance at RF (10 – 100 MHz)!

UHV compatible materials

- If uncertain, check vapor pressure curves!
- Check outgassing properties at NASA or ESA websites

  Google *e.g.* “NASA outgassing”
- In recent years, many UHV-compatible materials have been discovered by ion trapping community
- Too many to list here, but sufficient to say that UHV-compatible **solders**, **epoxies**, **dielectrics** do exist!
- Ask someone of the larger ion trapper research groups

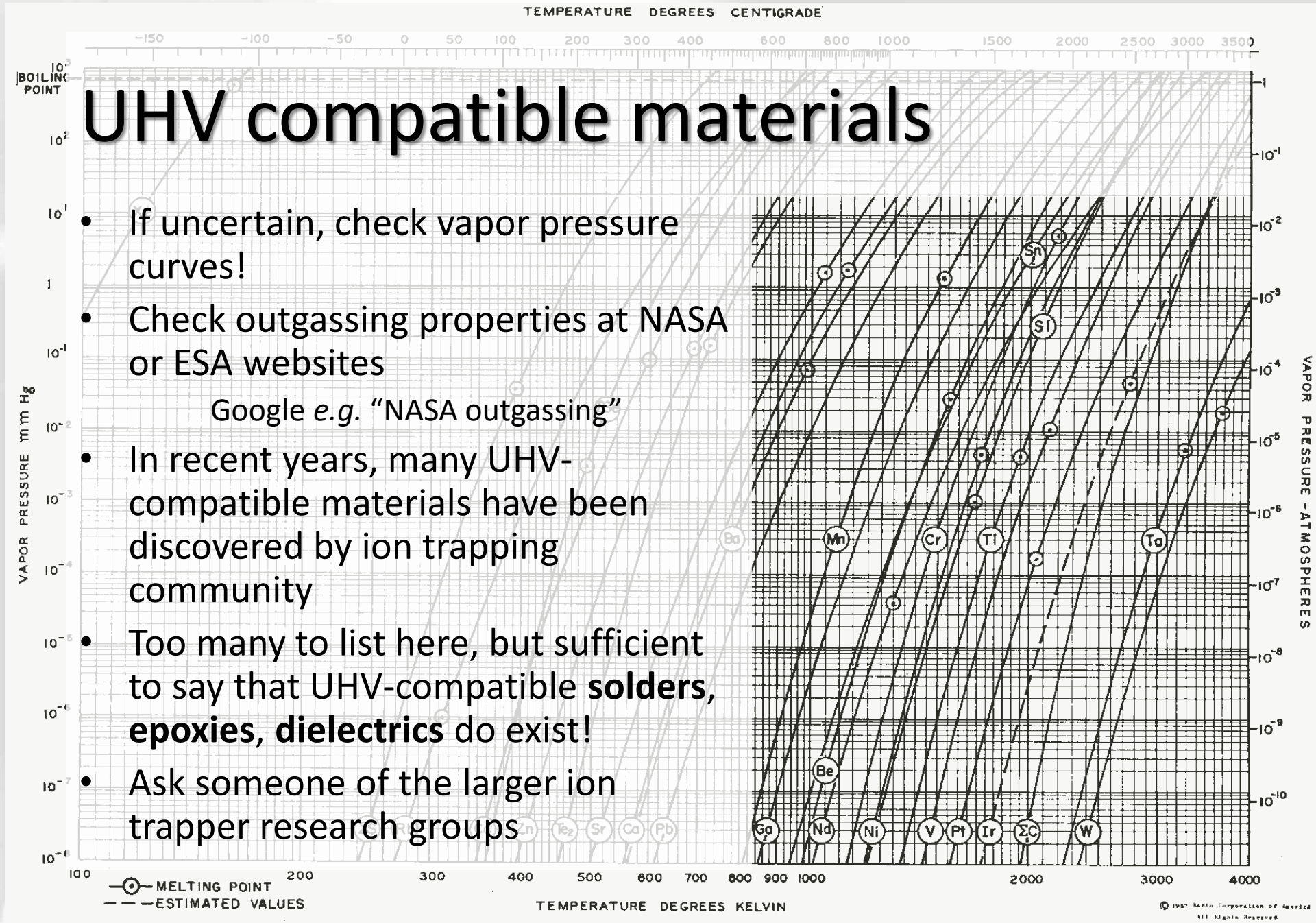


Figure A1(b). Vapor pressure curves for the more common elements (cont.). After Honig (Ref. 5:14). (*Courtesy RCA Laboratories.*)

Preparing for bakeout:

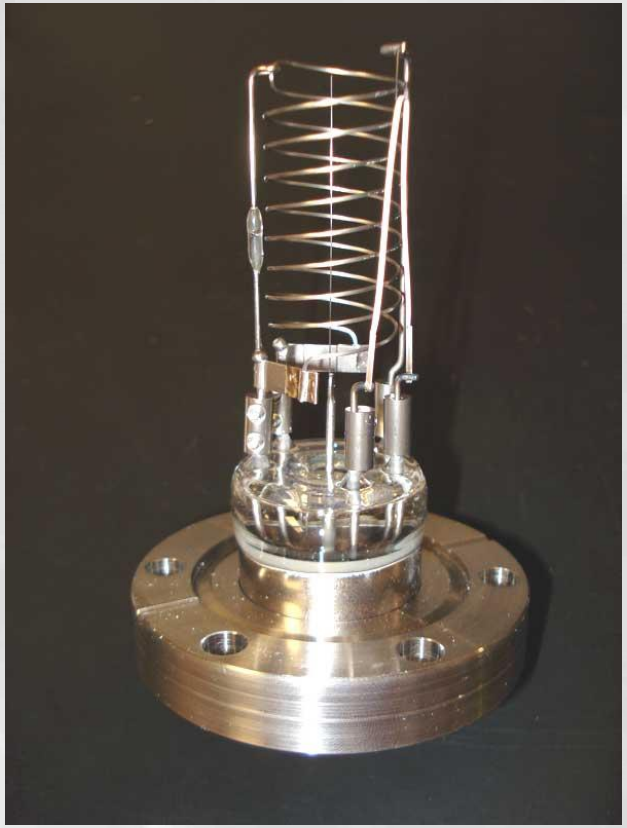
- Use multiple temperature sensors to monitor *T* gradients
- Use several independent heaters to minimize *T* gradients
- Avoid cold spots: desorbed gas will condense back onto them and slow down/stall gas removal
- Avoid hot spots near glass-to-metal transitions and CF flanges (leaks may appear & bolts may loosen up - spontaneously!)
- Tip: build an ‘oven’ with reflective walls (multiple layers of tin/alu foil), use radiative heaters and oven fan to circulate air
- Keep pressure gauge/ion pump/TSP cables out of hot zone
- Oven, e-gun feeds: use Kapton wire or bare copper wire insulated by ceramic beads
- Connect TSP & ion pump controller cables, keep access to all-metal valve to TMP inlet

During bakeout

- Avoid gradients at glass-metal, ceramic-metal transitions! (rule of thumb: $\Delta T < 15$ K)
- Better be patient when increasing T (< 15 K/hr)
- Keep record of bakeout stats
- Once pressure < 10^{-6} mbar: close inlet TMP, and switch on ion pump (if applicable)
- After pressure leveled off (~10^{-9} mbar), decrease T somewhat (<150°C) and degas e-guns, ovens:
  - Run overnight at ~75% of nominal operating current
- **At same *T*: fire TSP filaments to degas filaments, and to deposit Ti sticky layer**

UHV pressure monitoring

- UHV Bayard-Alpert gauge
  (10^{-4} – 10^{-12} mbar range)
- Ionization of background gas constituents & current measurement
- Note: different calibrations for different gas species
- Observe vacuum conductance towards gauge
- After exposure to high pressure: degas filaments
- Electric charging by nearby gauge has been observed – better to keep distance/use shielding between gauge and ion trap, EM detectors, …



Pressure > 10^{-4} mbar: use Pirani-type gauge

Venting your UHV apparatus

- *Oh noes – something broke down and can't be fixed without opening the UHV chamber… now what?*
- Do NOT vent with air – use dry nitrogen or pure argon or helium instead
  - Pores/holes in metal walls will be filled with the first atoms/molecules that get there – and N_2 is way easier to remove afterwards than H_2O!
  - Purge chamber with dry N_2 while it is open
  - UHV may be restored after pumping down and/or after a mild (70 - 90°C) bakeout

Some good venting advice…

- Use a pressure indicator to avoid overpressure (>1 atmosphere) – glass and ceramics are often constructed to withstand overpressure from outside, NOT from inside
- A manometer or even a simple rubber balloon may do the trick…

Credits

- Thanks to all vacuum experts I have known at:

  VU University Amsterdam (Netherlands)

  ENS Paris (France)

  NIST Boulder (USA)

  H.-H. University Düsseldorf (Germany)

Google

Agilent website (former Varian Inc.)
Pfeiffer Vacuum website