Vacuum Systems

Joint ICTP-IAEA Workshop on Accelerator Technologies, Basic Instruments and Analytical Techniques

21 – 29 October 2019

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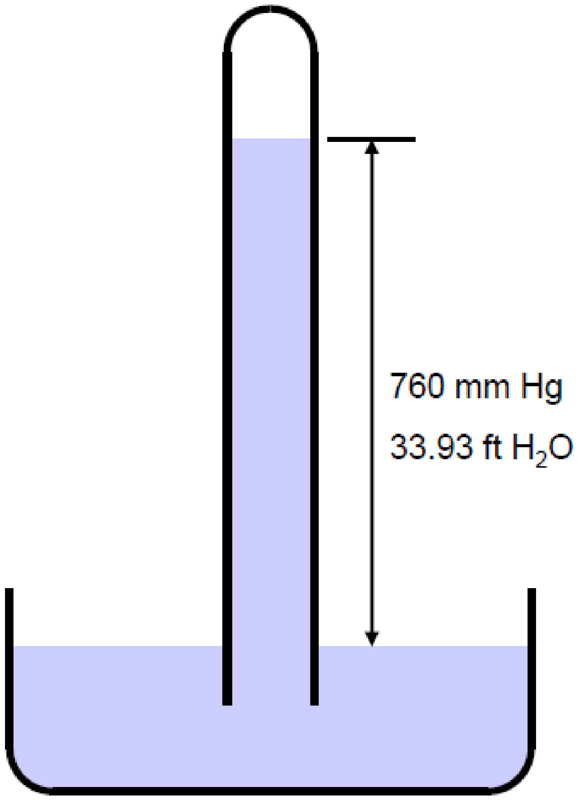
Overview of the lecture

- General background
- Gas laws and mean free path
- Different types of vacuum pumps
- Different types of vacuum gauges
- Leak detector
- Residual gas analysers

Units of Pressure Measurement

- 1 atmosphere =
  - 760 mm Hg = 760 torr
  - 760,000 millitorr or microns
  - 29.9213 in. Hg
  - 14.6959 psi
  - 1.01325 bar
  - 1013.25 millibar
  - 101,325 pascals (Pa)
  - 407.189 in. H_2O
  - 33.9324 ft. H_2O



1 Pascal = 1 N/m^2
1 torr = 1 mm Hg
1 micron = 1 μm Hg

SI units of pressure is Pascals (1 pascal = 1 newton per square metre, 1 N/m^2).

Partial Pressures of Gases in Air at STP

Gas	Symbol	Volume Percent	Partial Pressure, Torr
Nitrogen	N_2	78	593
Oxygen	O_2	21	159
Argon	Ar	0.93	7.1
Carbon Dioxide	CO_2	0.03	0.25
Neon	Ne	0.0018	1.4×10^{-2}
Helium	He	0.0005	4.0×10^{-3}
Krypton	Kr	0.0001	8.7×10^{-4}
Hydrogen	H_2	0.00005	4.0×10^{-4}
Xenon	Xe	0.0000087	6.6×10^{-5}
Water	H_2O	Variable	5 to 50, typ.

Standard Temperature and Pressure (STP): 0°C and 1 atm.

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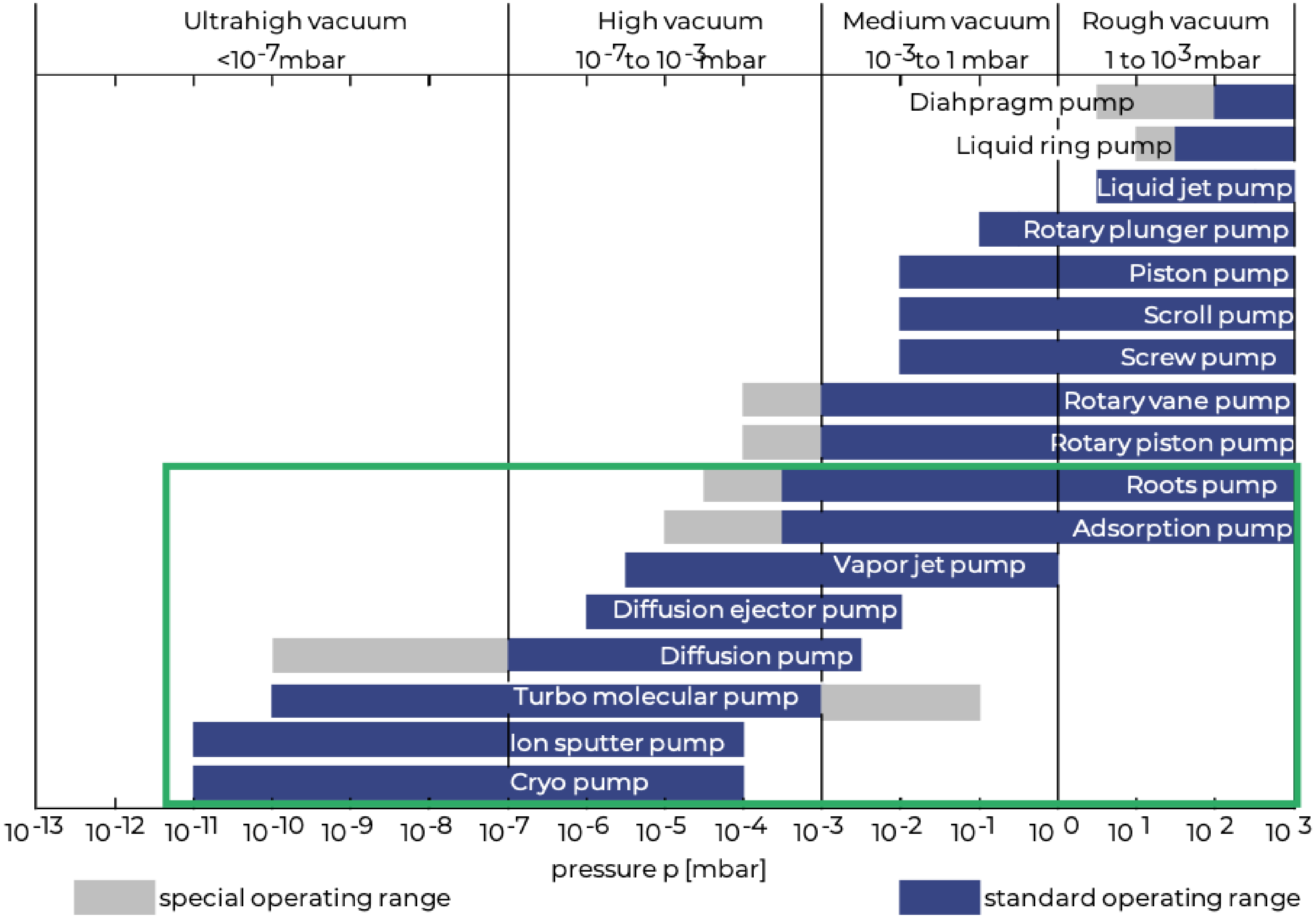
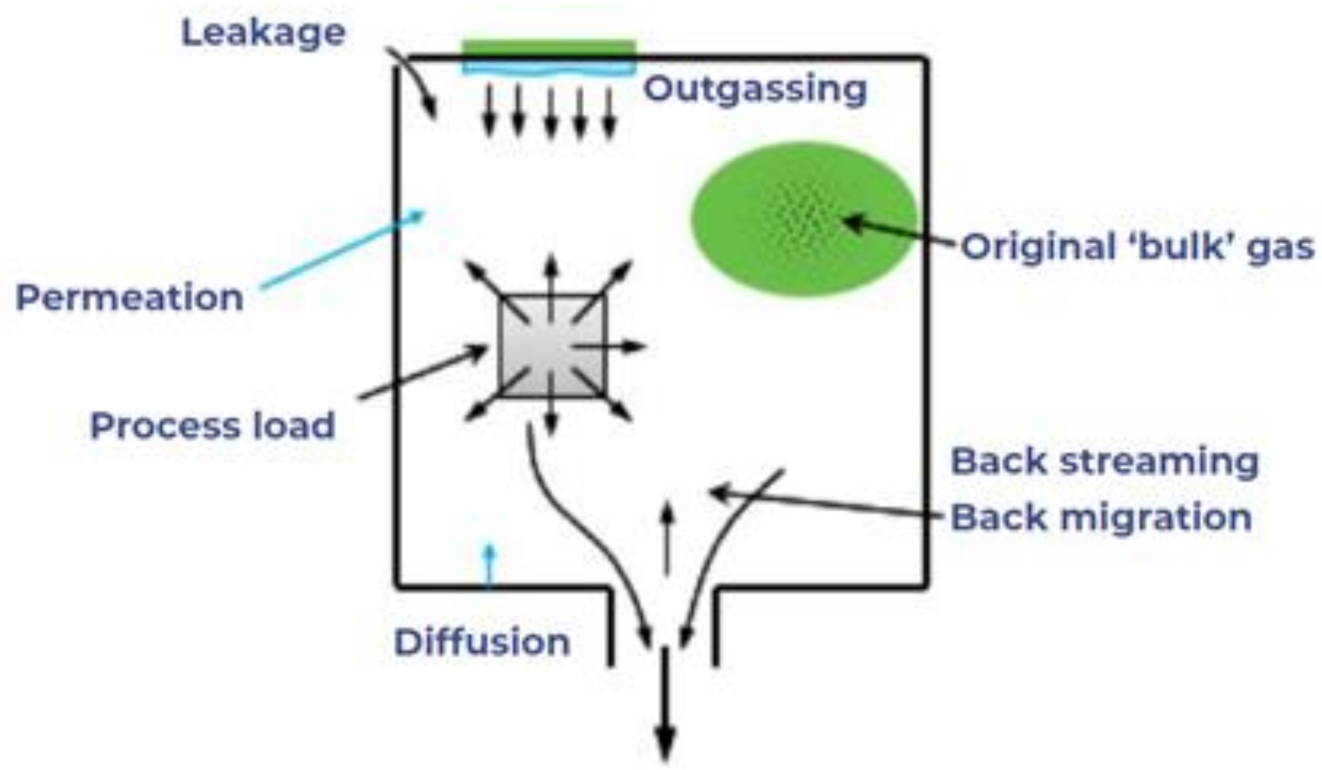
Different ranges of Vacuum, Rough, Medium, high and ultrahigh



Source: Leybold UK

There are six main contributions to the gas load within a system

1. the initial gas contained in the system/chamber
2. the gas load due to the pumping process
3. entrapped gases (which will add further to the load)
4. back-streaming/migration (which can be reduced by using cold traps, anti-suck-back valves etc.)
5. outgassing (which is a major contributor to gas load and is the hardest to counteract)
6. and leaks



Rate of outgassing of different materials

At room temperature									
Standard values[1)]			**Metals**				**Non-Metals**		
(mbar · l · s^{-1} · cm^{-2})			10^{-9} ... · 10^{-7}				10^{-7} ... · 10^{-5}		
Outgassing rates (standard values) as a function of time									
Examples:	1/2 hr.	1 hr.	3 hr.	5 hr.	Examples:	1/2 hr.	1 hr.	3 hr.	5 hr.
Ag	1.5 · 10^{-8}	1.1 · 10^{-8}	2 · 10^{-9}		Silicone	1.5 · 10^{-5}	8 · 10^{-6}	3.5 · 10^{-6}	1.5 · 10^{-6}
Al	2 · 10^{-8}	6 · 10^{-9}			NBR	4 · 10^{-6}	3 · 10^{-6}	1.5 · 10^{-6}	1 · 10^{-6}
Cu	4 · 10^{-8}	2 · 10^{-8}	6 · 10^{-9}	3.5 · 10^{-9}	Acrylic glass	1.5 · 10^{-6}	1.2 · 10^{-6}	8 · 10^{-7}	5 · 10^{-7}
Stainless steel		9 · 10^{-8}	3.5 · 10^{-8}	2.5 · 10^{-8}	FPM, FKM	7 · 10^{-7}	4 · 10^{-7}	2 · 10^{-7}	1.5 · 10^{-7}

1 All values depend largely on pretreatment!

Source: Leybold UK

Ideal Gas Law - 1

- V = volume of enclosure
- N = number of molecules
- N_m = number of moles = N/N_A
- n = particle density = N/V
- P = pressure
- T = absolute temperature
- k_B = Boltzmann's constant = 1.381×10^{-23} J/K
- N_A = Avogadro's number = 6.022×10^{23} particles/mole
- R = Gas constant = $N_A k_B$ = 8.315 J/mole-K

$$PV = N_m RT$$

$$PV = Nk_B T$$

$$P = nk_B T$$

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Ideal Gas Law - 2

- Historical Laws for ideal gases:
  - Boyle's Law: $P_1V_1 = P_2V_2$ at constant T
  - Charles' Law: $V_1/T_1 = V_2/T_2$ at constant P
  - Gay-Lussac's Law: $V = V_0(1 + T/273)$
  - Henry's Law: $P_i = K_ix_i$ (partial pressure of a gas above a liquid)
  - Dalton's Law: $P = P_1 + P_2 + \ldots + P_n$ (sum of partial pressures)

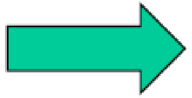
- Real gases are better described by van der Waals' equation of state:

$$\left(P + \frac{an^2}{V^2}\right)(V - nb) = nRT$$

  - an^2 term accounts for intermolecular attraction
  - nb term accounts for intermolecular repulsion

Mean Free Path

- MFP is the average distance a gas molecule travels before colliding with another gas molecule or the container walls.
- It depends only on the gas density and the particle size.
- σ is the diameter of the particles
- $\pi\sigma^2$ is the cross-sectional area for hard-sphere collisions



$$\mathrm{MFP} = \lambda = \frac{V}{N\pi\sigma^2\sqrt{2}} = \frac{k_B T}{P\pi\sigma^2\sqrt{2}}$$

For common gases, {H_2O, He, CO_2, CH_4, Ar, O_2, N_2, H_2}, at T = 300 K:

$$\text{Mean Free Path (cm)} = \frac{5 \times 10^{-3}\ \text{torr-cm}}{\text{Pressure (torr)}}$$

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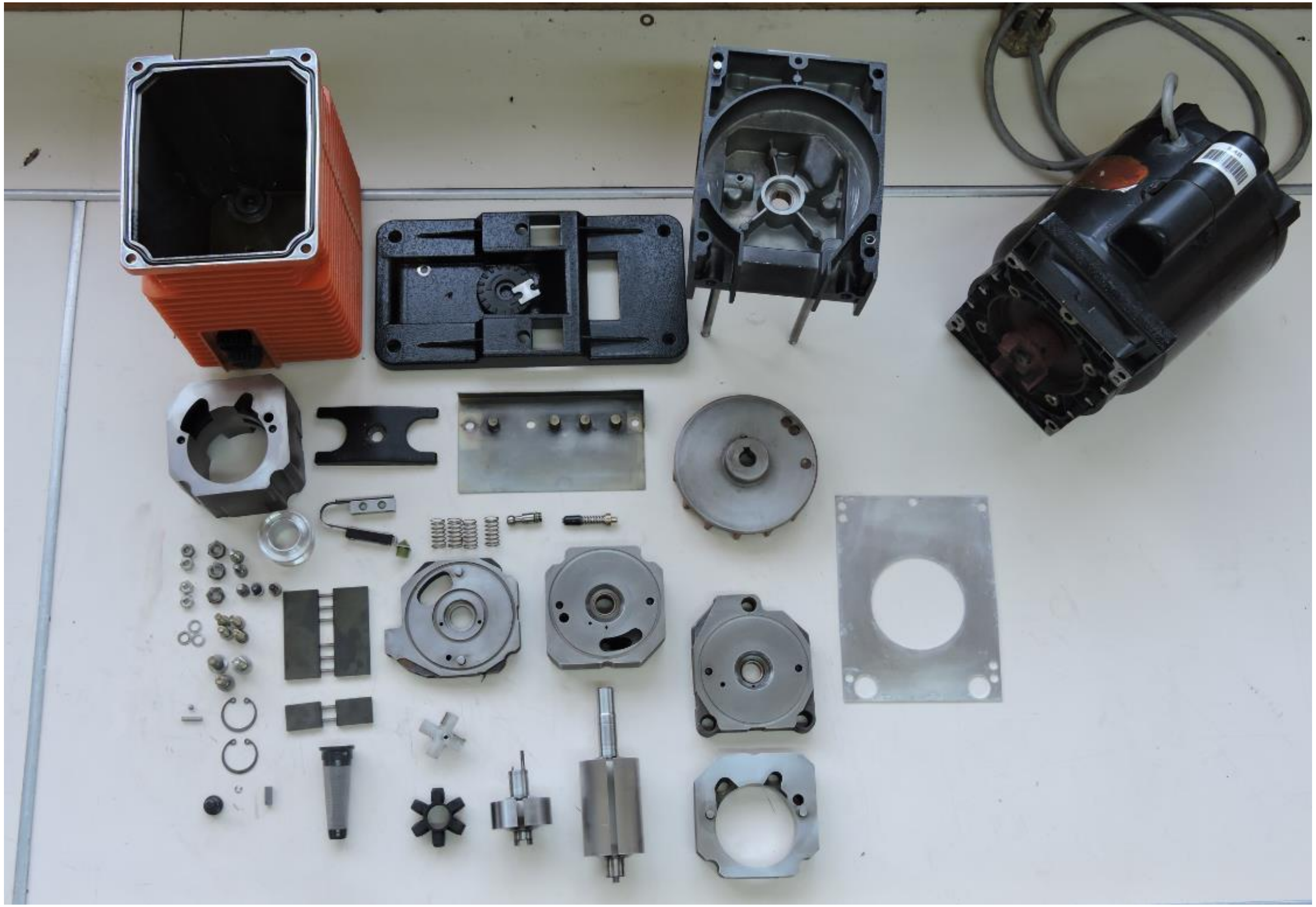
Vacuum Pumps

Two fundamental principles:

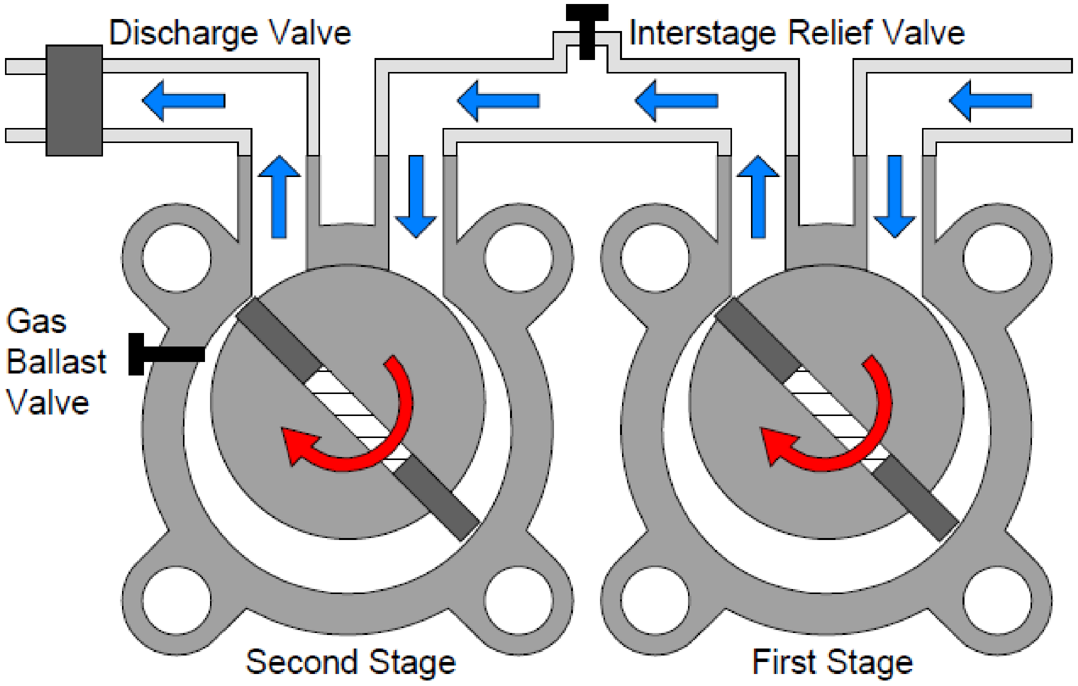
- Gas entering the pump is compressed and expelled out the pump (These pumps can run continuously)

- Gas entering the pump and get trapped, there is no outlet (these pumps must be regenerated)

Rotary vane mechanical pumps



Rotary Vane Mechanical Pumps - 2



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Rotary Vane Mechanical Pumps - 3

- Gases are removed by compressing them slightly above atmospheric pressure and then forcing them through a check valve.
- The rotary vane modules are immersed in an oil bath.
- The purpose of the oil is to:
  - cool the pump
  - lubricate the rotary vanes
  - provide a lip seal for the vanes
  - open the second stage exhaust valve at low inlet pressures
- They are powered by an electric motor:
  - Belt drive: 250 to 400 rpm
  - Direct drive: 1725 rpm (most common type)

Rotary Vane Mechanical pumps

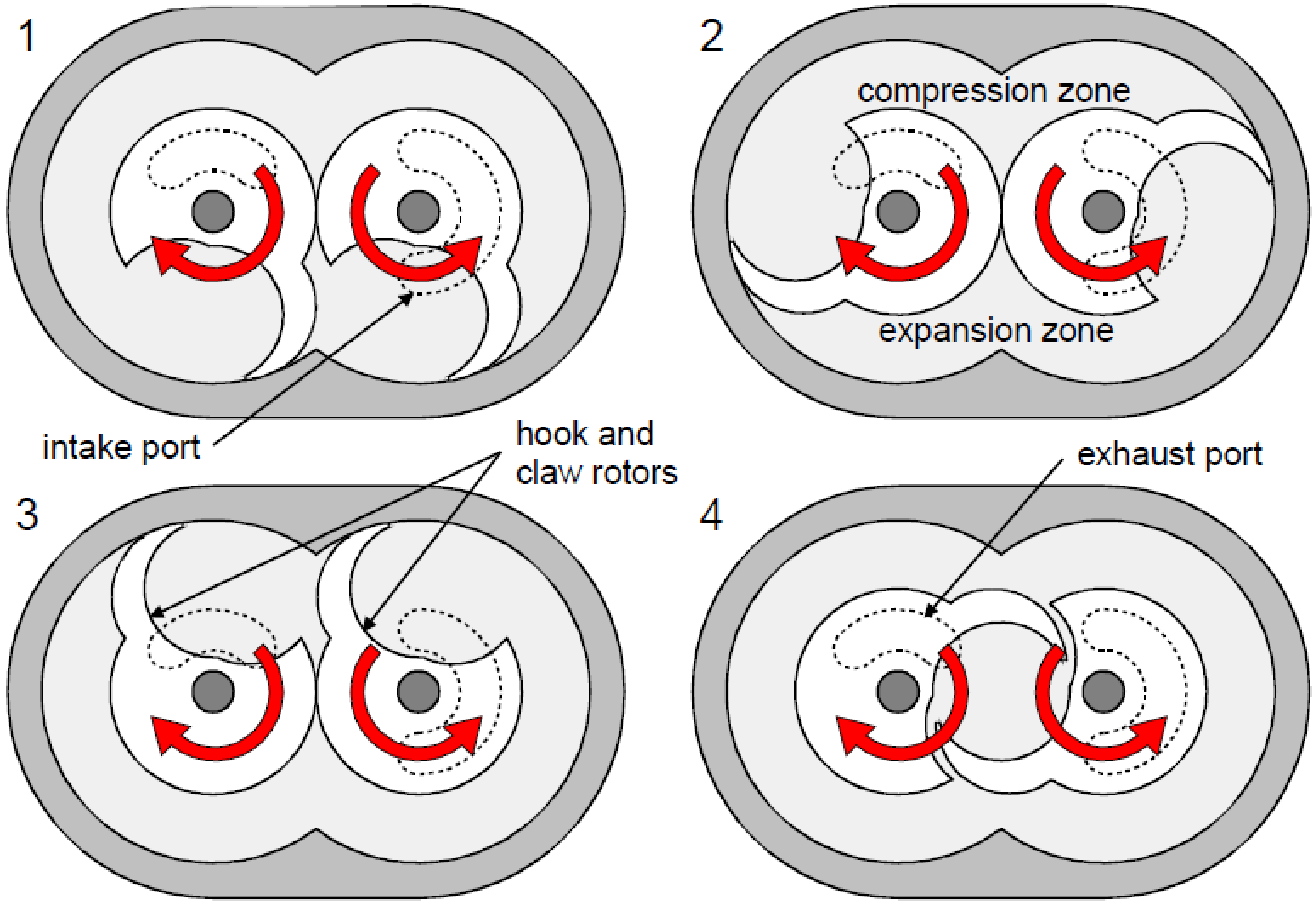
- Oil must have low vapour pressure to achieve the necessary performance
- Back streaming of oil can happen at low pressures
- Large gas loads can froth the oil and prevent sealing

Dry mechanical pumps

Dry mechanical pumps

- These pumps do not use oil, thus no oil back streaming
- Many of the designs can discharge directly into atmospheric pressure
- Most designs are base on two counter rotating shafts with claws and hooks that compress the gas.
- Quite expensive
- Typical pumping speed 500 to 1000L/min

Hook and Claw Style Dry Mechanical Pumps



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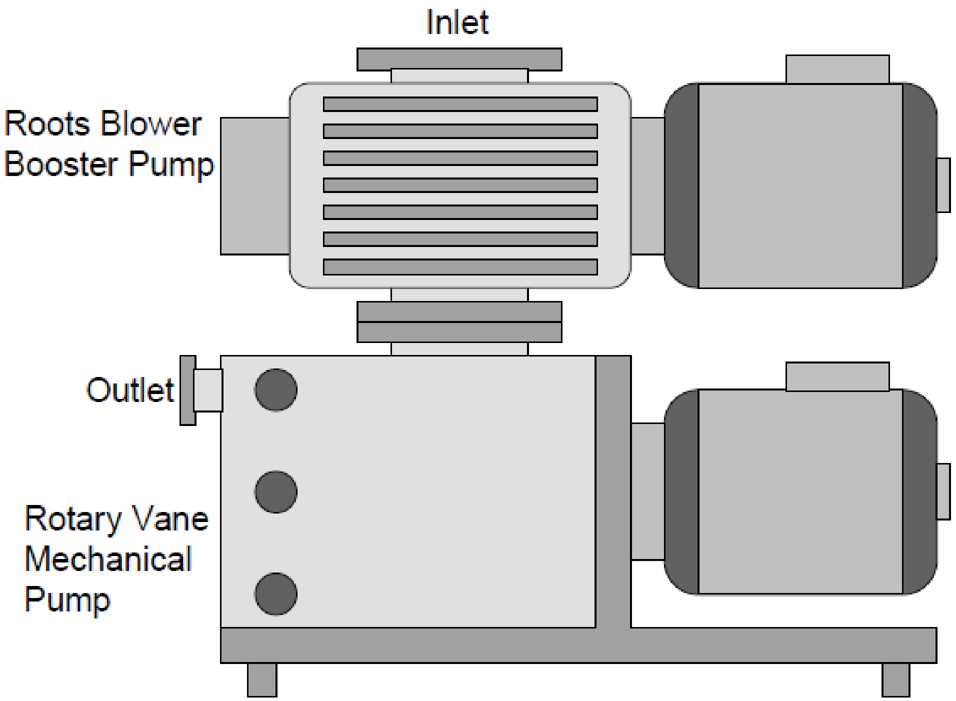
Roots Blowers / Booster Pumps - 1



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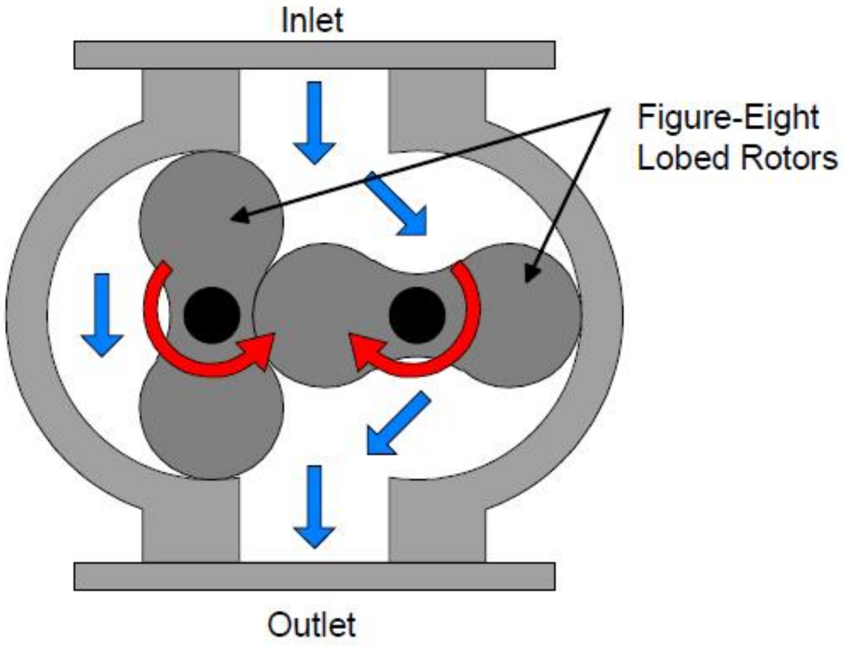
Roots Blowers / Booster Pumps - 2



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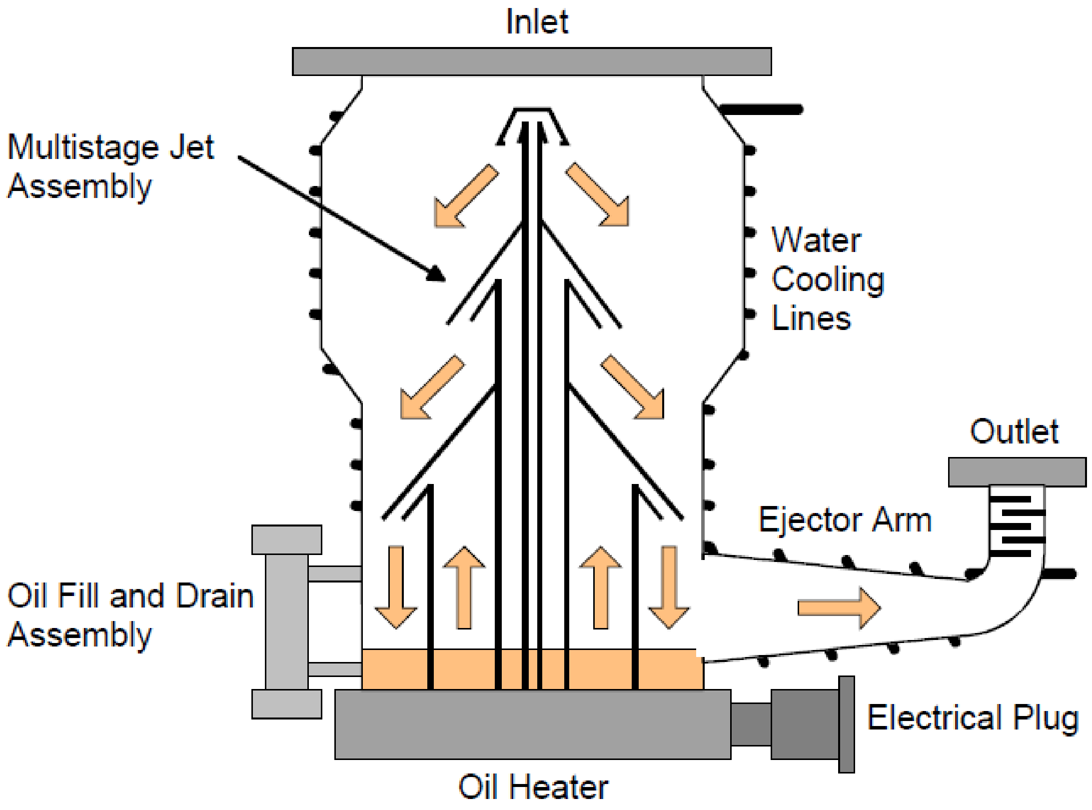
Oil Diffusion pumps

Diffusion Pumps - 2



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Diffusion Pumps - 3

- Oil is vaporized and propelled downward by an internal boiler and multistage jet assembly.
- Oil vapor reaches speeds of 1220 km/h or more
- Oil vapor streams trap and compress gases into bottom of pump, which are then ejected out into the foreline arm.
- Oil vapor is condensed on sides of pump body which are water cooled.
- Can only operate at foreline pressures of ~100 millitorr or less.
- A mechanical foreline pump is required for operation.
- Multistage jet assembly is designed to fractionate the oil, using lighter weight fractions for higher vapor velocities.
- Typically 300 - 2800 L/s pumping speeds.
- Very high reliability pumps, since there are no moving parts.
- Gravity collects oil in the base, so pumps must be mounted pointing upwards.

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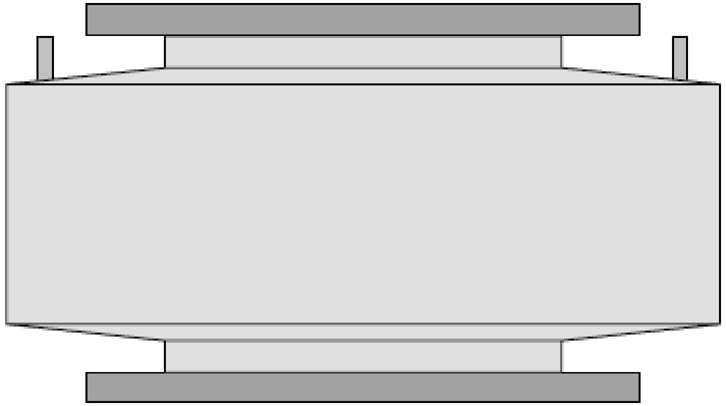
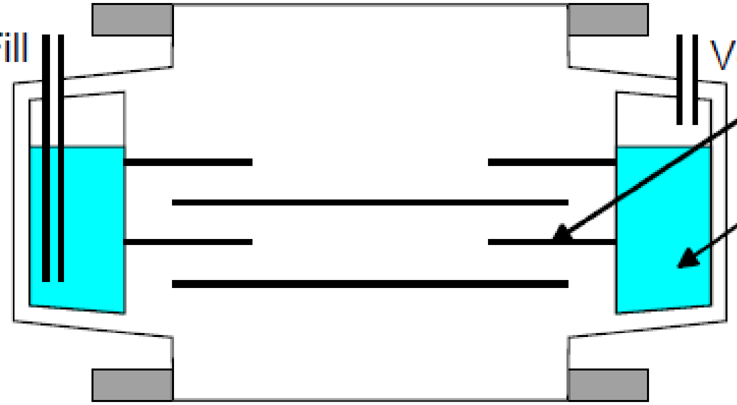
Diffusion Pumps - 4

- Potential Problems:
  - Backstreaming of oil vapor can occur if forepressure becomes too large.
    - Backstreaming occurs for pressures of 1 to 10 mTorr.
    - A cold cap on top of the multistage jet assembly helps to reduce this.
    - A liquid nitrogen filled cryotrap also helps to reduce this.
    - The maximum tolerable foreline pressure (critical fore pressure) must not be exceeded, or pump will "dump" or "blow-out", sending oil up into the chamber.
  - Pump can overheat if cooling water fails
    - Most pumps have a thermal cutout switch.
  - Pumping requires low vapor pressure oil
    - Water, dirt, or other impurities will raise vapor pressure.
    - Only special oils are suitable for diffusion pump use.

Liquid Nitrogen Traps / Baffles - 1

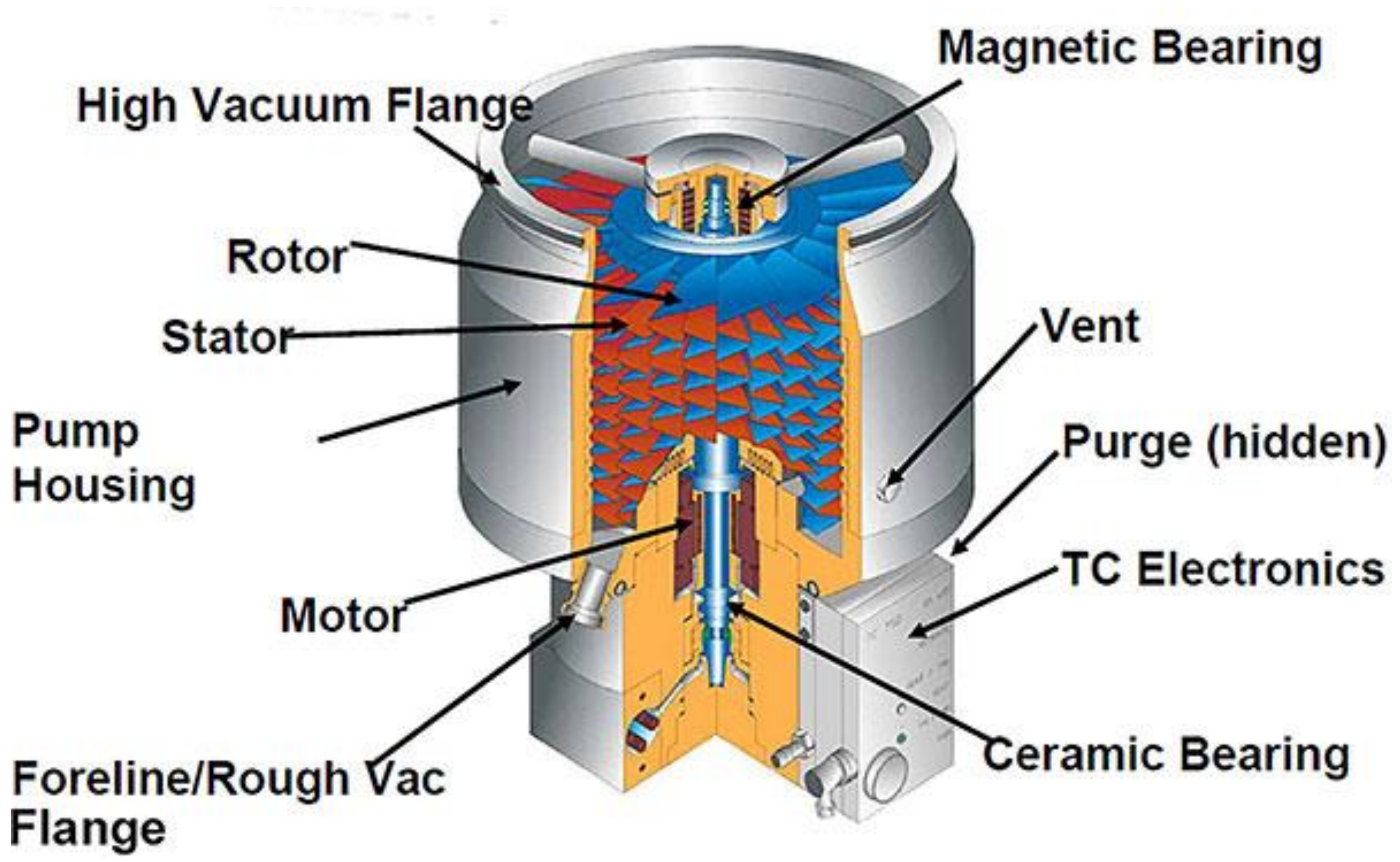




Liquid Nitrogen Traps / Baffles - 2

- Baffles and traps in the pumping lines can greatly help to reduce backstreaming:
  - low pressures in mechanical rough pumps (0.1 to 1.0 torr)
  - high pressures in diffusion pumps (1 to 100 millitorr)
  - most important within the "cross-over" region.
- LN_2 cryotraps should not experience air pressure above 100 millitorr, or they will frost completely over.
- Residual water in a cryotrap can be frozen and cause trap to break, causing catastrophic failure of vacuum system.
  - Blow out any water vapor with dry N_2 before filling with LN_2.
- LN_2 cryotraps require constant refilling.
  - Expensive, but autofill valves are available.

Turbomolecular Pump



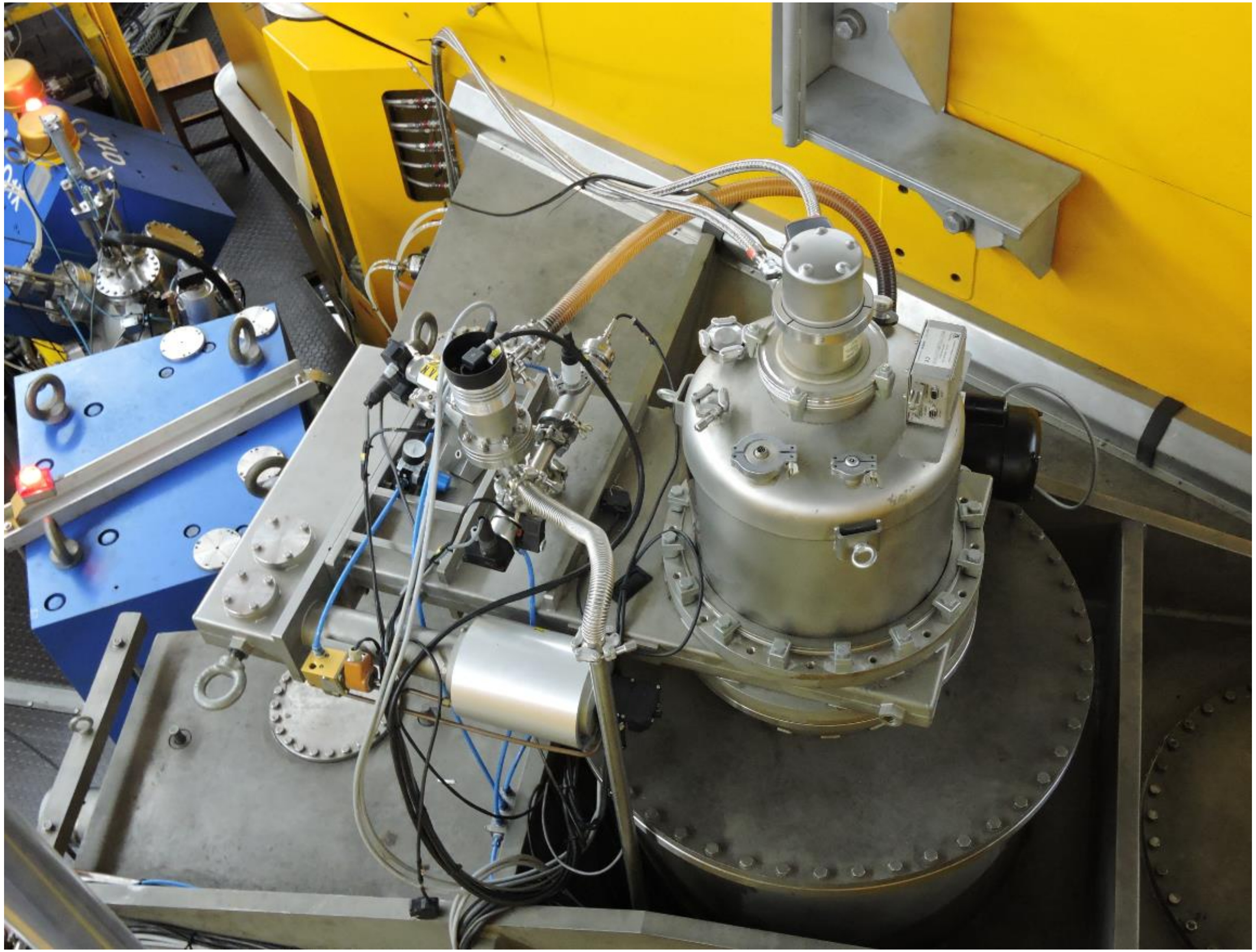
Turbomolecular Pumps

- Very clean mechanical pumps
- High speed rotational blades are used to transfer speed and directions to gas molecules
- Typical rotor speed are 9 000 to 90 000 rpm
- Require mechanical foreline pump
- Pumping speeds 100 to 3000l/sec

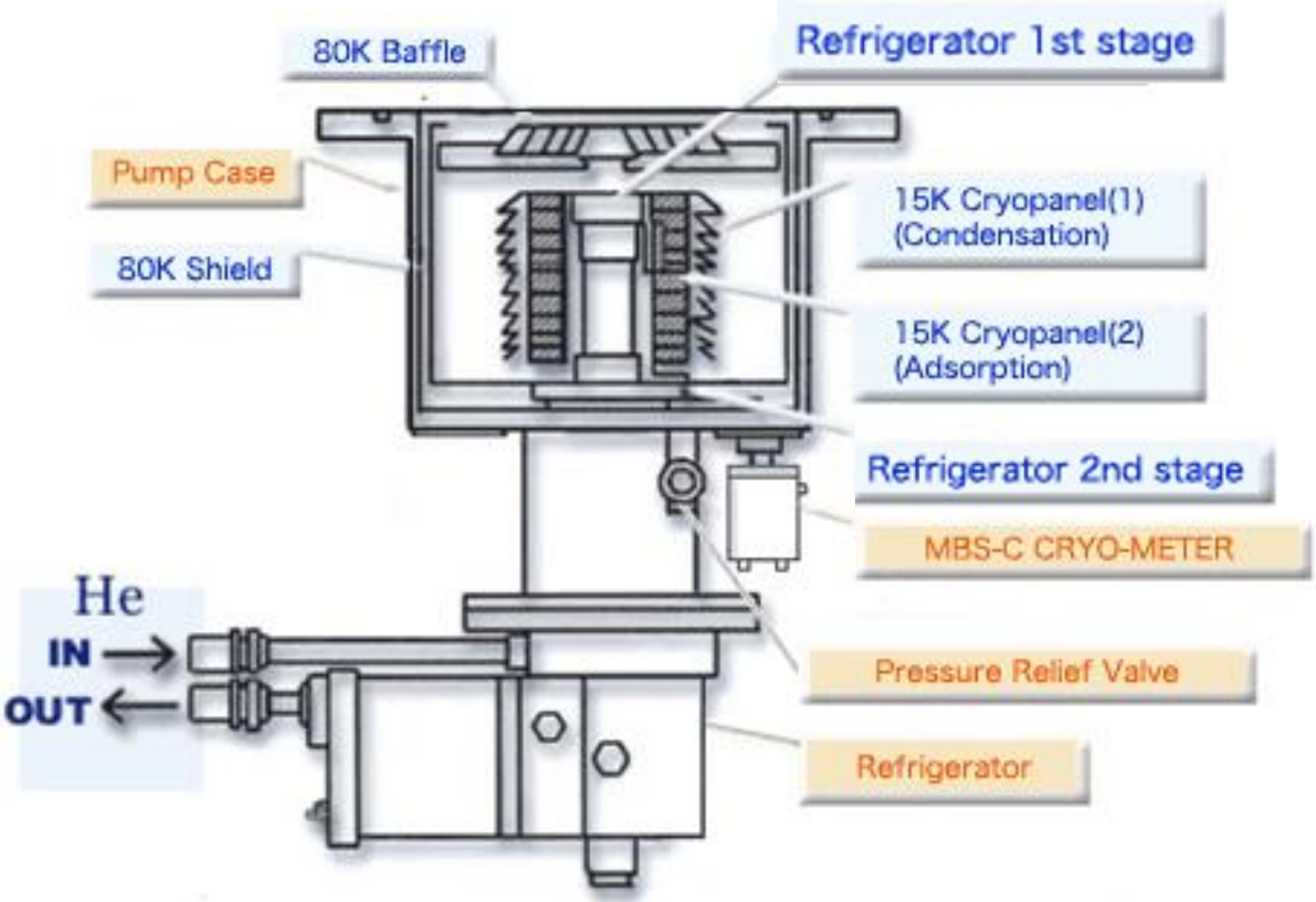
Turbomolecular Pumps - 4

- Potential Problems:
  - Very high speed rotor blades have close-mating stator blades.
    - Slight imbalances can cause vibration and bearing wear problems.
    - A sudden blast of atmospheric pressure can bend the blades down, causing catastrophic failure, "crashing the pump."
  - Lubrication of the high speed rotor is an engineering problem.
    - Circulating oil is most reliable, but pump must be right-side-up.
    - Grease-lubricated bearings are less reliable, but allow pump to be placed at any orientation.
    - Some fancier models use magnetic bearings.
  - Too high of a pressure will cause aerodynamic lift and drag.
    - A mechanical foreline pump must be used.
    - Aerodynamic lift can bend blades, causing catastrophic failure.


HiPACE
PFEIFFER VACUUM
P2VK
HiPACE



Cryopump



80K Baffle
Refrigerator 1st stage
Pump Case
15K Cryopanel(1)
(Condensation)
80K Shield
15K Cryopanel(2)
(Adsorption)
Refrigerator 2nd stage
MBS-C CRYO-METER
He
IN
OUT
Pressure Relief Valve
Refrigerator

Cryopumps - 3

- These use a closed-loop helium cryogenic refrigerator.
- The primary parts are:
  - Compressor: uses He for its high heat capacity
  - Expander: uses Joule-Thompson expansion of He gas for cooling
  - Cold Head: thermally insulated from pump bucket, 2+ stages
- Gases are pumped by two processes:
  - Cryocondensation (H_2O, CO_2, N_2, O_2, Ar, solvent vapors)
    - Gases are condensed into a solid phase on cryogenically cooled surfaces. (They become frost!)
  - Cryosorption (H_2, He, Ne)
    - Non-condensable gases are adsorbed onto surfaces of cryogenically cooled porous media, usually activated charcoal or zeolites.
- Typical pumping speeds are 100 - 1000 L/s.

Cryopumps - 4

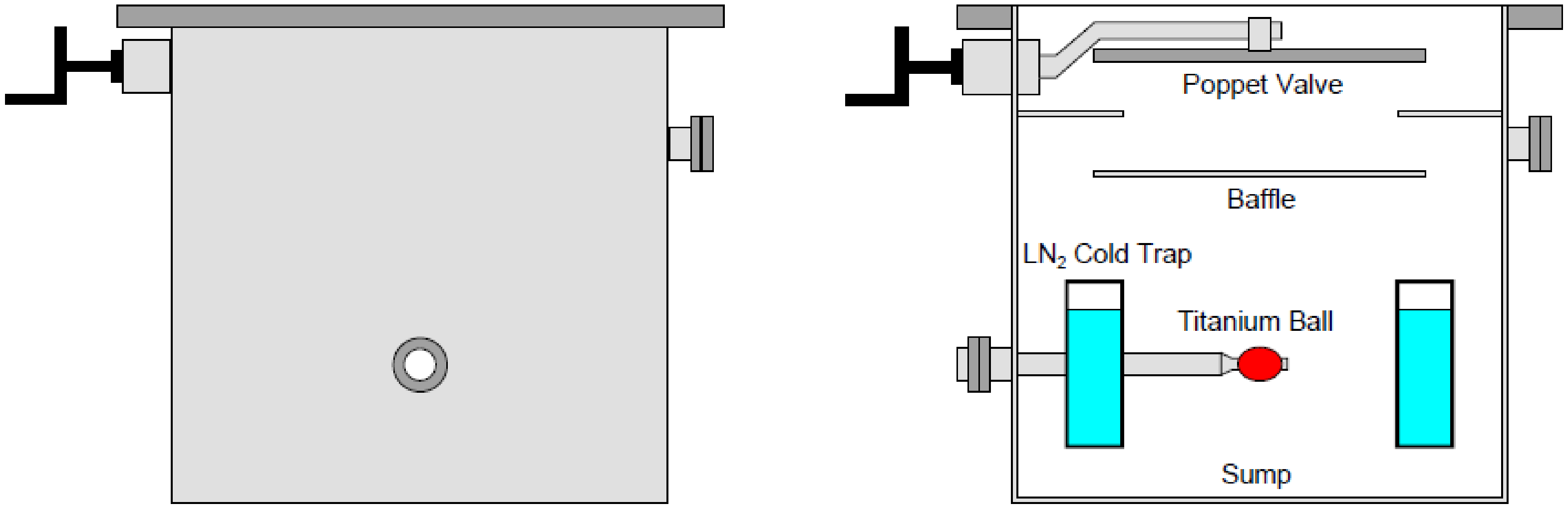
- The first stage array operates at 50 to 80 K.
  - Primarily used for pumping water vapor and carbon dioxide.
- The second stage array operates at 10 to 20 K.
  - Primarily used for pumping other condensable gases.
- Activated charcoal in the second stage provides cryosorption.
  - Primarily used for pumping other non-condensable gases.

- They offer completely oil free operation.
- They can operate from any orientation.
- They offer very clean vacuum with high pumping speed.
- They have very high impulsive pumping capacity.

Cryopumps - 5

- Potential Problems:
  - They must be regenerated to extract the trapped gases.
    - Allow to warm to room temperature (slow), or
    - Use a built-in heater to warm to 250 C and outgas (fast).
    - Regeneration takes the pump off-line for several hours.
  - The regeneration process can produce considerable pressure.
    - Pumps have a safety pressure relief valve on the bucket.
  - They must be started from below 100 millitorr.
    - They require the use a mechanical roughing pump to initially pump out the bucket, but once done, the rough pump is no longer needed.

Titanium Sublimation Pumps - 1



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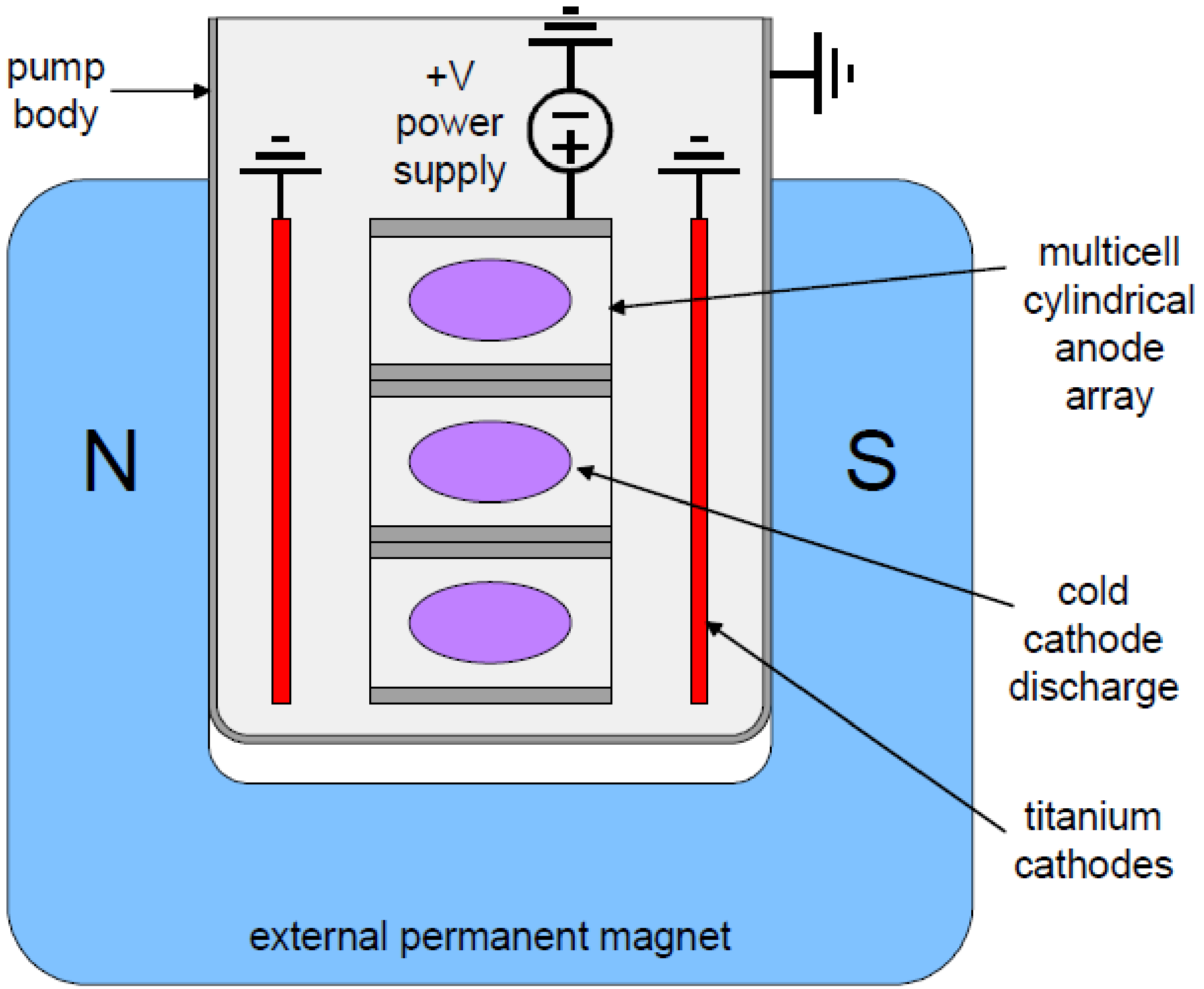
Titanium Sublimation Pumps (TSP)

- Titanium Sublimation Pumps (TSP) are true UHV pumps.
- It consists of a titanium filament through which a high current (typically around 40 Amps) is passed periodically. This current causes the filament to reach the sublimation temperature of titanium, and hence the surrounding chamber walls become coated with a thin film of clean titanium.
- Since clean titanium is very reactive, components of the residual gas in the chamber which collide with the chamber wall are likely to react and to form a stable, solid product. Thus the gas pressure in the chamber is reduced.
- But after some time, the titanium film will no longer be clean and hence the effectiveness of the pump is reduced. Therefore, after a certain time, the titanium filament should be heated again, and a new film of titanium re-deposited on the chamber wall

Non-Evaporable Getter Pumps

- These are also known as "NEG" pumps.
- They usually employ a Zr-V-Fe alloy that is formed into a cartridge over a constantan strip heater.
- These pump all of the time, until loaded with gas molecules.
- They can be regenerated by heating to ~350°C for 30 mins. to degas the alloy.
- They are very simple in construction and operation.

Ion Pumps - 1



Diode Ion Pump

Ion Pumps - 3

- Operation is based upon a rarefied gas electric discharge.
  - A high electric field can ionize a gas molecule, forming a free electron and a gas ion.
  - The free electron is collected by the anode, while the gas ion is collected by the cathode.
  - Fast electrons, accelerated by the E-field, will collide with and ionize other gas molecules.
  - A coaxial magnetic and electric field will produce spiral orbits for the free electrons; the larger paths greatly increase the ionization.
  - This creates a Penning cell which traps positive ions inside.
  - Higher ionization levels will sustain a cold cathode discharge.
  - Gas ions accelerated into the cathode can stick and therefore be pumped.

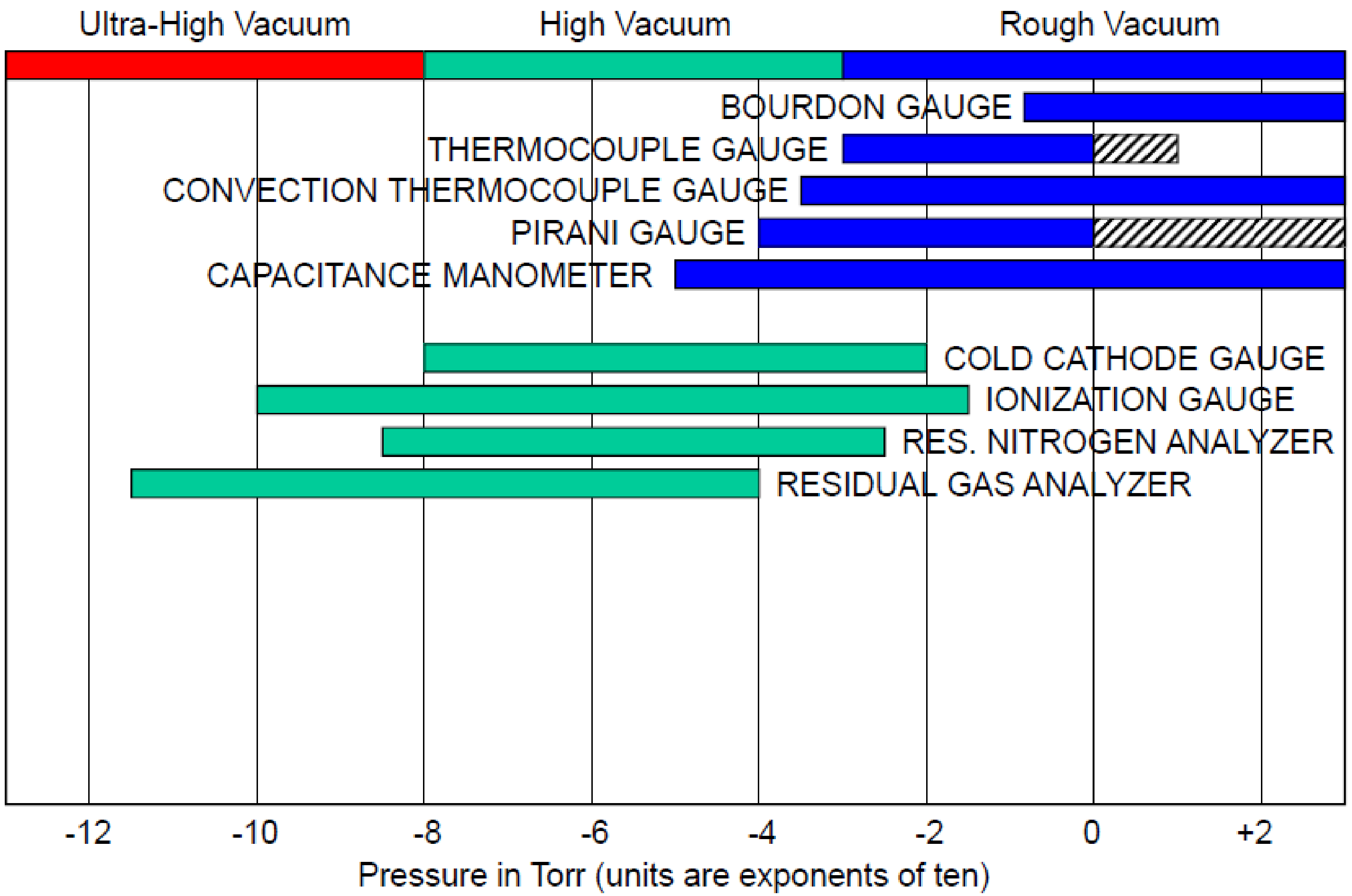
Ion Pumps - 4

- The cathode plates are made of titanium (Ti).
- Pumping mechanisms:
  - Incident gas ions may be implanted into the Ti cathode plates.
  - Incident gas ions may sputter Ti from the cathode plates into the cylindrical anode cells, thus providing additional getter pumping.
  - H_2 is directly absorbed by the fresh Ti surfaces.
  - Gas molecules may be trapped and buried by sputtered Ti.
  - The electric discharge cracks larger molecules into smaller ones that are more readily pumped.
- Ion pumps must be started at 10^{-5} torr or less.
- Intermediate pumping is usually provided by a sorption or a cryo pump.

Special Considerations for Ultra-High Vacuum Systems

- Achieving pressures below ~10^{-7} torr requires:
  - Extreme attention to the cleanliness of all surfaces
  - Elimination of all virtual leaks
  - Baking out of chamber to allow inside wall surfaces to desorb
    - Usually ~200-300°C for 6-12 hours
    - Special baking blankets or heater tapes are used on the exterior of the chamber
  - Patience to achieve the base pressures
    - Can sometimes take >24 hours
  - UHV systems often have a load-lock system so that the main chamber does not need to come up to full atmospheric pressure to load and unload samples

Vacuum Gauge Pressure Ranges



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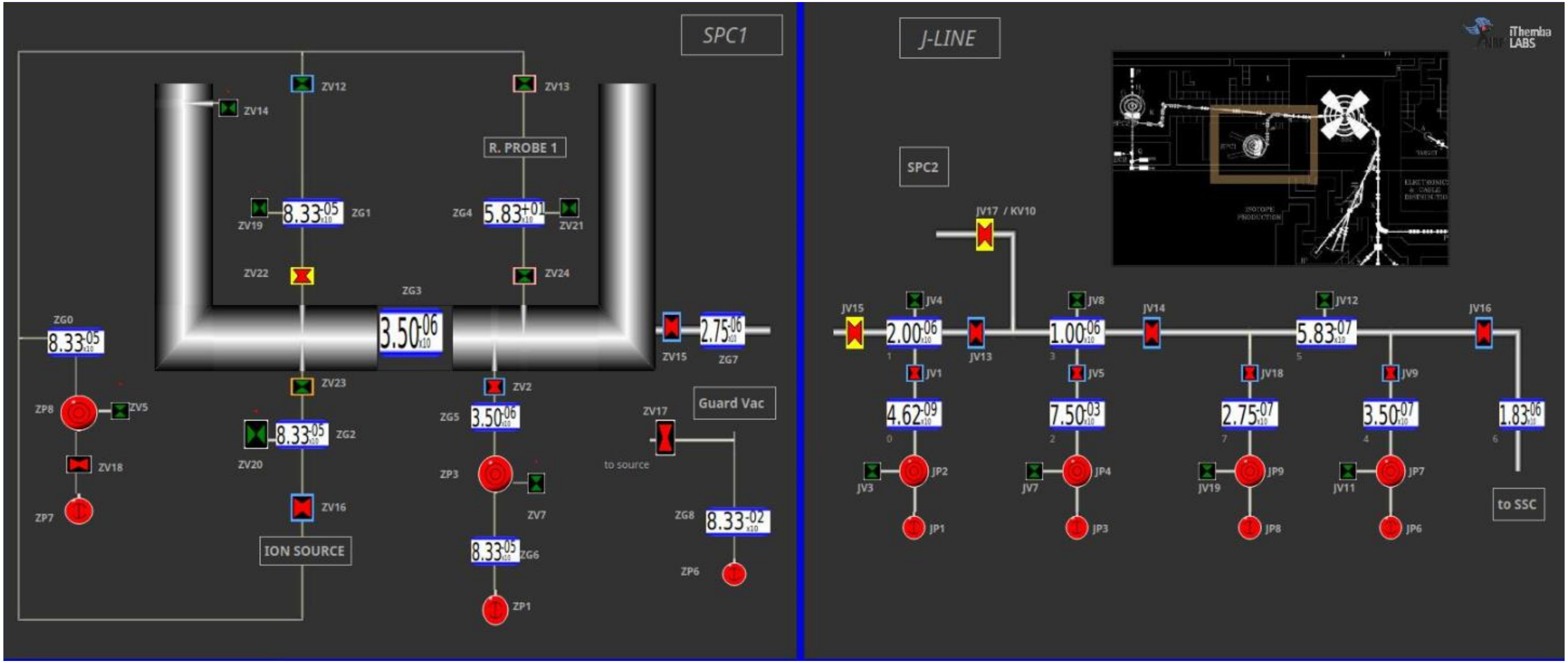
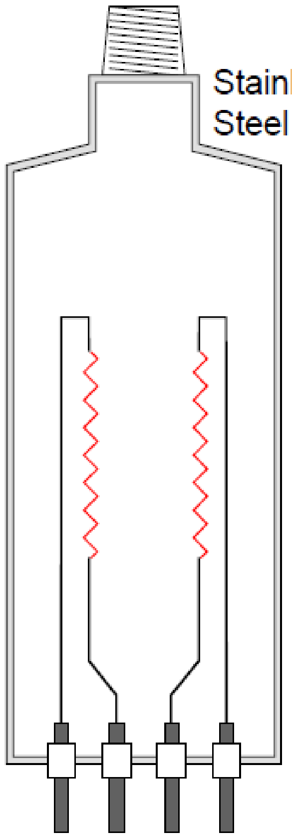
K-LYN
EPS2
G0
G1
G2
G3
G4
G5
G6
G7
G8
G9
G10
P0
P4
P5
P6
P7
P9

Vacuum display of cyclotron and beam line at iThemba LABS



Pirani Gauges - 1



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Pirani Gauges - 2

- Similar to a TC gauge, an electrically heated filament takes on a temperature that depends upon the rate of heat loss to the surrounding gas.
- The temperature of the filament is sensed by measuring the change in the resistance of the filament as it is heated.
  - For most metals, the TCR is about +200 ppm/°C.
- Pirani gauges require a more sophisticated controller, but are more accurate and faster responding than a TC gauge.
- Most use a Wheatstone bridge circuit to linearize the filament against a compensating filament that is held at atmospheric pressure.
- Pirani gauges are also sensitive to the gas composition.

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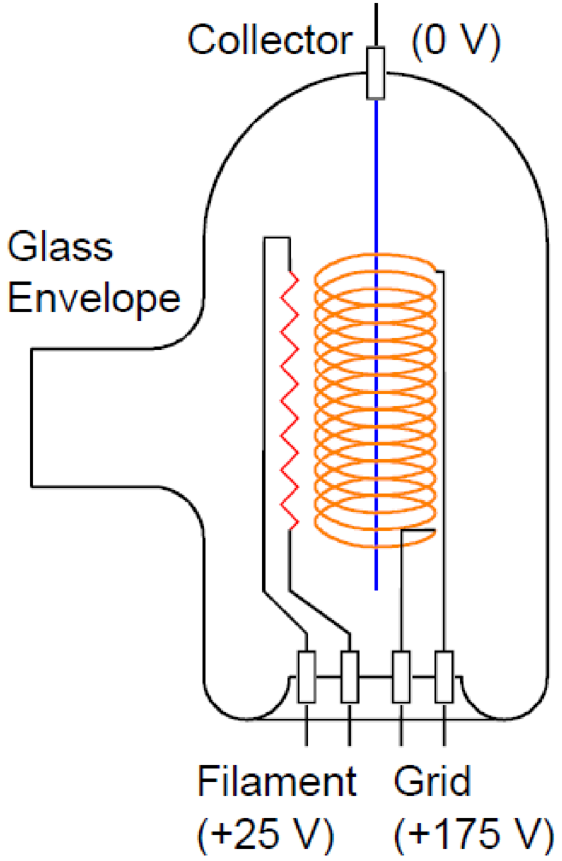
Hot Filament Ionization Gauges - 1



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Hot Filament Ionization Gauges - 2

- Also know as "Bayerd-Alpert" gauges.
- Electrons are thermionically emitted from a hot filament and then accelerated by a grid electrode.
- The accelerated electrons will ionize any gas molecules in the vicinity of the grid, and the positively charged gas ion will contribute to a current through the collector electrode.
- $I_P = I_E \cdot S \cdot P$, where
  - I_P = positive ion current through collector electrode
  - I_E = electron emission current through filament
  - S = gauge sensitivity parameter
  - P = gas pressure

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Hot Filament Ionization Gauges - 3

- The ionization rate depends upon the gas species, so ion gauges are sensitive to the gas composition.
- Accuracy is about 10% of full scale, when calibrated.
- Ion gauges can work from 10^{-3} to 10^{-11} torr!
- The lower pressure limit is set by soft x-ray emission from electrons striking the grid.
- The hot filament requires some precautions:
  - Exposure to pressures above 10^{-3} torr will burn out the filament.
  - Most ion gauge controllers automatically turn off the filament when the collector current exceeds a maximum value.
  - The hot filament is an ignition source which can trigger explosions in the process chamber with combustible gases.

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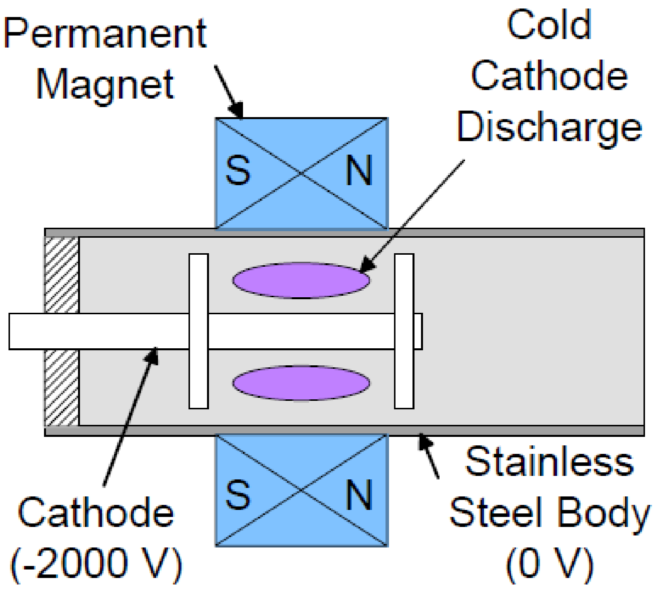
Cold cathode penning and pirani gauge head and in-house built pirani penning vacuum gauge

Cold Cathode Ionization Gauges - 1



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Cold Cathode Ionization Gauges - 2

- A cold cathode "Penning" discharge replaces the hot filament for producing ionizing electrons.
- Electrons orbit around the center post with long trajectories which are very effective in ionizing gas molecules.
- The ionized gas molecules are collected by the negatively charged cathode, and the electric current is proportional to the gas pressure. Typically produce 1-5 Amps/torr.
- These can operate from 10^{-2} to 10^{-8} torr.
- More rugged than a hot filament ion gauge, but less accurate, typically only about 50% of full scale.
- Cold cathode discharge is still a potential source of ignition for combustible process gases.

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Leak detectors

The gas from any potential leaks is collected and pumped into the leak detector’s mass spectrometer for analysing, and any value above the background trace of helium is evidence of a leak. The spectrometer itself works in the following way: any helium molecules sucked into the spectrometer will be ionized, and these helium ions will then bend into the ion trap where the ion current is analysed and recorded. Based on the ionization current the leak rate is then calculated

Residual gas analyzers

The **residual gas analyzer** ionizes the different components of the **gas** to create various ions, and then detects and determines the mass-to-charge ratios with a mass analyzer. The residual gas analyser is very helpful in determining the nature of the leak.

Acknowledgement to Dr Bruce Darling

I use a number of slides from Prof Bruce Darling lecture Vacuum Systems from the Washington University Electrical department. He gave me full permission to use the slides

Thank you