



the  
**abdus salam**  
international centre for theoretical physics

*ICTP 40th Anniversary*

*SCHOOL ON SYNCHROTRON RADIATION AND APPLICATIONS*  
*In memory of J.C. Fuggle & L. Fonda*

**19 April - 21 May 2004**

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**1561/1**

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**Vacuum Technology (part 1 - 2 - 3 - 4 - 5)**

**Oswald Gröbner**

School on Synchrotron Radiation

**UHV - Technology**

Oswald Gröbner

Trieste, 20-21 April 2004

- 1) Introduction and some basics
- 2) Building blocks of a vacuum system
- 3) How to get clean ultra high vacuum
- 4) Desorption phenomena
- 5) Practical examples

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# Literature

## Books

The Physical Basis of Ultrahigh Vacuum, P.A. Redhead, J.P. Hobson, E.V. Kornelsen, American Vacuum Society Classics, American Institute of Physics, 1993

Foundations of Vacuum Science and Technology, Ed. J.M. Lafferty, John Wiley & Sons, 1998

Handbook of Accelerator Physics and Engineering, A. W. Chao, M. Tigner, World Scientific, 1998

CAS CERN Accelerator School : Vacuum Technology, Ed. : S. Turner. CERN 99-05, 19 August 1999

Handbuch Vakuumtechnik, M. Wutz et. al, Vieweg, Braunschweig/Wiesbaden, 2000

## Journals:

VACUUM

Journal of Vacuum Science and Technology (A)

Nuclear Instruments and Methods (Section A)

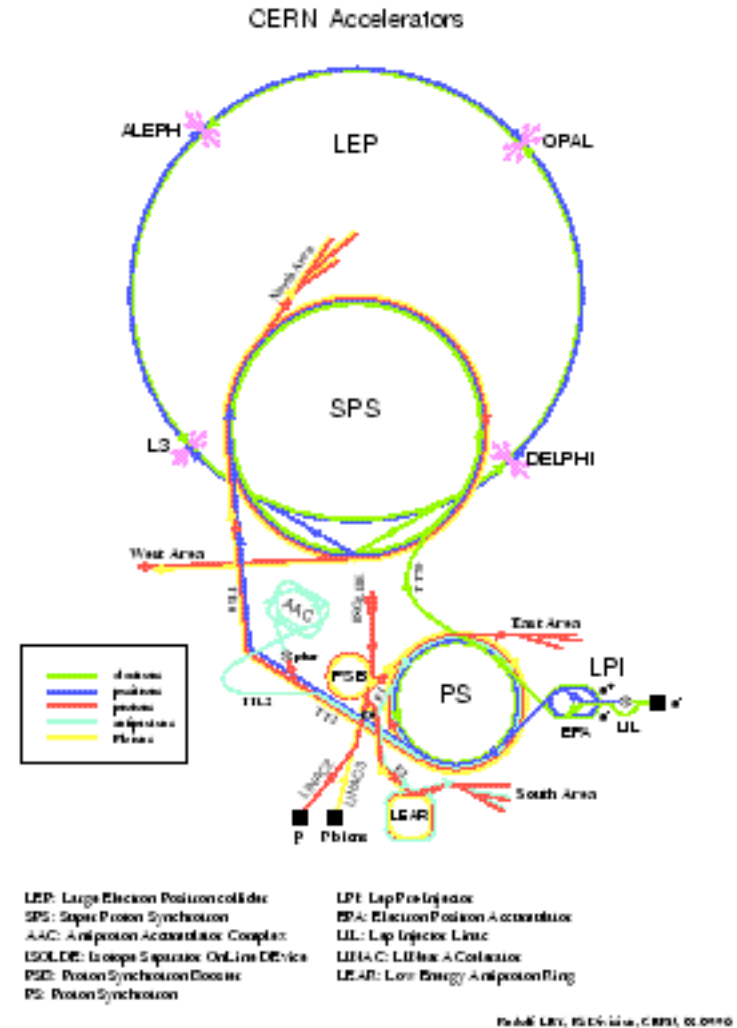
## Accelerators at CERN

Historical map from 1996

Nearly all CERN accelerators require ultrahigh vacuum

Total length of vacuum systems over 60 km

LEP to be replaced by LHC (cryogenic vacuum system)





## Pressure and Molecular Density

**Ideal gas law:**  $P V = \frac{N}{N_0} R T$

$P$  pressure,  $V$  volume,  $T$  temperature

$N$  number of molecules

$R$  gas constant = 8.31 kJ kmol<sup>-1</sup> K<sup>-1</sup>,

$N_0 = 6.02 \cdot 10^{26}$  molecules kmol<sup>-1</sup>

**Molecular density**  $n = N/V$

**Pressure :**  $P = n k T$

Boltzmann constant  $k = 1.38 \cdot 10^{-23}$  J/K

Note :  $R = N_0 k$

Note: In nearly all cases, it is the **gas density** rather than the **pressure** which matters.

Units :

Pressure : Pa (N/m<sup>2</sup>), mbar = 100 Pa, Torr = 133 Pa

Gas load : Pa m<sup>3</sup> = 7.5 Torr l, mbar l ~ 2.4 · 10<sup>19</sup> molecules at RT

Specific outgassing rate : Gas release from the walls

Pa m<sup>3</sup>/s/m<sup>2</sup> ~ 7.5 · 10<sup>-4</sup> Torr l/s/cm<sup>2</sup>

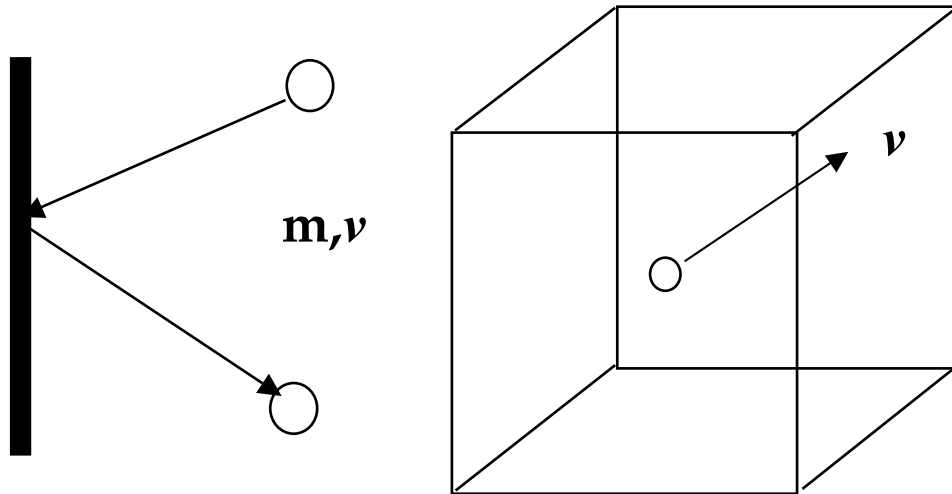
Leak rate : Pa m<sup>3</sup>/s or W, mbar l/s or Torr l/s

## Wall collisions

Frequency of wall collisions

$$\square = \frac{1}{4} n \bar{v}$$

proportional to the number density  $n$  and to the average molecular velocity  $\bar{v}$ .



Momentum transfer to the walls is  $2 m \bar{v}$ , hence the pressure is proportional to  $m \square \bar{v}$

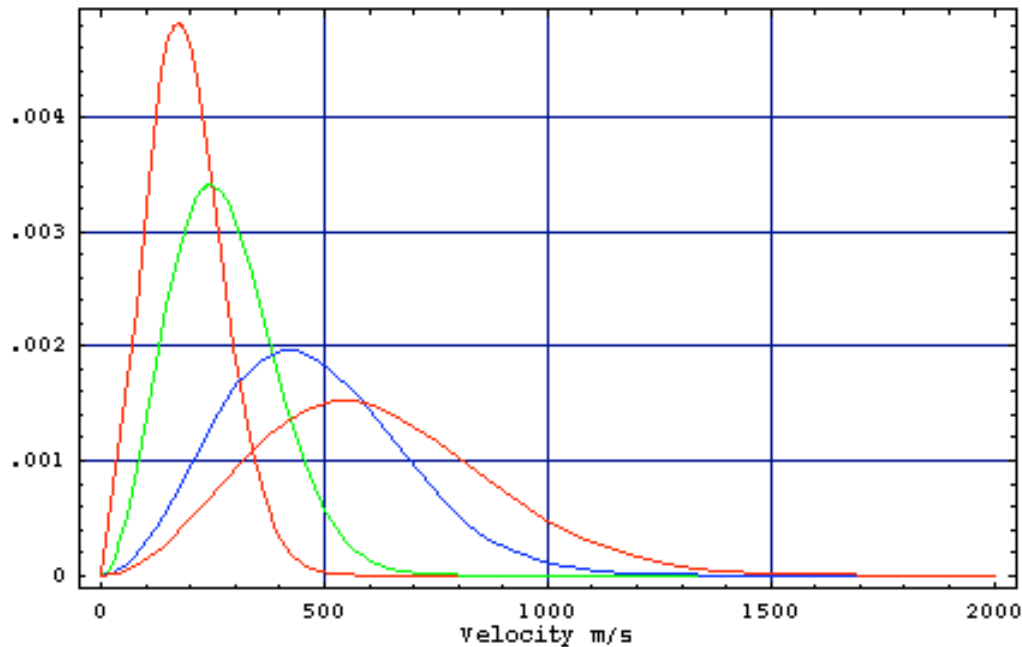
## Distribution of Molecular Velocities

Maxwell-Boltzmann distribution of molecular velocities at the temperature T

$$\frac{1}{N} \frac{dN}{dv} = \frac{4}{\sqrt{\pi}} \left( \frac{m}{2kT} \right)^{3/2} v^2 e^{-\frac{mv^2}{2kT}}$$

The average velocity is given by ( $m = M m_o$ ):  $\bar{v} = \sqrt{\frac{8kT}{\pi M m_o}}$ , numerically  $\sim 146 \sqrt{\frac{T}{M}}$  ( $\text{m s}^{-1}$ )

Molecular velocities for  $\text{N}_2$  at 50, 100, 300 and 500K.



Mean molecular velocities at 20°C (m/s)

| $\text{H}_2$ | $\text{N}_2$ | Air | A   | Kr  |
|--------------|--------------|-----|-----|-----|
| 1754         | 470          | 464 | 393 | 272 |

## Pressure

$$p = \text{const } m \bar{v} \square$$

$m$  mass of a molecule  
 $\bar{v}$  average molecular velocity  
 $\square$  // rate of wall collisions

A complete calculation gives  $\text{const} = \square/2$

Thus one finds

$$p = \square/2 m \sqrt{\frac{8kT}{\square m}} n \sqrt{\frac{kT}{2\square m}}$$

and the final result

$$p = n kT$$

Considering a molar quantity,  $n = \frac{N_o}{V_o}$

One finds again the familiar result  
 with the gas constant  $R$

$$pV_o = N_o kT = RT$$

## Mean Kinetic Energy

The kinetic energy : 
$$E_{kin} = \frac{1}{2} m \bar{v}^2 = \frac{1}{2} M m_o \frac{8kT}{M m_o} = \frac{4}{2} kT$$

$M$  molecular weight,  $m_o = 1.66 \cdot 10^{-27}$  kg

does not depend on the molecular mass,  $M$ , but only on temperature  $T$ .

In **thermal equilibrium** heavy molecules move sufficiently slowly and light molecules move sufficiently fast to carry on average the same kinetic energy.

## Total and Partial Pressures

For each gas component  $n_1, n_2, n_3, \dots$  the contribution to the total pressure :  $P_i = n_i kT$

The total pressure is therefore the sum of the partial pressures: 
$$P = \sum_i P_i = kT \sum_i n_i$$

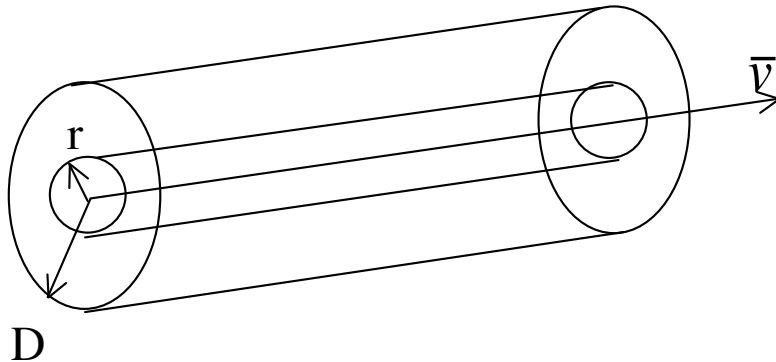
Partial pressures for atmospheric air

| Gas             | %                      | Pi (Pa)                |
|-----------------|------------------------|------------------------|
| N <sub>2</sub>  | 78.1                   | 7.9 · 10 <sup>4</sup>  |
| O <sub>2</sub>  | 20.5                   | 2.8 · 10 <sup>3</sup>  |
| Ar              | 0.93                   | 1.2 · 10 <sup>2</sup>  |
| CO <sub>2</sub> | 0.0033                 | 4.4                    |
| Ne              | 1.8 · 10 <sup>-3</sup> | 2.4 · 10 <sup>-1</sup> |
| He              | 5.2 · 10 <sup>-4</sup> | 7 · 10 <sup>-2</sup>   |

## Mean Free Path

$$l = \frac{l}{\sqrt{2} \pi D^2 n}$$

D molecular diameter ( $\sim 3 \cdot 10^{-8}$  m)



Volume traversed by a molecule per second :  $\pi D^2 \bar{v}$

Molecule collides with all other molecules contained within the cylinder of radius  $D$ .

Number of collisions:  $Z \pi \pi D^2 \bar{v} n$ .      The mean free path  $l = \frac{\bar{v}}{Z} = \frac{l}{\sqrt{2} \pi D^2 n}$

It also follows that  $n l \quad P l \pi \text{const.}$

Note:  $\sqrt{2}$  accounts for the fact that molecules in the cylinder are not stationary.

For air the product  $n l \pi \text{const}$  is  $\sim 2.5 \cdot 10^{14} \text{ m}^{-2}$ .

For  $\text{N}_2$  at 20 deg. C and at a pressure of 1 Pa  $\rightarrow l \sim 9$  mm

## Molecular Flow Conditions

**Knudsen relation:** gas flow  $Q \propto P$  applies if the mean free path  $\gg$  relevant dimensions of system

$$\text{Molecular flow conductance } c = \frac{4}{3} \frac{\bar{v}}{L} \frac{H}{A^2} dl \quad (\text{m}^3/\text{s})$$

L length of the element (L  $\gg$  transverse dimensions).

H perimeter, A cross section of the element.

The conductance is proportional to the mean molecular velocity, i.e. to  $\sqrt{\frac{T}{M}}$ .

A cylindrical duct with uniform section and radius  $r$ :

$$c = \frac{4}{3} \bar{v} \frac{r^3}{L} \sim 306 \cdot \frac{r^3}{L} \sqrt{\frac{T}{M}}$$

An orifice (pumping orifice,  $L \sim 0$ ):

$$c = \frac{1}{4} \bar{v} A \sim 36.5 \cdot A \sqrt{\frac{T}{M}}$$

Conductance of elements in series or in parallel add the same as for electric circuits

$$\text{Series: } \frac{1}{c} = \frac{1}{c_1} + \frac{1}{c_2} \quad \text{and parallel: } c = c_1 + c_2$$

For complicated geometries it is often necessary to use Monte Carlo calculations for the molecular flow.

## Molecular conductance of circular tubes

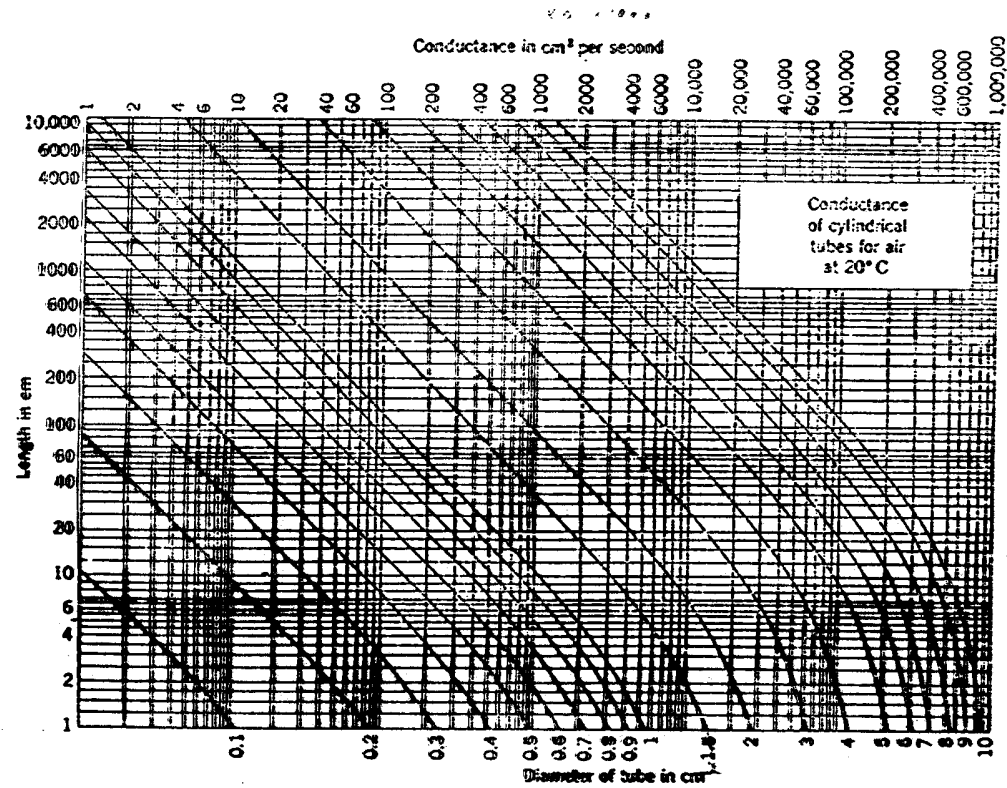


Fig. 2.1. Plots of conductance of cylindrical tubes for air at 20° C calculated according to equation 2.40.



## Vacuum characteristics

gas : Nitrogen, N<sub>2</sub>, 20°C, M = 28

Summary expressions:

$$n = \frac{P}{kT}$$

$$kT = 4.04 \cdot 10^{-21} \text{ Joule}$$

$$\rho = M m_O n$$

$$M m_O = 4.65 \cdot 10^{-26} \text{ kg}$$

$$m_O = 1.66 \cdot 10^{-27} \text{ kg}$$

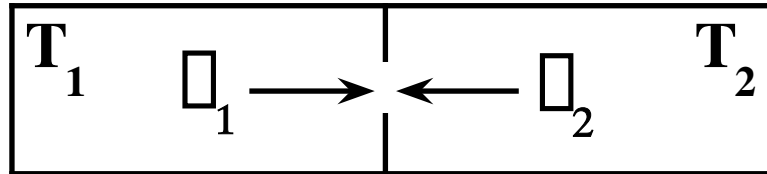
$$\lambda = \frac{l}{4} n \bar{v} \quad \text{and} \quad \bar{v} = 146 \sqrt{\frac{T}{M}}$$

$$l = \frac{l}{\sqrt{2} \lambda D^2 n}$$

$$D(\text{N}_2) = 3.15 \cdot 10^{-10} \text{ m}$$

| Pressure          | P<br>Pa                              | n<br>m <sup>-3</sup>                             | $\rho$<br>kg m <sup>-3</sup>                        | $\lambda$<br>m <sup>2</sup> s <sup>-1</sup>      | l<br>m                                       |
|-------------------|--------------------------------------|--|---|--|--|
| atm               | 10 <sup>5</sup>                      | 2.5 · 10 <sup>25</sup>                           | 1.16  | 2.9 · 10 <sup>27</sup>                           | 9 · 10 <sup>-8</sup>                         |
| primary<br>vacuum | 1<br>10 <sup>-1</sup>                | 2.5 · 10 <sup>20</sup><br>2.5 · 10 <sup>19</sup> | 1.16 · 10 <sup>-5</sup><br>1.16 · 10 <sup>-6</sup>  | 2.9 · 10 <sup>22</sup><br>2.9 · 10 <sup>21</sup> | 9 · 10 <sup>-3</sup><br>9 · 10 <sup>-2</sup> |
| high<br>vacuum    | 10 <sup>-4</sup><br>10 <sup>-7</sup> | 2.5 · 10 <sup>16</sup><br>2.5 · 10 <sup>13</sup> | 1.16 · 10 <sup>-9</sup><br>1.16 · 10 <sup>-12</sup> | 2.9 · 10 <sup>18</sup><br>2.9 · 10 <sup>15</sup> | 9 · 10 <sup>1</sup><br>9 · 10 <sup>4</sup>   |
| uhv               | 10 <sup>-10</sup>                    | 2.5 · 10 <sup>10</sup>                           | 1.16 · 10 <sup>-15</sup>                            | 2.9 · 10 <sup>12</sup>                           | 9 · 10 <sup>7</sup>                          |
| xhv               | <10 <sup>-11</sup>                   |  |   |  |  |

# Thermal transpiration



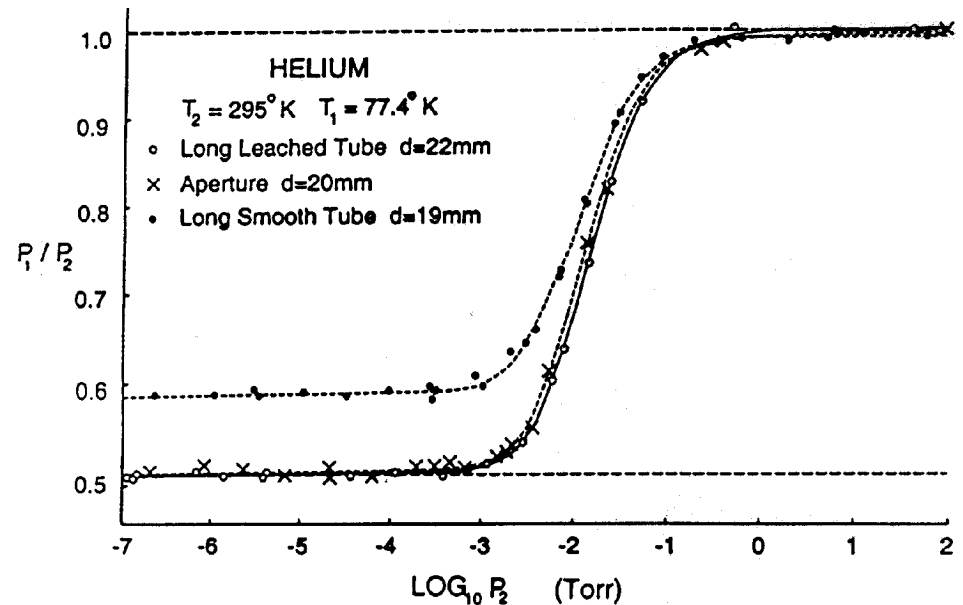
At high pressure, the pressures on both sides are equal.

In molecular flow, the net number of molecules traversing the separating wall must be zero.

$$\varrho_1 = \varrho_2$$

Since  $\varrho = \frac{1}{4} \bar{v} n$  and  $\bar{v} = \sqrt{\frac{8kT}{\pi m}}$

$$n_1 \sqrt{T_1} = n_2 \sqrt{T_2} \quad \text{or} \quad \frac{P_1}{\sqrt{T_1}} = \frac{P_2}{\sqrt{T_2}}$$

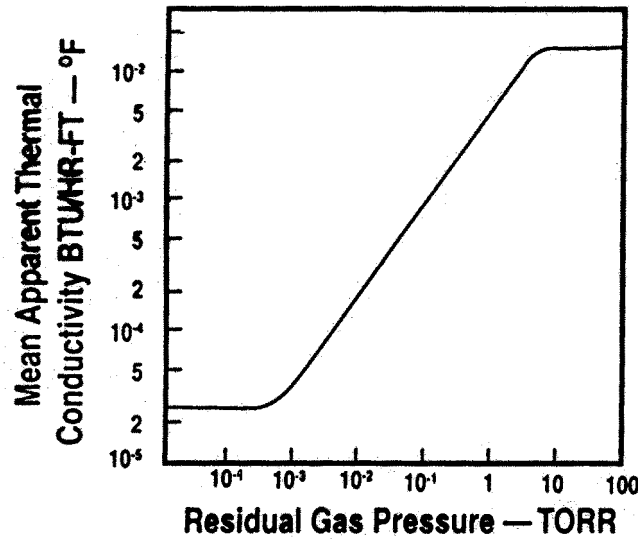


## Thermal Conductivity

Thermal conductivity of a gas is independent of the pressure when the pressure is well above the molecular flow regime.

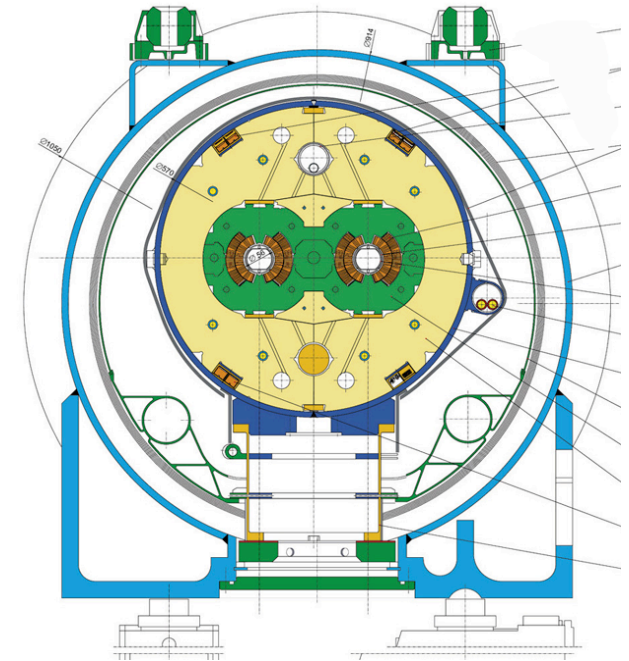
In the transition regime, the heat transfer is proportional to the pressure and to the temperature difference. Principle of pressure measurement with a Pirani gauge.

$$10^{-3} \text{ Torr} < P < 10 \text{ Torr}$$



At very low pressures, the heat transfer by conduction is negligible : vacuum for thermal insulation in cryogenics.

is



LHC Cryodipole

## Basic Vacuum System

V volume (m<sup>3</sup>), F surface (m<sup>2</sup>)

P pressure (Pa), S pumping speed (m<sup>3</sup>/s)

q specific outgassing rate (Pa m<sup>3</sup>/s/ m<sup>2</sup>)

Stationary conditions (P is independent of volume)  $P = \frac{q F}{S}$

Dynamic pressure  $V \frac{\partial P}{\partial t} = q F - S P$

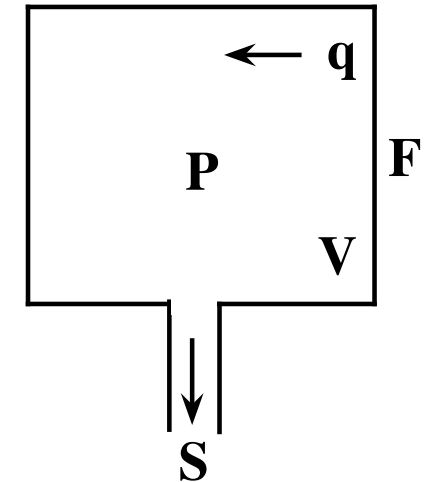
Solution (constant K depends on initial conditions)  $P(t) = K e^{-\frac{S}{V}t} + \frac{q F}{S}$

The time constant of the pump down:  $\frac{V}{S}$

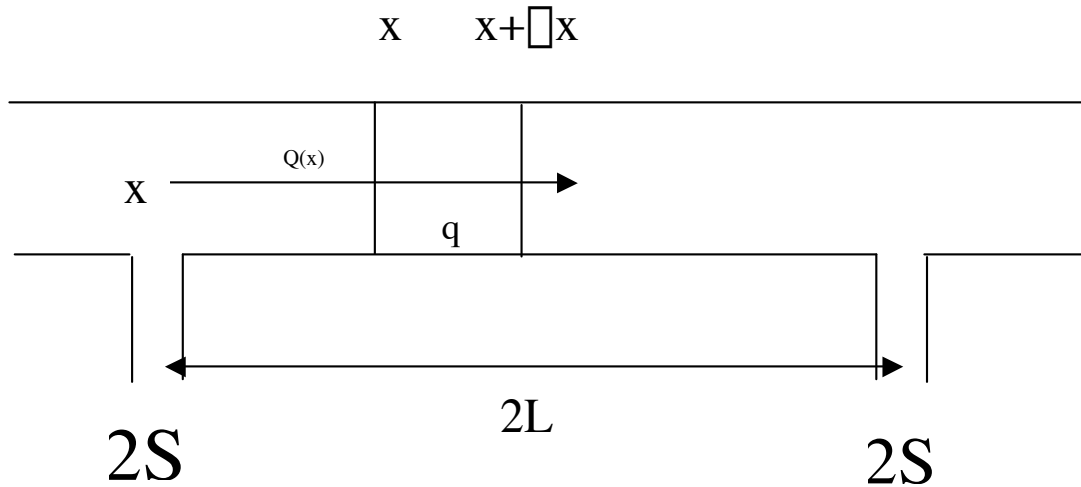
To obtain a low pressure :

Low outgassing rate of the surface, **No leaks**□

Large pumping speed



## Linear Vacuum System (Accelerators)



Gas flow:  $Q(x)$  [ $\text{Pa m}^3 \text{s}^{-1}$ ], specific outgassing rate:  $q(x)$  [ $\text{Pa m s}^{-1}$ ]

Specific surface area per unit length:  $A$  [ $\text{m}$ ], molecular conductance per unit length:  $c$  [ $\text{m}^4 \text{s}^{-1}$ ]

$$\frac{dQ}{dx} = Aq \quad \text{and} \quad Q(x) = c \frac{dP}{dx}$$

$$c \frac{d^2 P}{dx^2} = Aq$$

Boundary conditions for this configuration:

By symmetry  $\left. \frac{dP}{dx} \right|_{x=L} = 0$  and the pressure at  $x = 0$

$$P(x=0) = \frac{Q(x=0)}{2S}$$

$$Q(0) = 2 A q L$$

One obtains a parabolic pressure distribution:

$$P(x) = A q \left[ \frac{2Lx - x^2}{2c} + \frac{L}{S} \right]$$

The average pressure, relevant for the beam:

$$P_{av} = \frac{1}{2L} \int_0^{2L} P(x) dx = A q \left[ \frac{L^2}{3c} + \frac{L}{S} \right]$$

Note: the pressure is limited by the molecular conductance of the system.

In spite of an increase of the pumping speed  $S$ , the average pressure is limited to the value

$$P_{av \min} = \frac{A q L^2}{3c}$$

Two important requirements : Large diameter of the vacuum chamber  
Close spacing of pumps

Time to form one monolayer

$$t = \frac{\Gamma}{\frac{1}{4} \bar{v} s n}$$

Mono layer coverage:

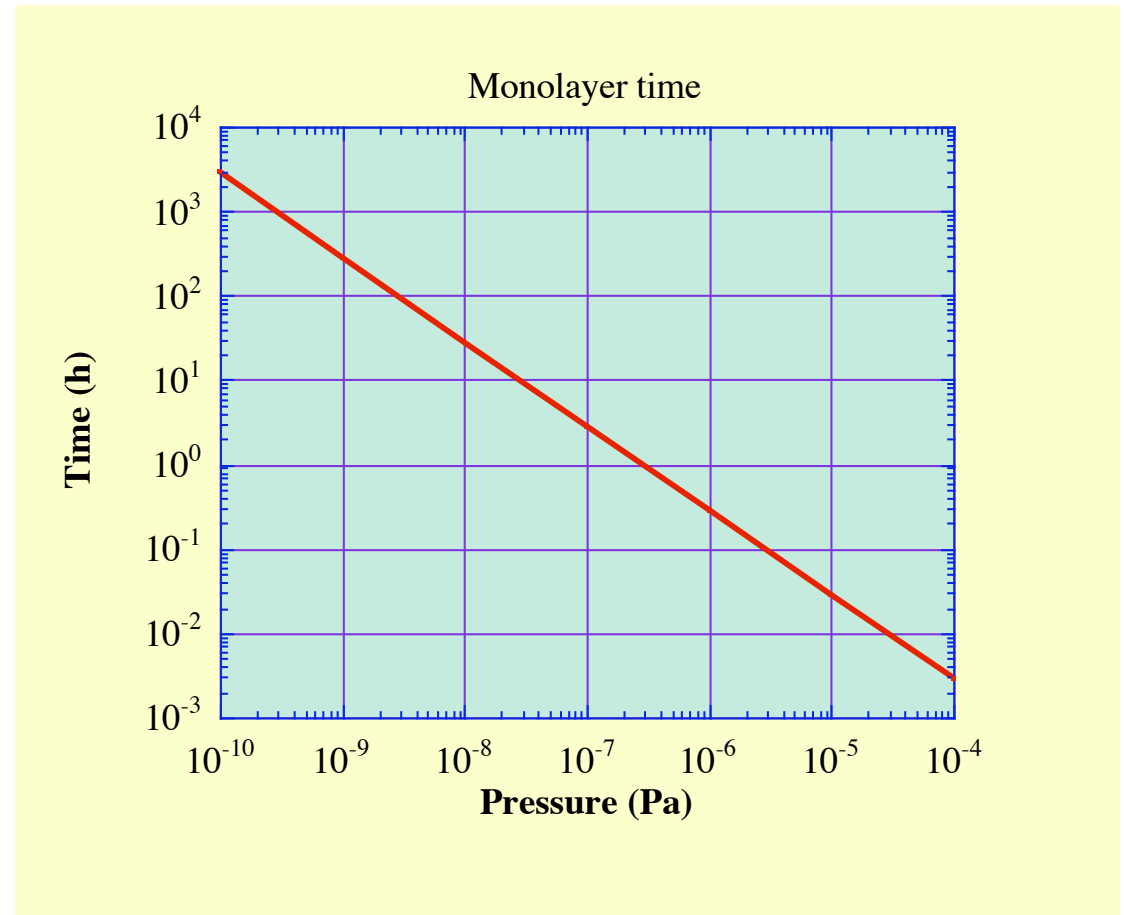
$\Gamma$  ( $\sim 3 \cdot 10^{19}$  molecules  $\text{m}^{-2}$ )

Molecular velocity  $\bar{v}$  ( $\text{m s}^{-1}$ )

Gas density  $n$  (molecules  $\text{m}^{-3}$ )

Sticking probability  $s < 1$

UHV becomes indispensable for surface analysis and for thin film technology  
 -> Historically the main motivation to develop uhv techniques



Note: Area occupied per molecule  $A \sim 2\sqrt{3}r^2$

## Beam Lifetime due to Vacuum

Beam loss by Bremsstrahlung :  $\frac{dE}{dx} = \frac{E}{X_0}$

$$\text{Lifetime } \frac{1}{\tau} = \frac{1}{N} \frac{dN}{dt} = \frac{c \rho}{X_0} W$$

$X_0$  radiation length,  $c$  speed of particles and  $\rho = \frac{m_0 M}{kT} P$  is the density of the residual gas at the pressure  $P$ .

$$\text{Here } W = \log\left(\frac{E}{\Delta E}\right)$$

Represents the probability per radiation length to emit a photon with an energy larger than the energy acceptance of the machine so that the particle will be lost.

The lifetime  $\tau = \frac{X_0}{c \rho W} = \frac{X_0}{P}$

For nitrogen or CO one finds typically  $\tau P = 3.410 \times 10^8$  (Torr hours)

Consequence : UHV is required for storage rings.

Heavy molecules with short radiation length must be avoided.





## Rotary Pumps

Single stage and double stage pumps

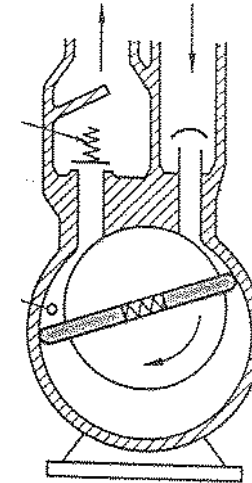
Oil sealed

Typical end pressures :  $10^{-2}$  to  $\sim 10^{-3}$  mbar

Typical pumping speeds : 4 to  $\sim 40$  m<sup>3</sup>/h  
(used for systems with small volume)

Filter for oil vapour is required.

**Dry pumps**, without oil are now available but very expensive!



## Turbomolecular Pump

Molecules collide with the surface and gain a velocity component in the direction of the movement.

Pumping speed of a turbomolecular pump  $S = v A$

$S$  independent of pressure

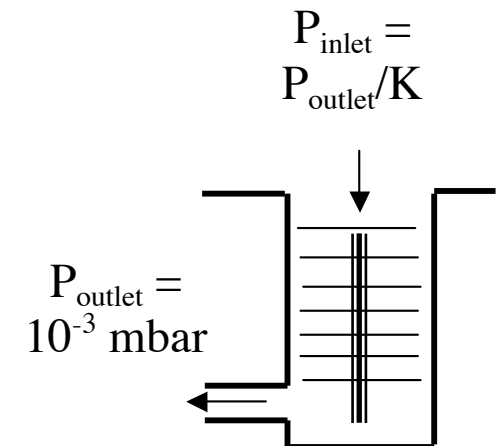
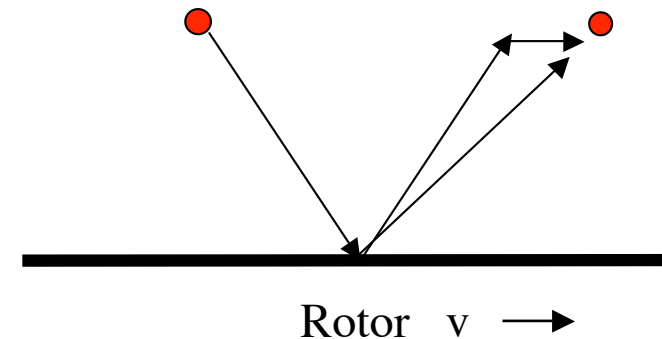
$v$  rotational speed, typically at least >40000 rpm

$A$ : pump geometry, large entrance flange

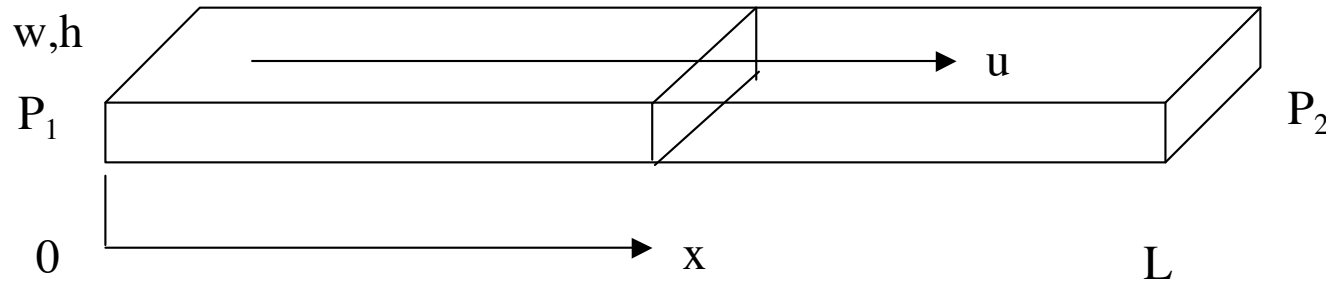
Compression ratio of the pump is defined as  $K = \frac{P_{outlet}}{P_{inlet}}$

$K$  is an exponential function of the molecular weight and of the rotational speed ( $10^3$  for  $H_2$  to  $10^9$  for  $N_2$ )

Hence the compression ratio is large for heavy molecules -> 'clean vacuum' without heavy hydrocarbon molecules. Oil contamination from primary pump can be avoided.



## The Molecular Pump Principle



The molecular conductance of a rectangular duct, with the perimeter  $H = 2(w + h)$  and the section  $A = w \cdot h$  is given by  $\square$

$$c = \frac{4}{3} \frac{\bar{v}}{x} \frac{H}{A^2} dl = \frac{2}{3} \bar{v} \frac{h^2 w^2}{x(h+w)}.$$

In molecular flow  $\square$   $\frac{dP}{dx} = \frac{Q}{c}$ .

Assuming that one surface is moving with the speed  $u$ , the gas flow which traverses a given cross section corresponds to  $\square$   $Q = PV = P \frac{u}{2} A$ .

Thus  $\frac{dP}{dx} = \frac{u}{2c} AP$ .

Integrating this equation over the length of the duct

$$\int_{P_1}^{P_2} \frac{dP}{P} = \frac{u}{2c} A \int_0^L dx$$

one obtains the result

$$K = \frac{P_2}{P_1} = e^{\frac{u}{2c} AL}.$$

K defines the compression ratio of a pump i.e. the ratio between input and output pressures.

K increases exponentially with the molecular weight, and with the rotational speed of the pump.

Typical values of commercial pumps :      for hydrogen  $\sim 2 \cdot 10^3$

for nitrogen  $\sim 10^8$  !

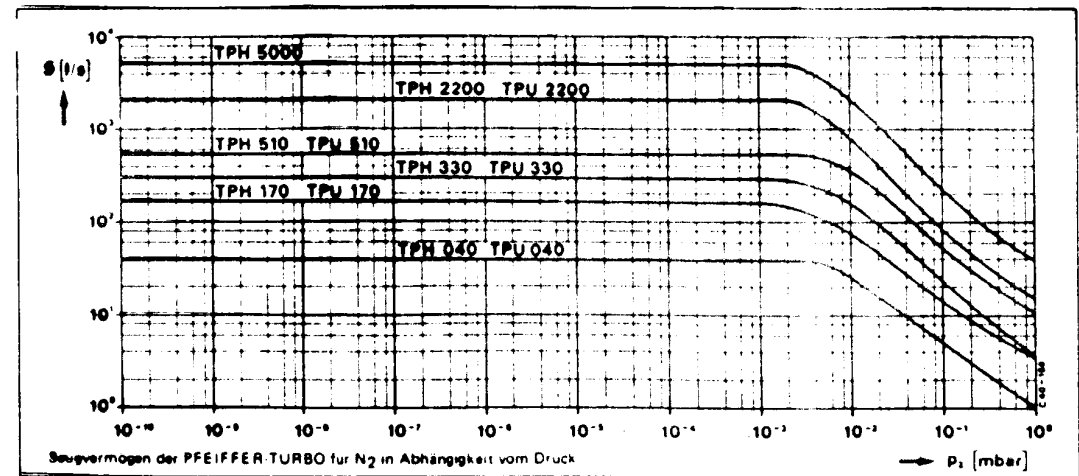
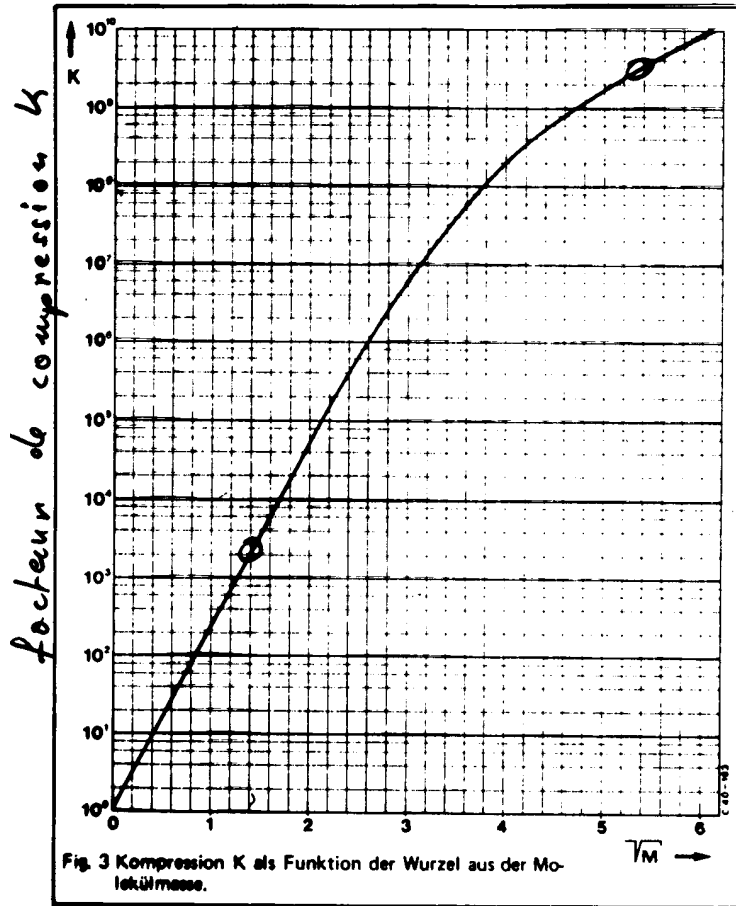
A large compression ratio for heavy molecules results in a 'clean vacuum' without hydrocarbon (C-H) contamination.

The pumping speed S is proportional to the rotational speed and depends on the geometry of the pump.

S does not depend on the molecular weight M.

## Turbomolecular pumps

Compression factor and pumping speed curves of commercial turbomolecular pumps

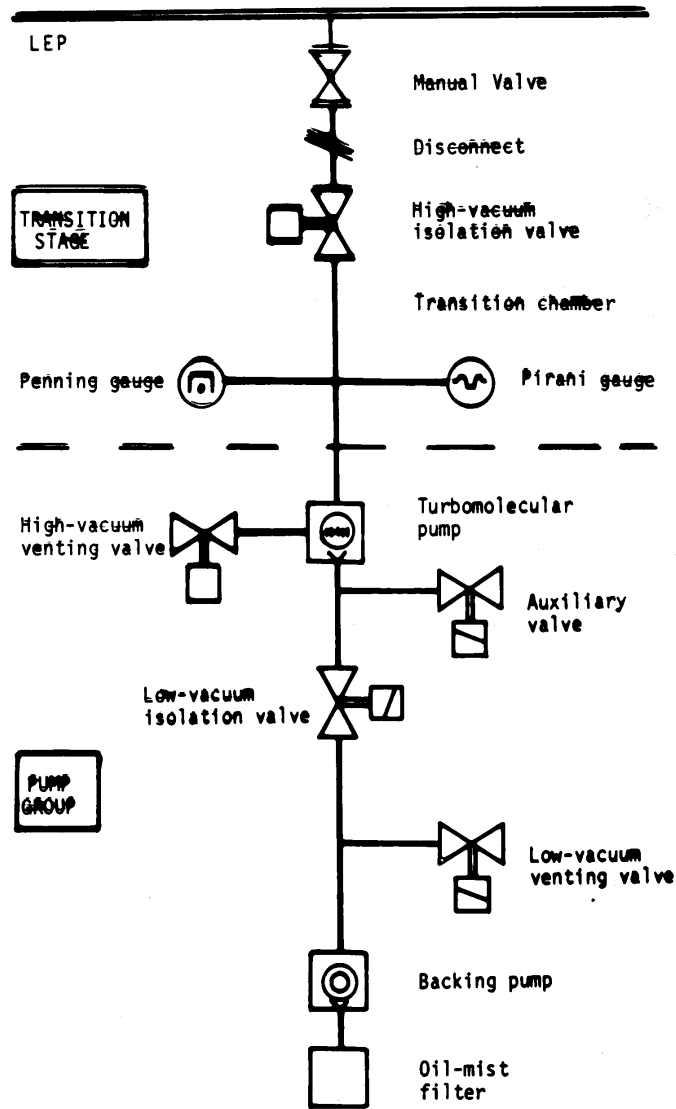


## Pumping station for LEP

Schematic layout:

Pumps required for the initial pump down only.

During operation of the accelerator, the separation valve is closed. The mobile pumps can be removed from the tunnel.



## Sputter-Ion-Pump

Configuration of a parallel electric and magnetic field produces a self-maintained discharge plasma.

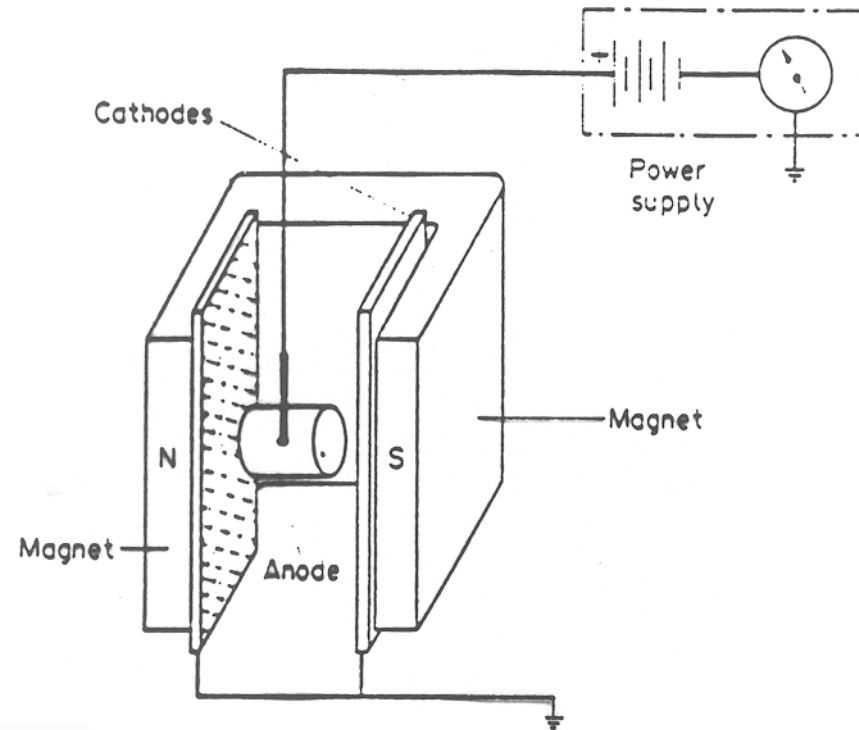
-> Penning configuration

Ionised residual gas molecules are accelerated towards the Ti cathode and 'trapped' and removed from the gas phase.

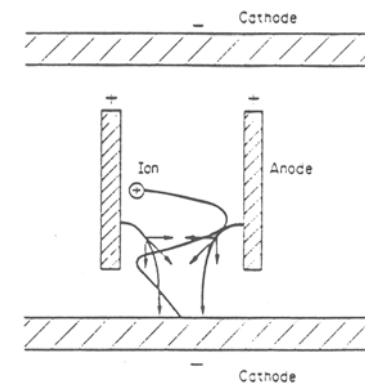
Sputtering of Ti from cathode produces a clean gettering film.

In a particle accelerator, the magnetic field is provided by bending magnets. --> integrated, linear ion-pumps.

To increase the pumping speed, arrays of cells are used



Basic configuration of a sputter-ion pump.





## Pumping mechanisms

**Gettering** -> chemisorption of active species  $H_2$ ,  $CO$ ,  $N_2$ ,  $O_2$ ,  $CO_2$

**Diffusion** of  $H_2$  into the Ti- cathode (re-diffusion!)

**Cracking** of inert hydrocarbons into C, H, O which can be pumped (chemisorbed) separately

**Nobel gases**: energetic ions of He, Ne, Ar by implantation into the cathode: “ion burial” of energetic ions. -> Argon instability after pumping of air.

To increase the discharge intensity and thus the pumping speed it is desirable to increase the sputtering rate of the titanium cathode

→ Triode Sputter-Ion pump with grazing incidence of ions on a grid cathode

Note:

Molecules are not removed from the vacuum system. Therefore, memory effect of previously pumped gas (Argon).

## Surface Pumping

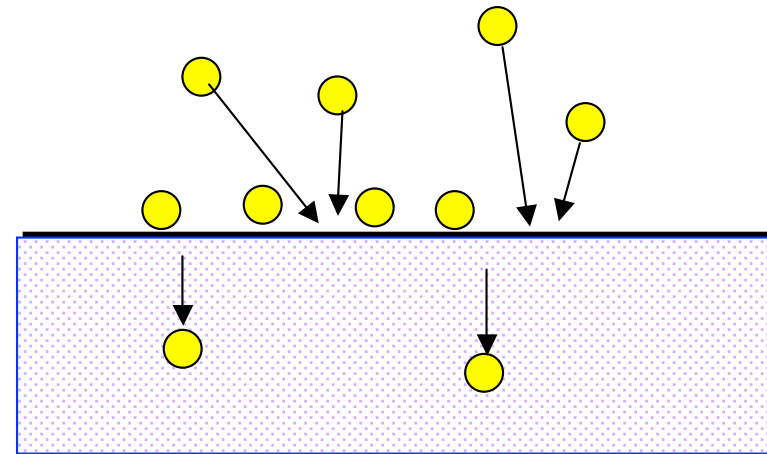
### Getters (chemisorption $E \sim eV$ )

Evaporable getter pumps (Ti sublimators)

Non Evaporable Getters (NEG)

Ti, Zr, V

Surface pumping  $\rightarrow S = \frac{1}{4} \bar{v} n F$



Gettering surface achieved by sublimation from a Ti-filament.

Or by surface activation (heating  $\rightarrow$  reduction of surface oxide layer and diffusion of O into the bulk)

### Cryo-pumps (physisorption $E \sim meV$ )

Sorption (capacity  $\sim$  monolayer)

Condensation (vapour pressure)

## Monolayer Capacity

To illustrate the significance of a monolayer of gas, let us assume an evacuated sphere which has one monolayer molecules adsorbed on the inner surface.

$$N_{ads} = 4\pi r^2 \sigma$$

In case this gas is desorbed it would correspond to a volume density

$$n = \frac{N_{ads}}{V} = \frac{3\sigma}{r}$$

Taking, e.g.  $1 \text{ m}^3$  as the volume and  $p \approx 3 \cdot 10^{19} \text{ m}^{-2}$  the pressure at room temperature would increase to typically

$$p = n kT \approx 0.4 \text{ Pa}$$

Hence:

To reach uhv conditions, the ‘surface gas’ is extremely important.

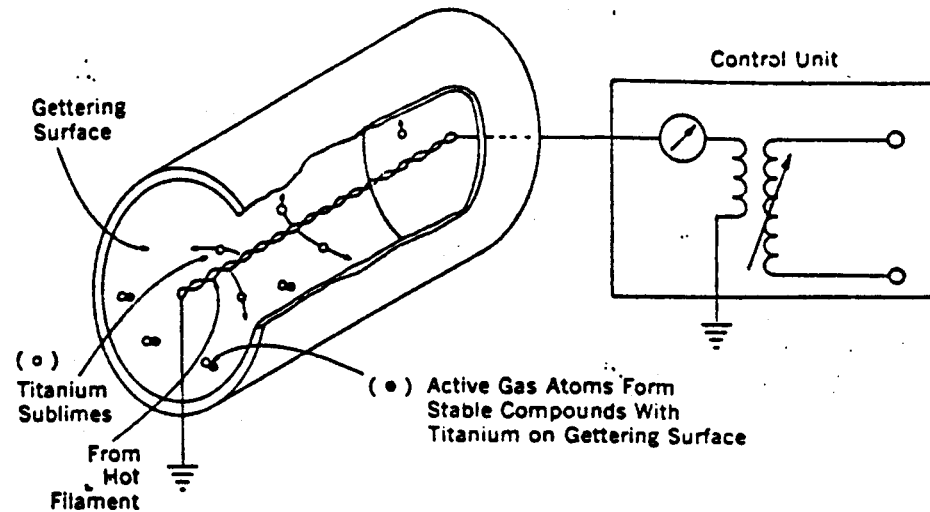
Inversely, adsorbing molecules permanently on an ‘active’ surface (getters or cryo-pumps) is an extremely efficient pumping principle.

## Getter Pumps

### Evaporable getters:

### Titanium sublimation pump

Configuration



Deposition of a thin film of fresh Ti on the inner surface of the vacuum chamber

Filament temperature  $\sim 1300$  deg C

To increase the lifetime of the pump one uses pump holders with several filaments (3 – 6)

Depending on the amount of gas pumped, the film has to be regenerated (e.g. typically at  $10^{-6}$  Pa after one hour)

The pumping speed increases with the surface of the pump and can be very substantial.

Note : only chemically active molecules can be pumped.

## **Non-Evaporable Getters or Bulk getters (NEG) :**

Getter material ( e.g. Ti, Zr, V) produced in the form of an alloy e.g. with Al and used as a bulk material.

For LEP : metal ribbon coated with a thin layer of getter powder has been used.

Clean, active gettering surface is produced by heating under vacuum. Gas adsorbed on the surface diffuses into the bulk and a 'clean' surface can be obtained.

Activation requires heating from 350 °C up to 700°C for one hour depending on the specific getter.

A new development consisting of a combination of evaporable getters and of bulk getters is under development at CERN ->

**sputter deposited getter films** (few  $\mu\text{m}$  only) coated directly onto the inner surface of vacuum chambers. First use in insertion chambers (ESRF) and for LHC vacuum (CERN).

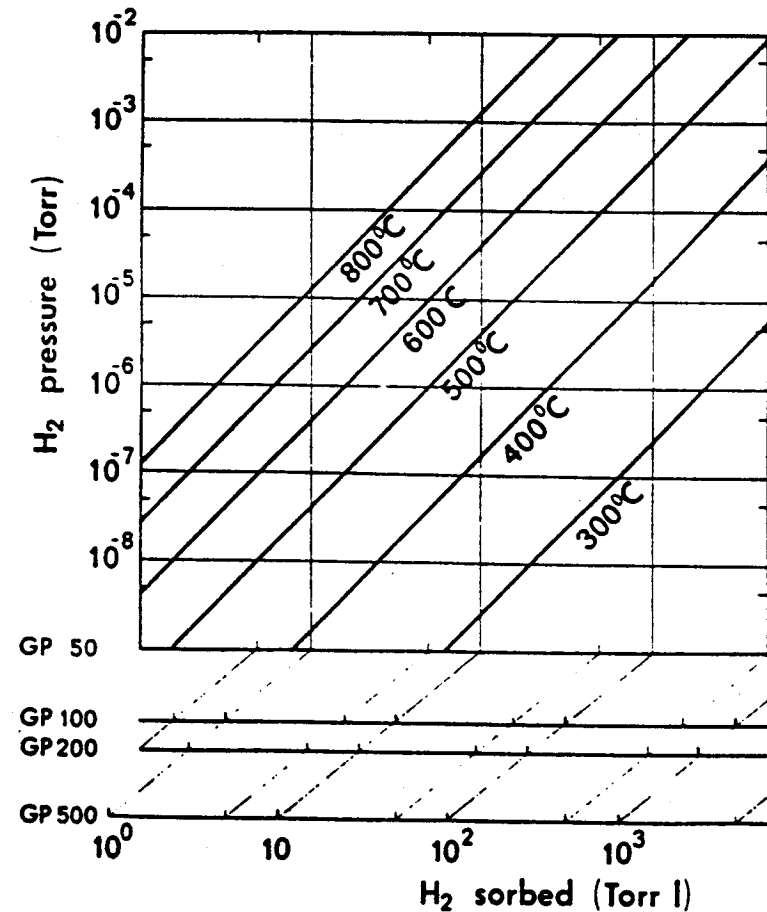
Note: Getters have a limited total pumping capacity and a memory effect of the gas previously pumped.

Getters pump only chemically active gas i.e. noble gases and hydrocarbons (methane, ...) are NOT pumped. Combination with ion pumps is required.

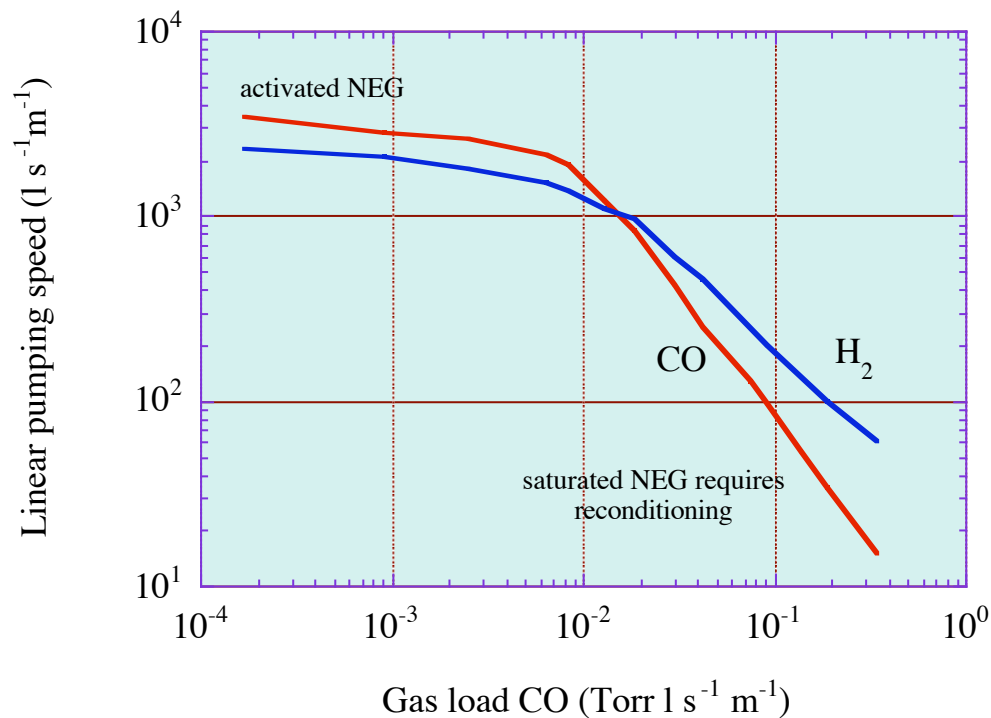
## Reversible pumping of hydrogen by the NEG st101.

The equilibrium pressure of hydrogen is a function of the concentration of H<sub>2</sub> in the bulk,  $q$  (Pa m<sup>3</sup>g<sup>-1</sup>) and of the temperature,  $T$  (K) □

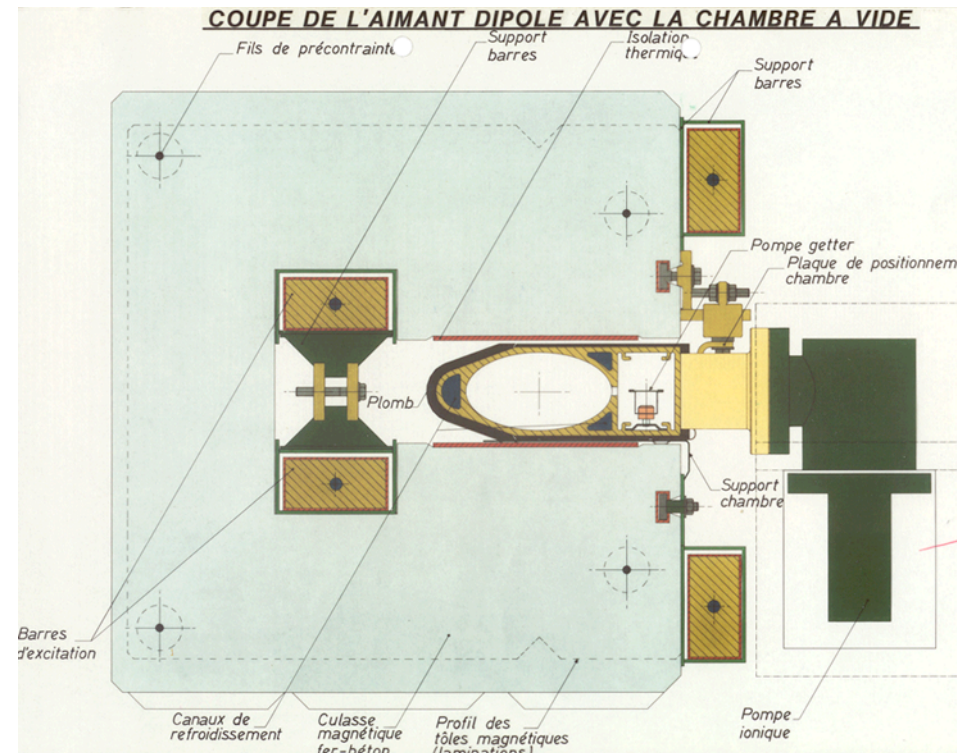
$$\ln P_{equ} = 8.275 + \ln q^2 - \frac{7000}{T}$$



## Pumping speed of a St101 getter

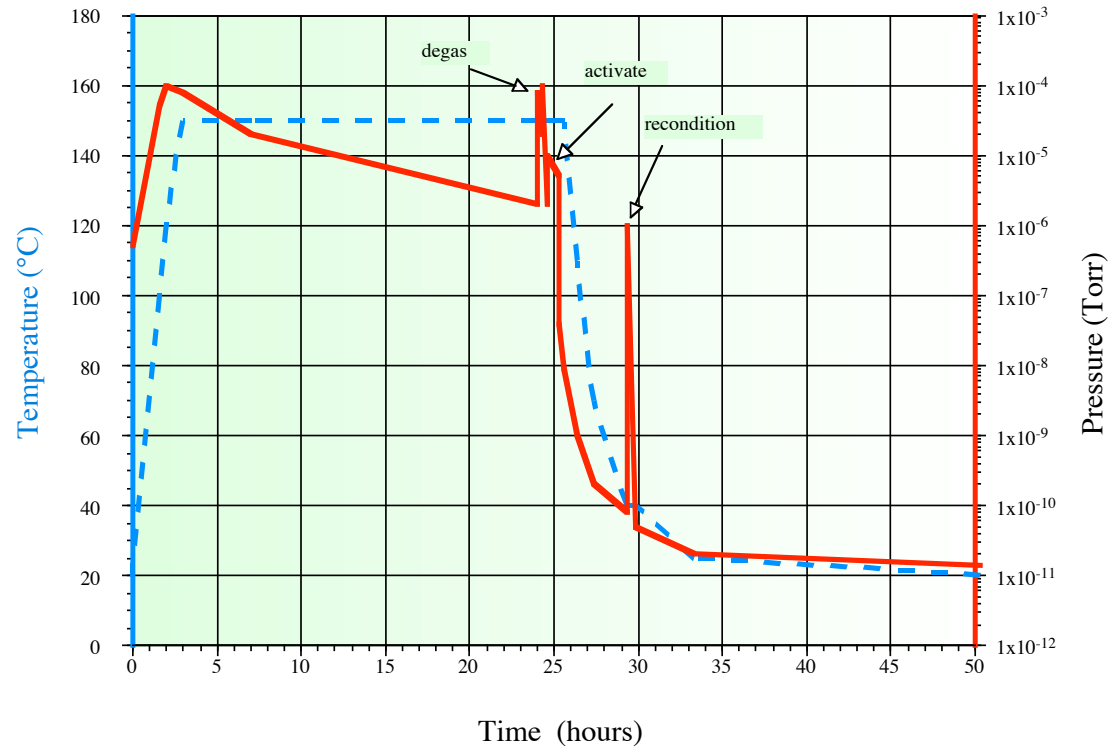


Reduction of the pumping speed due to the gradual saturation of the surface by adsorbed molecules. During **re-activation**, molecules diffuse into the bulk. A fresh surface is obtained for the next pumping cycle.



## Preparation of LEP vacuum system with NEG pumps

Typical bakeout cycle with NEG



Within less than 12 hours after the bakeout uHV conditions can be achieved.

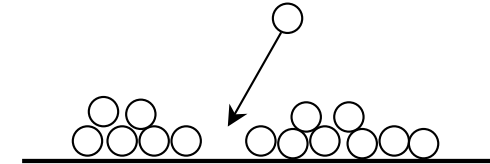


## Cryo-Pumping

Adsorption of molecules at low temperature -> e.g. at liquid helium temperature

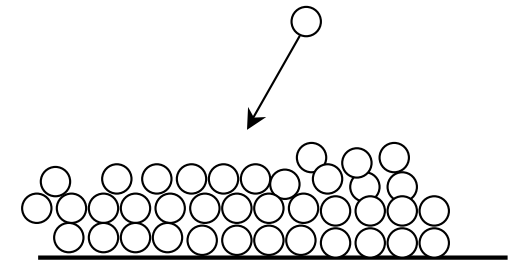
### A) Sorption

Adsorption of gas molecules with low surface coverage, to avoid the effect of the vapour pressure of the condensate. Increasing the effective surface area by a coating with a large specific surface area e.g. charcoal. -> Adsorption isotherms.



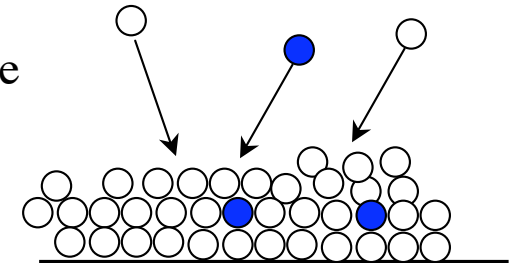
### B) Condensation

adsorption in multi-layers -> limitation due to the vapour pressure of the condensed gas.



### C) Cryo-trapping

Cryo-sorption of a gas e.g.  $H_2$  or  $He$  with a **high** vapour pressure in the presence of an easily condensable carrier gas e.g.  $Ar$ .



## Characteristics of cryo-pumping :

### Large pumping speed

Low limit pressure  $\square$  -> limitation occurs only by the vapour pressure of the condensed gas

Large capacity -> **Attention**  $\square$ ! hazardous over-pressure may build-up during warming up of a cryo-pumped system. ! -> a safety valve required  $\square$

-> 'clean' vacuum -> absence of heavy hydro-carbon molecules.

-> in combination with superconducting magnets or accelerating cavities, very effective **integrated cryo-pumping** can be obtained -> e.g. LHC vacuum system.

-> the walls of the vacuum system act as pumps (LHC).

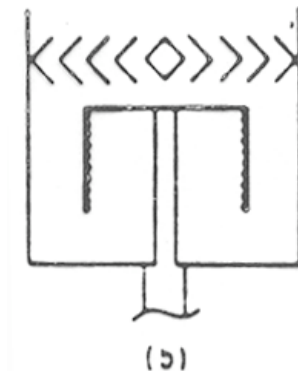
The saturated vapour pressure limits the total quantity of gas which can be cryosorbed at low pressure.

Increase of the specific surface area of a cryo pump by using special cryo-sorbing materials with a large specific surface -> e.g. activated charcoal.

Design of a commercial cryo-pump with a cryo-panel :

Baffle is needed to intercept thermal radiation.

Risk of overpressure during warm-up when too large amount of gas has been condensed.



## Cryopumps in accelerator vacuum systems

In combination with superconducting magnets or accelerating cavities, at little (or no) extra cost very effective **integrated cryo-pumps** can be obtained in an otherwise conductance limited vacuum systems.

Large freedom in the design of cryopumps : since the cold walls of the vacuum system act as pumps (LHC).

The limitations of cryopumps due to the exposure to environmental room temperature radiation and to the bombardment by beam induced energetic particles (photons, electrons, ions) must be taken into account.

Imposes -> LN<sub>2</sub> cooled baffles and the LHC beam screen. This requirement arises not only for heat load reasons but mainly to avoid re-desorption of molecules.





## Pirani gauge, thermal conductivity gauge

Change of the thermal conductivity with pressure

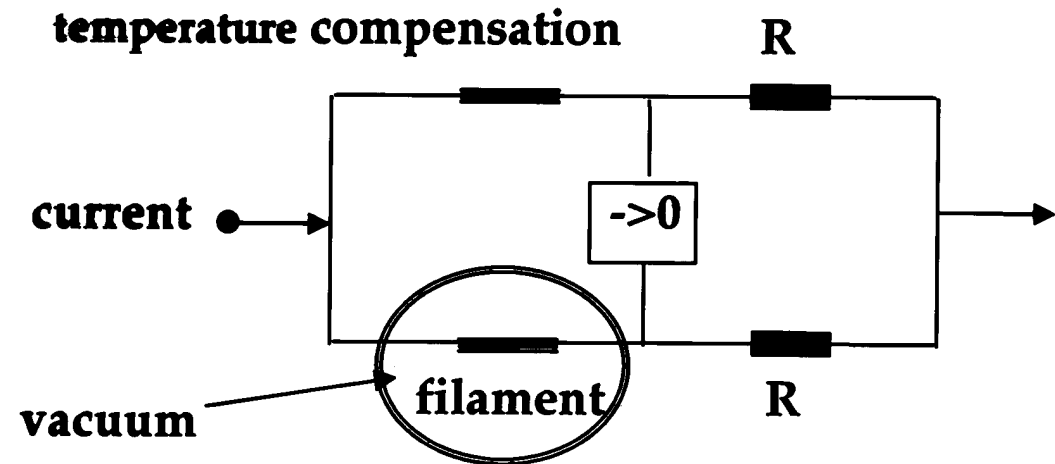
Reliable and simple system.

Pressure range:

atmospheric pressure to  $< 0.1$  Pa

A resistor with a large temperature coefficient is mounted inside the vacuum. This resistor is heated to a constant temperature and the required heating current is a measure of the pressure.

The electronic circuitry (Wheatstone-bridge) provides temperature compensation and linearization of the pressure reading.



## **Cold Cathode Ionisation Gauge, Penning Gauge**

Based on the operating principle of an ion pump:

Ratio of pressure and pump current  $\sim$  constant.

Useful pressure range :  $10^{-2}$  to  $10^{-7}$  Pa

Limitations:

At high pressure the discharge is unstable (arcing)

At low pressure the discharge extinguishes  $\rightarrow$  zero pressure reading

Leakage current in the cables and in the gauge can simulate a higher pressure

Contamination of the gauge may change the calibration.

Extended operation at very high pressure can contaminate the gauge  $\rightarrow$  required demounting and cleaning of the gauge.

Improved version for low pressures are on the market: Inverted magnetron gauge

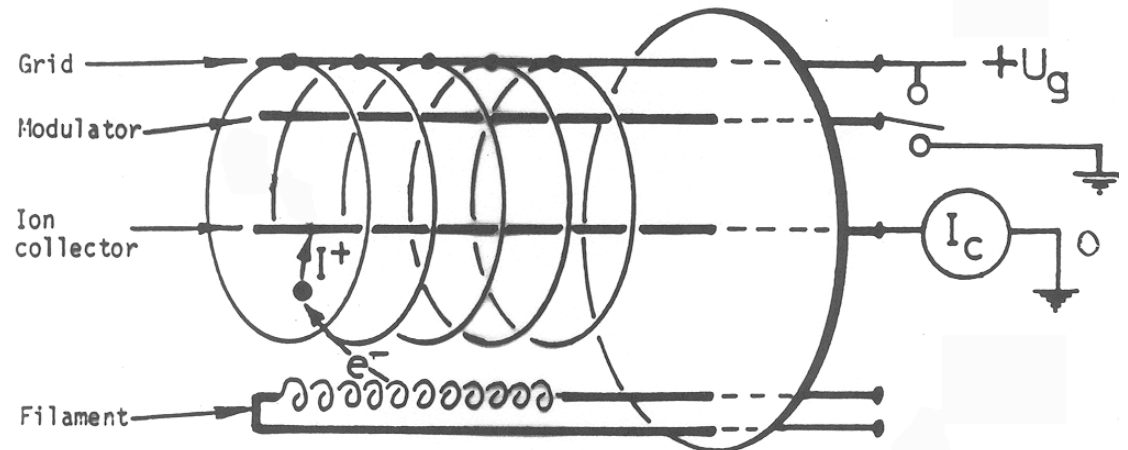
## Hot Filament ionization Gauge

### Operating principle

Residual gas molecules are ionized by the electrons emitted from a hot filament.

Ions are collected by a "collector electrode".

This ion current is proportional to the gas density,  $n$ , and hence to the pressure,  $P$ .



The ionization probability  $P_i$

(number of ion–electron pairs produced per m and per Pa) depends on the type of molecule and on the kinetic energy of the electrons.

Ion collector current :  $I^+ = I_e P_i L P$

Where  $I_e$  emission current of the filament

$L$  path length of the electrons

$P$  pressure

## Gauge Sensitivity

$$S = P_i L \quad [\text{Pa}^{-1}]$$

Obtained by calibration with a known pressure ( $N_2$ )

→ Nitrogen equivalent pressure  $N_2$ .

→ To measure a pressure for another gas, the relative gauge sensitivity for this particular gas with respect to nitrogen must be known.

→  $S_i/S_{N_2}$  must be known for different gas species.

For  $H_2$ , one finds typically  $S_{H_2}/S_{N_2} \sim 0.38$

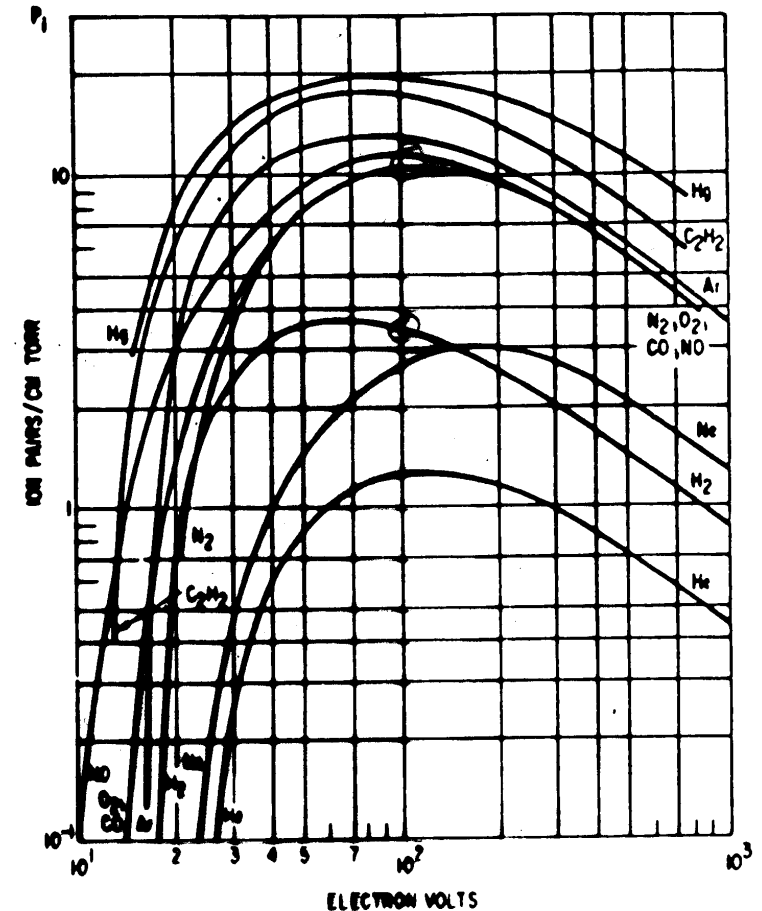


FIGURE 2. Probability of ionization as a function of electron energy for various gases at 1 Torr and 0°C.<sup>1</sup>



## “Outgassing” of an Ionization Gauge

Electrons emitted from the filament, reach the grid electrode with an energy of approximately  $\sim 150\text{eV}$  and may heat-up the grid and desorb gas molecules.

-> pressure increase which disturbs the measurement.

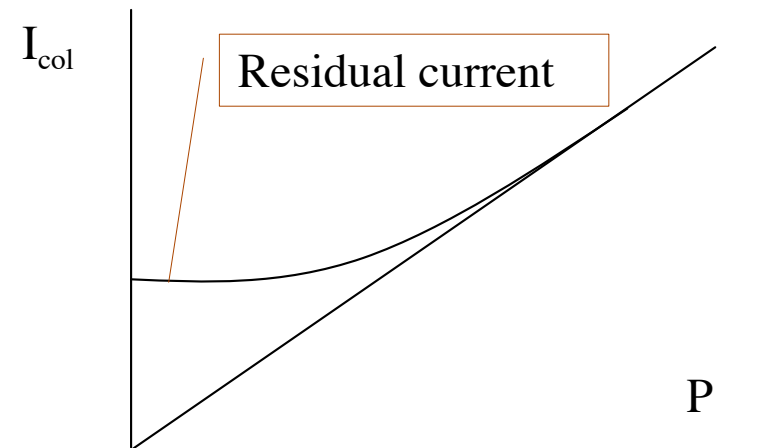
-> To suppress this effect, the grid and all other electrodes must be cleaned.

A common method to avoid this disturbance:

the emission current is increased to heat the grid electrode temporarily to a very high temperature. -> Outgassing mode of the gauge.

Ultimate pressure limitation:

Finite residual current -> limits pressure reading



## X-Ray Limit of an Ionization Gauge

Electrons, which hit the grid produce soft photons (x-rays) due to Bremsstrahlung.

A small fraction of these photons can reach the collector electrode and liberate photo-electrons which return to the grid -> this current is equivalent to a positive, “x-ray current”  $I_x$ , which is independent of the pressure.

The correction of this effect can be done by the «modulation» method

### ‘Modulated Bayard-Alpert’ gauge

|  |   |
|--|---|
| Modulator electrode                    |   |
| measurement (1), + $U_{\text{grille}}$ | $I_1 = I^+ + I_x$                                 |
| measurement (2), 0 V,                  | $I_2 = \square I^+ + I_x$                         |
| Corrected collector current            | $I^+ = \frac{I_1 \square I_2}{1 \square \square}$ |

The modulation factor:  $1 - \square$

can be determined easily by an independent measurement at high pressure where  $I^+ \gg I_x$ .

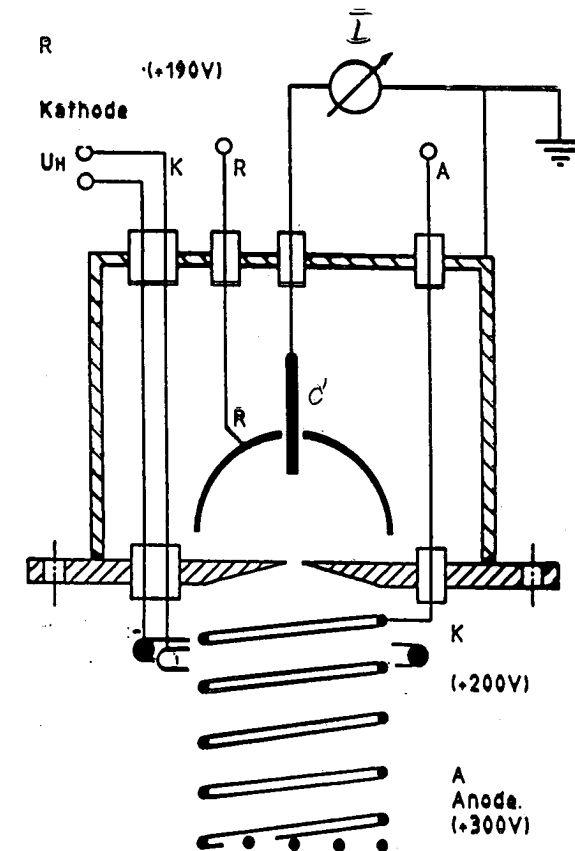
**Alternative design of an ionization gauge:**

Ions are 'extracted' from the grid volume to a collector, which is 'optically' screened from the photons of the grid.

**-> Extractor gauge by Redhead**

- R Reflector, bias voltage
- K Filament, cathode
- C Ion collector, at ground potential
- A Anode (grid)

Ions are extracted from the grid volume through a mask.  
The ion collector is 'optically' shielded from the x-ray photons produced at the grid.



## Partial Pressure Measurement

Combination of an ion source with a mass spectrometer.

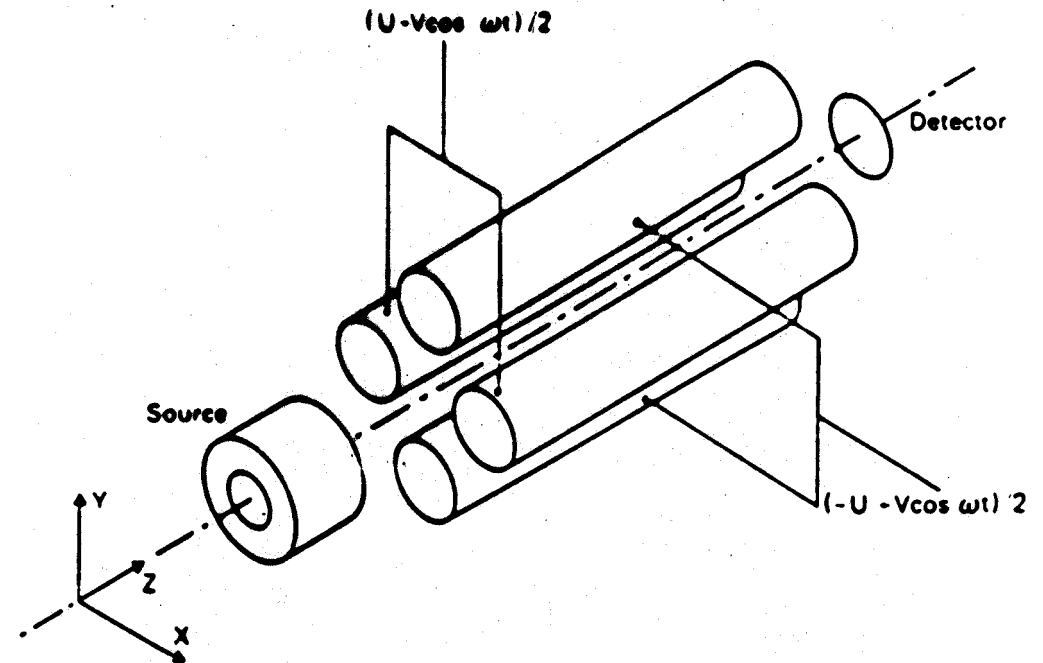
- 1) Ion source
- 2) Quadrupole mass filter
- 3) Ion collector, Faraday-cup or secondary electron multiplier

Operating principle of the quadrupole mass filter :

Ions with different mass to charge ratio are injected into the quadrupole structure. As they traverse the structure, ions are subjected

to a periodically varying transverse electric field, which excites transverse oscillations.

Ions with an incorrect charge to mass ratio have unstable orbits, are lost and do not reach the collector.



The ion trajectories are described by the equations :

$$\ddot{x} + \left(\frac{e}{m} r_o^2\right) \square x = 0$$

$$\ddot{y} \square \left(\frac{e}{m} r_o^2\right) \square y = 0 \quad \text{and} \quad \ddot{z} = 0$$

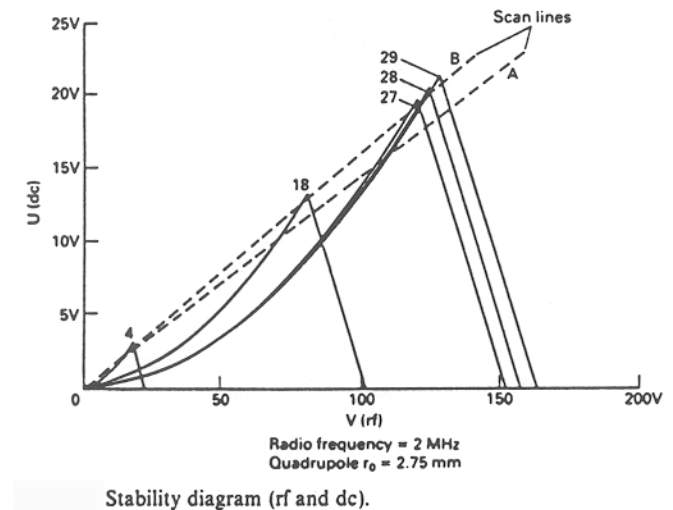
$r_o$  is the radius of the structure and  $\square$  the potential:  $\square = U \square V \cos(\square t)$ .

With  $a = 4 \frac{e}{m} \frac{U}{\square^2 r_o^2}$  and  $q = 2 \frac{e}{m} \frac{V}{\square^2 r_o^2}$  -> **Mathieu equation**

$$\frac{d^2 u}{ds^2} + \{a \square 2q \cos(2s)\} u = 0$$

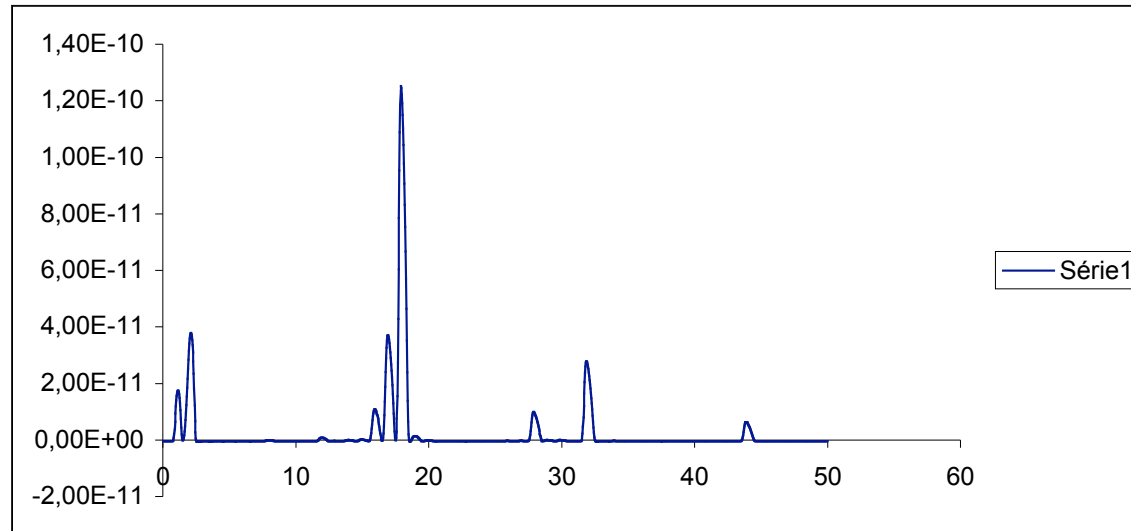
Solutions define regions with stable and unstable motion. Only the ions ( $e/m$ ) with stable trajectories through the quadrupole mass filter are measured.

### Stability Diagram



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## Residual gas spectrum



Ion current (A) corresponding to a particular molecular species (e/m)

N<sub>2</sub>-equivalent pressure can be obtained with a total pressure gauge.

**Qualitative analysis** is relatively straightforward, e.g. the identification of:



Distinction between CO/N<sub>2</sub> requires knowledge of the “**cracking pattern**” of the two molecular species.

**Quantitative analysis** requires a perfectly calibrated system for each residual gas component and can be very difficult. -> Note: do not rely on the manufacturer’s calibration.

**Typical cracking patterns of common molecules**

| peak             | 2   | 14   | 15 | 16  | 17 | 18  | 20 | 27 | 28   | 32  | 40  | 44  |
|------------------|-----|------|----|-----|----|-----|----|----|------|-----|-----|-----|
| H <sub>2</sub>   | 100 |      |    |     |    |     |    |    |      |     |     |     |
| CH <sub>4</sub>  | 2.4 | 10.7 | 85 | 100 |    |     |    |    |      |     |     |     |
| H <sub>2</sub> O |     |      |    | 1.8 | 27 | 100 |    |    |      |     |     |     |
| N <sub>2</sub>   |     | 6.2  |    |     |    |     |    |    | 100  |     |     |     |
| CO               |     | 0.9  |    |     |    |     |    |    | 100  |     |     |     |
| O <sub>2</sub>   |     |      |    | 18  |    |     |    |    |      | 100 |     |     |
| A                |     |      |    |     |    |     | 13 |    |      |     | 100 |     |
| CO <sub>2</sub>  |     |      |    | 7.5 |    |     |    |    | 18.5 |     |     | 100 |

Principal peak of each species is normalised to 100%.

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## Residual gas spectrum of a baked, 'clean' uhv system

Qualitative observations:

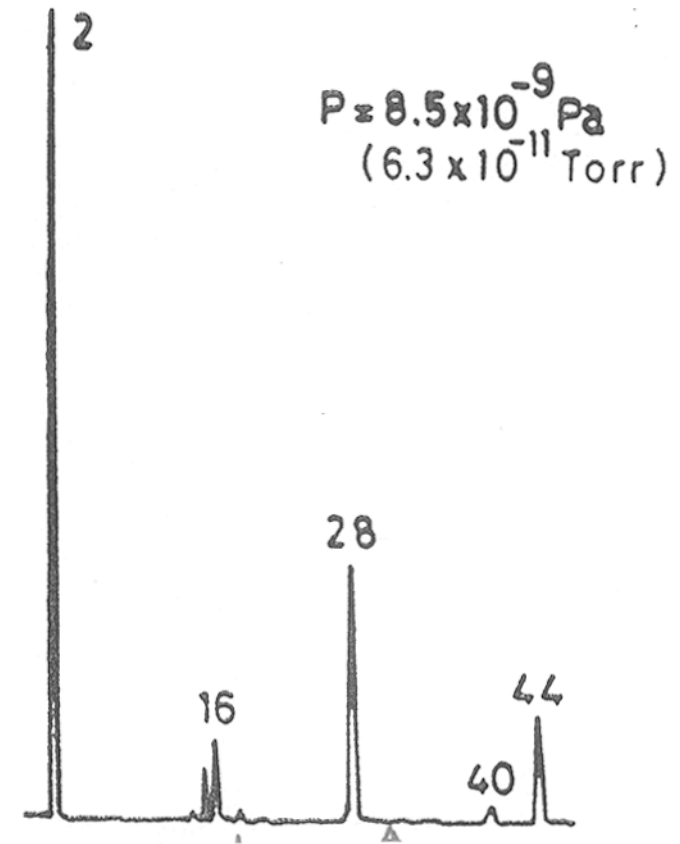
Absence of peaks 17,18 : H<sub>2</sub>O has been removed during the bakeout.

Absence of peaks due to hydrocarbons: small trace of CH<sub>4</sub>

Presence of peak 40 may indicate small air leak, or release of argon from an ion pump.

Warning:

CH<sub>4</sub> can be produced by the hot filament of an ionization gauge

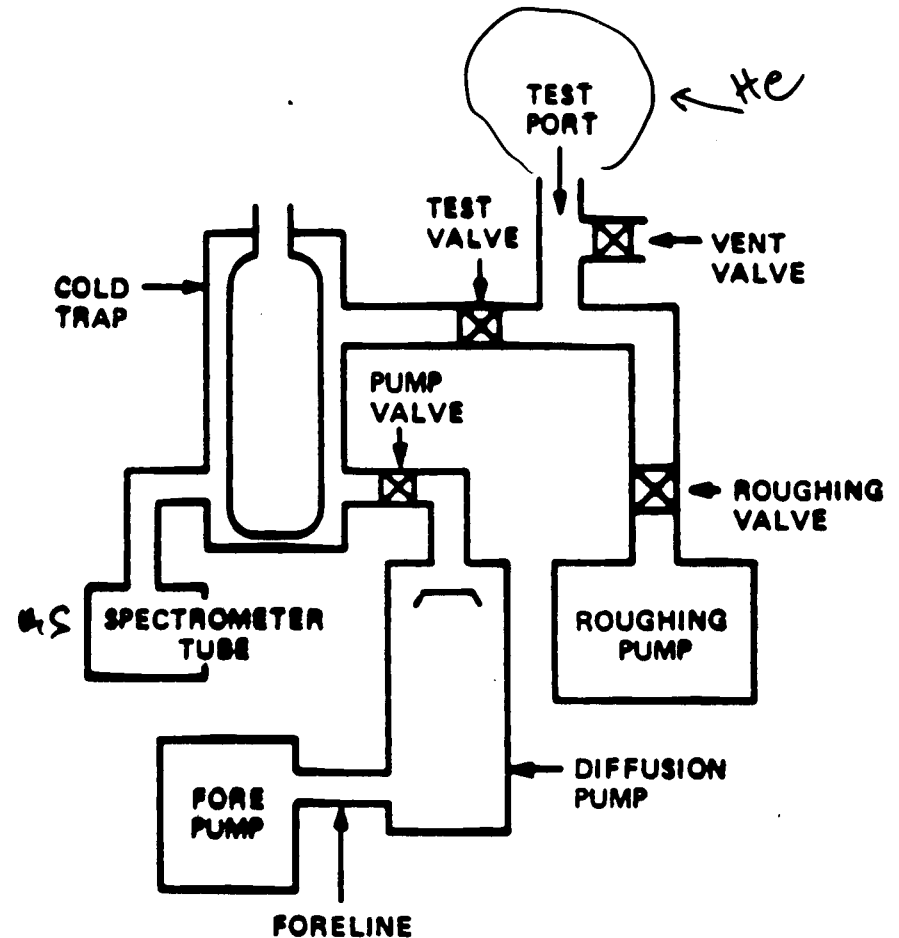




## Helium leak detector

Mass spectrometer tuned to Helium, which is commonly used as the 'tracer gas'.

Purpose of the  $\text{LN}_2$  filled cold trap is to remove oil vapours of the diffusion pump as well as water vapour from the spectrometer cell



## Leaks and leak detection

Common leaks to atmospheric pressure:

Gaskets

Porosities in the materials

Cracks in welds

**Virtual leaks:** are not found by a conventional leak check

Porosities, a dead volume enclosed inside the system

Example of a virtual leak: The volume enclosed by a bolt in a threaded hole.

Solution: bolts have to be drilled with a central hole or a separate hole must be drilled to pump the dead volume.

In a large vacuum system, leak checks of all sub-components are mandatory.

A global leak check after complete assembly should only concern those joints, which have been made during the final installation phase in the accelerator.

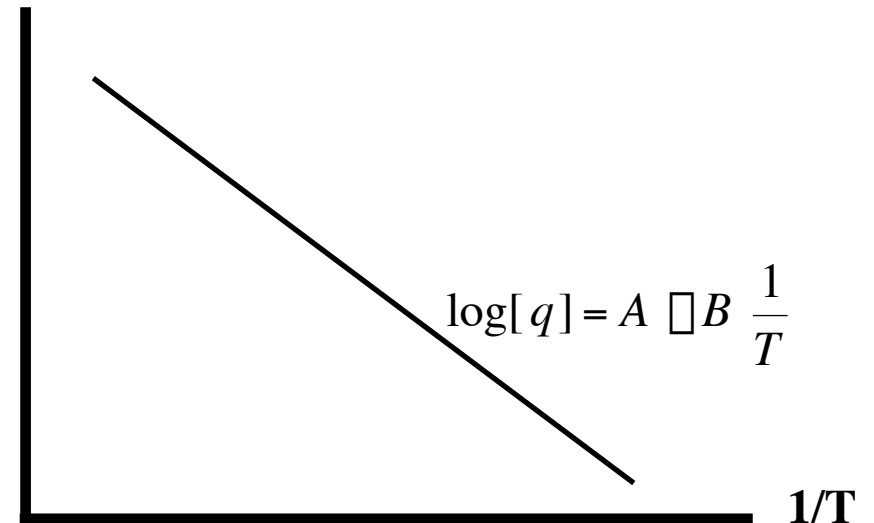
## Thermal Desorption

Specific desorption rate :  $q$  [Pa m<sup>3</sup> s<sup>-1</sup> m<sup>-2</sup>]  $q = \text{Const} \cdot e^{-\frac{E}{kT}}$

Molecular residence time  $\tau = \frac{1}{\nu_0} \cdot e^{\frac{E}{kT}}$

$E$  activation energy for desorption,  
 $\nu_0 \sim 10^{13}$  s<sup>-1</sup> vibration frequency in  
 the surface potential

Log[q]



**Physisorbed** molecules  $E < 40$  kJ/mole (0.4 eV)

**Chemisorbed** molecules  $E > 80$  kJ/mole (0.8 eV)

**Bakeout** between 150 – 300°C : reduced residence time.

Reduction for H<sub>2</sub>O, CO, CO<sub>2</sub> (by factors of 10<sup>-2</sup> to 10<sup>-4</sup>)

At higher temperature > 400-500°C -> cracking of hydrocarbon molecules (C-H)

Note: Strongly reduced thermal desorption at cryogenic temperatures

### Chemical solvent pre-cleaning procedure

- 1) Removal of gross contamination and machining oils using the appropriate solvents
- 2) Perchloroethylene ( $C_2Cl_4$ ) vapour degreasing at ( $121^\circ C$ ) to day no longer applicable
- 3) Ultrasonic cleaning in an alkaline detergent ( $pH = 11$ )
- 4) Rinsing in cold demineralised water (electrical conductivity  $< 5 \mu S cm^{-1}$ )
- 5) Drying in a hot air oven at  $150^\circ C$
- 6) Wrapping in clean Al-foil or paper

Cleaning method will depend on the material (stainless steel, aluminium, copper)

Important:

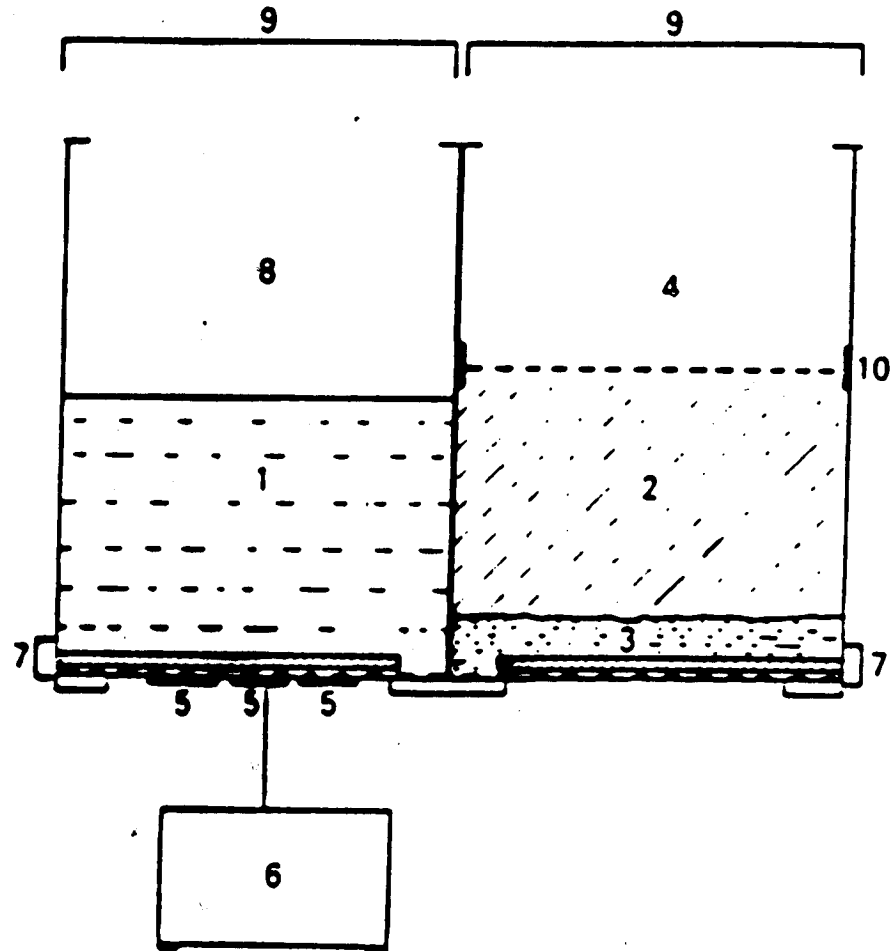
Any subsequent handling must be done with **clean gloves**.

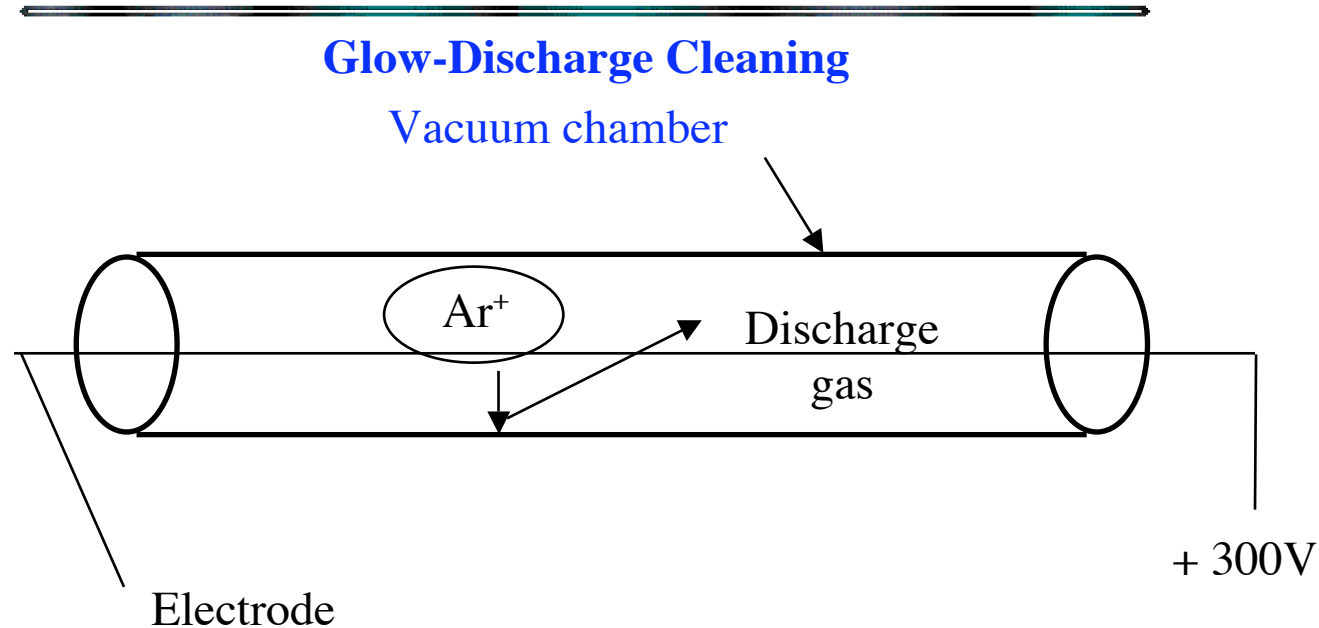
Contamination by any residues in the air must be avoided.

**No car exhaust gases, No smoking!!**

## Chemical cleaning facility

- 1) Hot detergent with ultrasonic agitation
- 2) Hot vapour zone
- 3) Hot solvent bath
- 4) Cooling zone
- 5) Ultrasonic generators
- 6) Ultrasonic controls
- 7) Heaters
- 8) Drying zone
- 9) Covers
- 10) Cooling zone for solvent vapour





Cleaning of the surface by energetic ion bombardment (Usually Argon or some other inert gas)

Dose approx.  $10^{18}$  -  $10^{19}$  ions/cm<sup>2</sup>

Argon pressure between  $10^{-1}$  –  $10^{-2}$  Pa for optimum conditions

Desorption of chemisorbed, strongly bound molecules corresponding to a high activation energy.

Effective cleaning by removing the top layer of the surface by sputtering. -> Tokamak vacuum systems

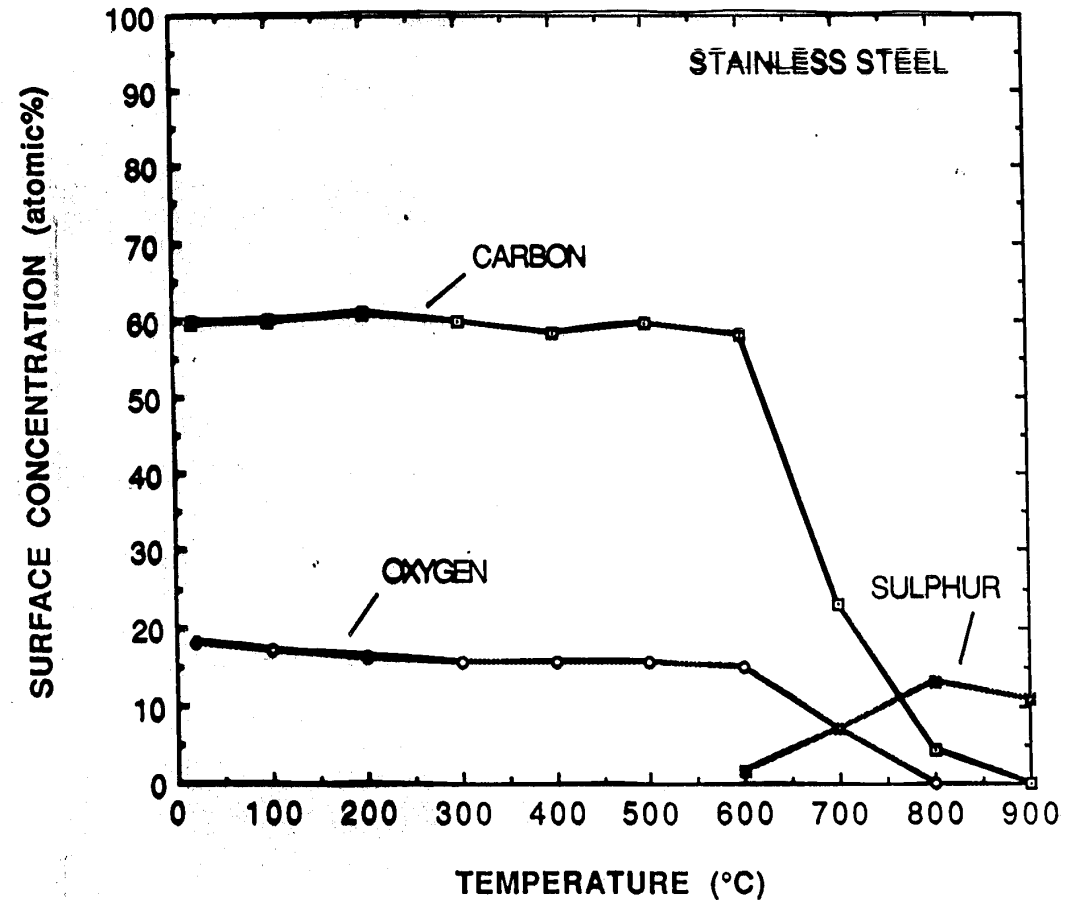
## Vacuum firing at high temperature

High temperature baking in a vacuum oven at ~950 deg C

Cracking of hydrocarbons and organic compounds.

Reduction of the surface oxide layer.

After the high temperature treatment, cool down in a clean gas to generate a controlled oxide layer



## Thermal outgassing Rates of some materials

### Comparison of organic materials and of metals

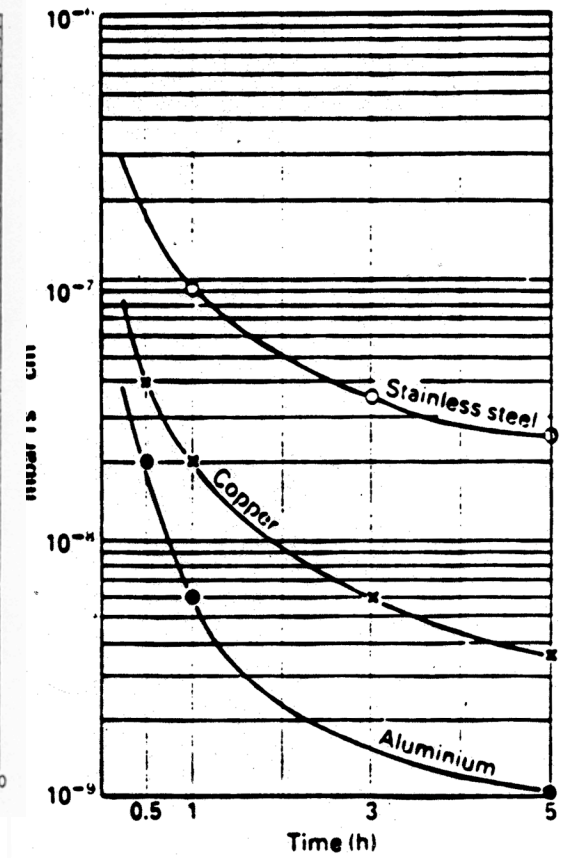
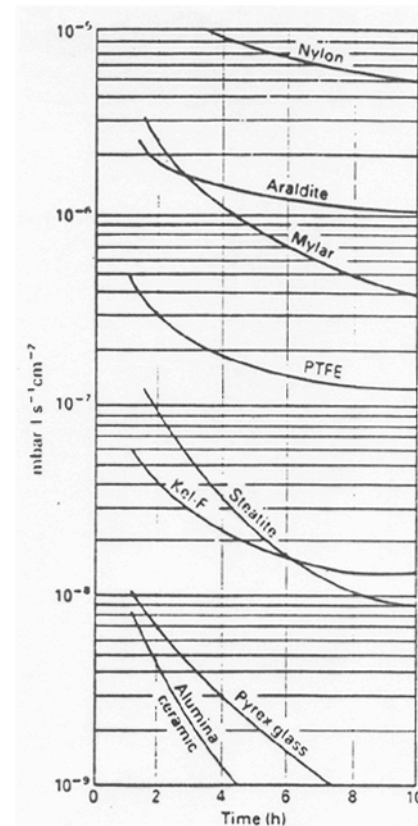
Unbaked samples (usually H<sub>2</sub>O dominates)

Baked samples

(24 hours at 150°C to 300 °C)

Typical values after 50 hours of pumping :  
(units : Torr l s<sup>-1</sup> cm<sup>-2</sup>)

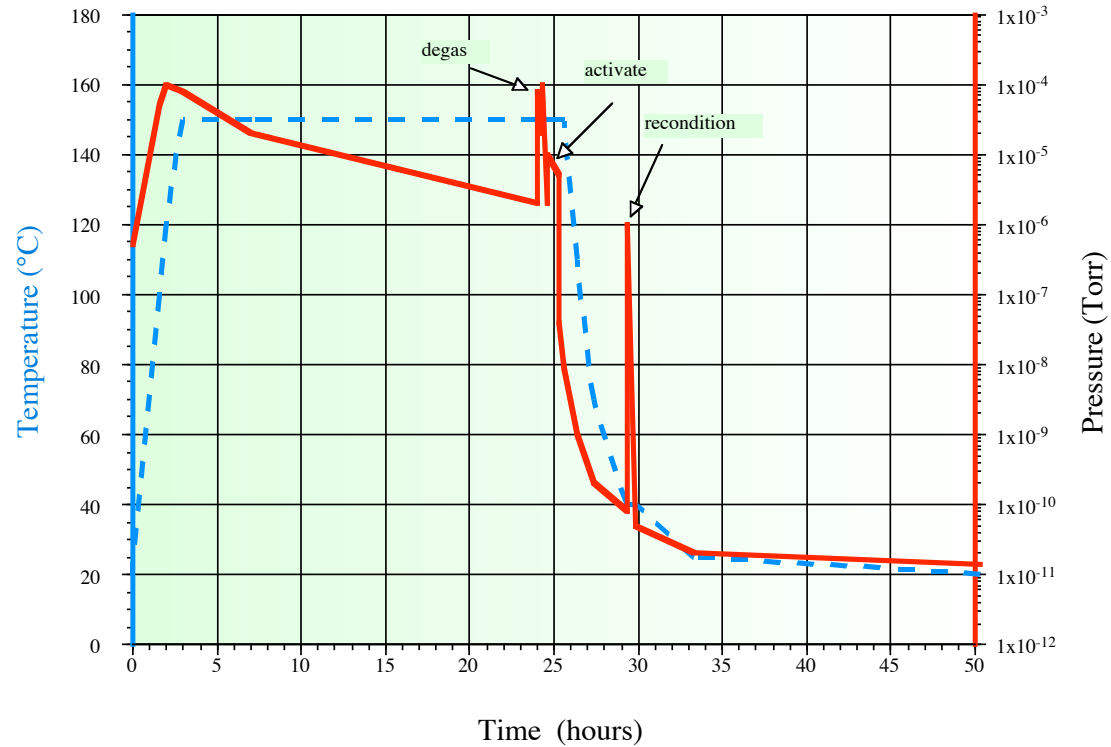
| Gas             | Al, Stainless steel |
|-----------------|---------------------|
| H <sub>2</sub>  | 5 10 <sup>-13</sup> |
| CH <sub>4</sub> | 5 10 <sup>-15</sup> |
| CO              | 1 10 <sup>-14</sup> |
| CO <sub>2</sub> | 1 10 <sup>-14</sup> |





## Bakeout of the LEP Vacuum System with NEG

Typical bakeout cycle with NEG



Within less than 12 hours after a bakeout uhv conditions can be achieved.

## Criteria influencing the Choice of Materials

Low outgassing rate

Low vapour pressure

Temperature resistant -> bakeout

Thermal and electrical conductivity -> beam interaction

Corrosion resistance -> leaks

Low induced radioactivity -> handling

High mechanical strength -> 1dN/cm<sup>2</sup> external pressure!

Machining, welding

Low cost

### Common choices:

Stainless steel

Aluminium

Copper

Ceramics for electric insulation

    Low porosity -> leaks

    Brazing to metal -> leaks

For particular applications

Organic materials (e.g. as composite materials (carbon-fibers & epoxy), polymers to be used in small quantities

## Synchrotron Radiation Induced Desorption

Radiated power (W)  $\square$   $P_{\square} = 88.6 \frac{E^4 I}{\square}$   $E$ , beam energy of electrons (GeV)  
 $I$ , beam current (mA),  $\square$ , bending radius (m),

Critical energy of the spectrum (eV)  $\square_c = 2.2 \cdot 10^3 \frac{E^3}{\square}$

Photon flux (s<sup>-1</sup>)  $\square = 8.08 \cdot 10^{17} I E$

Linear photon flux (m<sup>-1</sup> s<sup>-1</sup>)  $\frac{d\square}{ds} = 1.28 \cdot 10^{17} \frac{I E}{\square}$

Gas desorption occurs in two steps  $\square$  1 -> photons -> produce photo-electrons

2-> photo-electrons -> excite molecules which subsequently will desorb thermally

Gas flow  $\square$   $Q = \square \square$

$Q = K \square I E + Q_o$  with  $Q_o$ , the thermal desorption rate and  $\square$ , molecular desorption yield (molecules per photon).

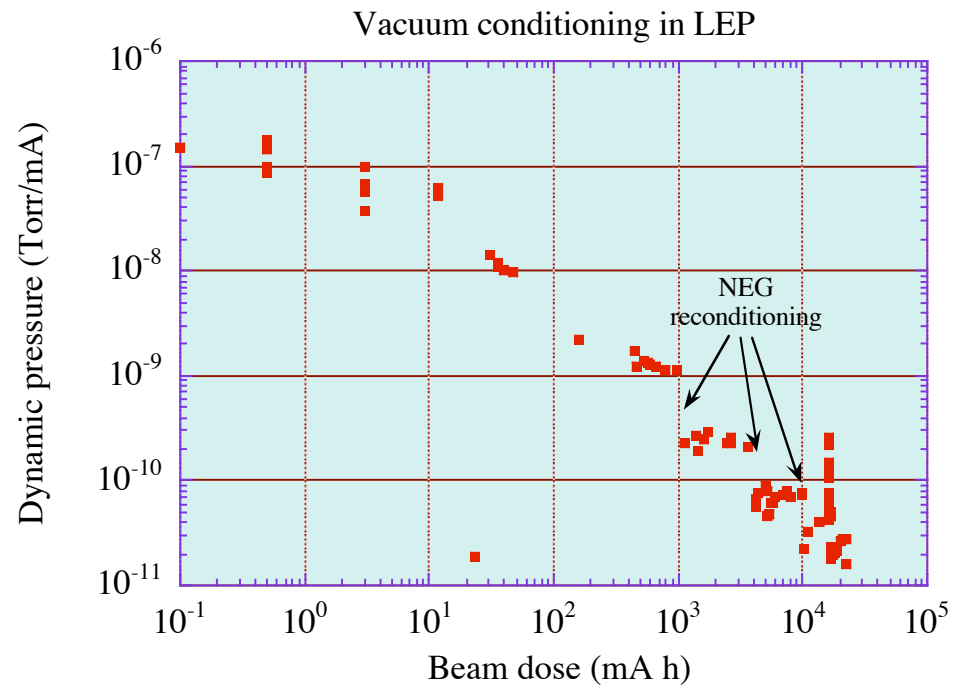
Dynamic pressure  $\square$   $P_{dyn} = \frac{Q}{S}$ .

The dynamic pressure increases proportionally with the beam intensity :  $\frac{\square P}{I}$  (Pa/mA).

‘Beam cleaning’ (scrubbing) of the vacuum system is a vital procedure.

## Beam cleaning (scrubbing) of the LEP vacuum system

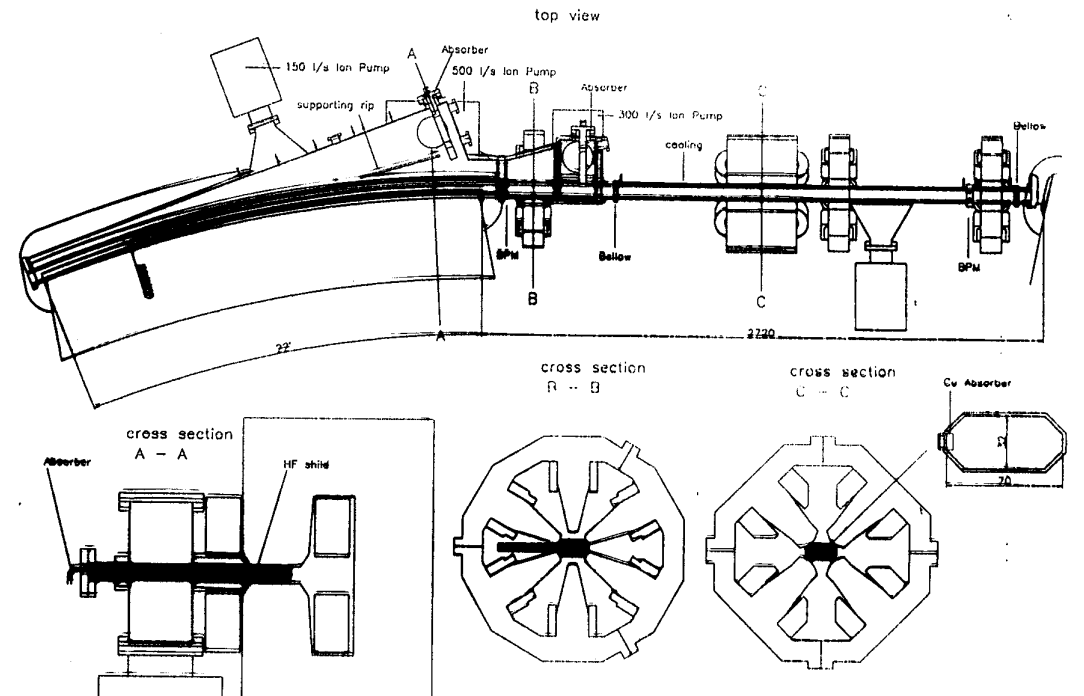
Dose scale may be given in terms of accumulated photons/m or more frequently in mAh.



## Vacuum vessel of a synchrotron radiation light source

Bending magnet vessel with 'ante chamber' and light port.

Synchrotron radiation absorbers protect critical elements of the machine.



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## Water cooled absorber with integrated vacuum pumps

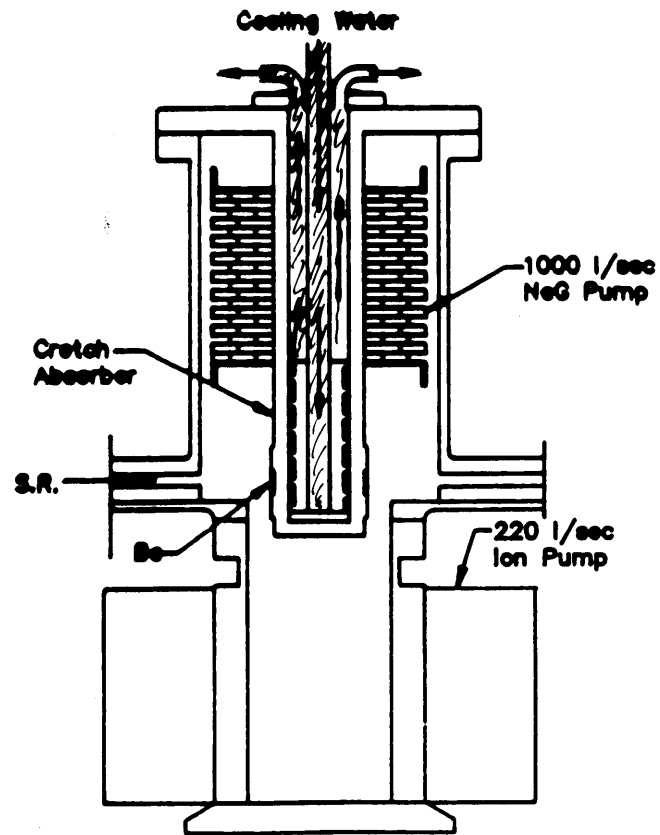
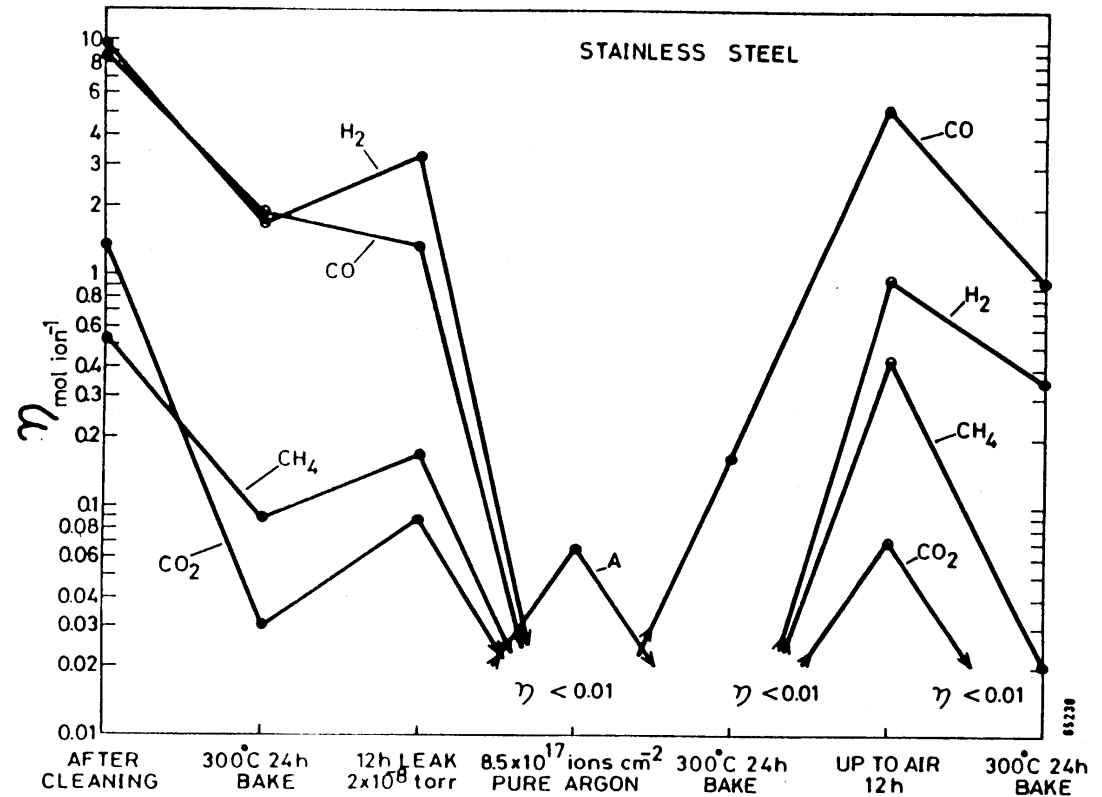


Fig: 5 Water cooled crotch absorber

## Comparison of successive vacuum treatments

Effect of various surface treatments including exposure to atmospheric air on the ion stimulated desorption yield.

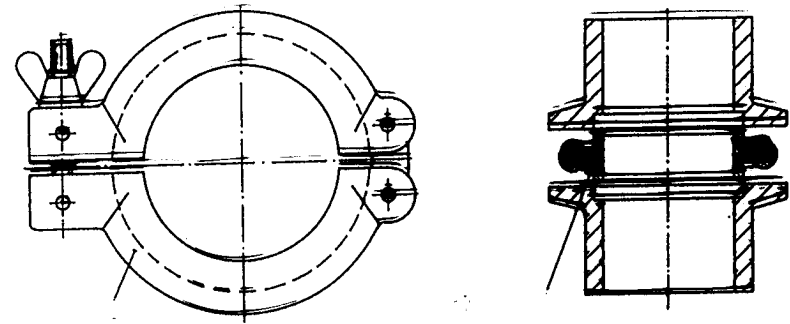
This result illustrates the importance to avoid exposure to ambient air to maintain a clean uhv system.



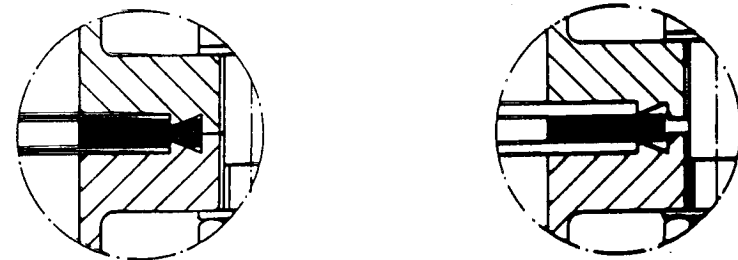
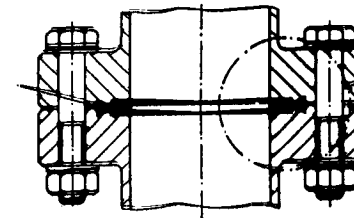
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## Flanges and gaskets for primary vacuum and for uhv applications

Flange with clamp and elastomer seal for high vacuum systems



'ConFlat' flange for uhv systems  
Copper gasket for 'all metal' vacuum system



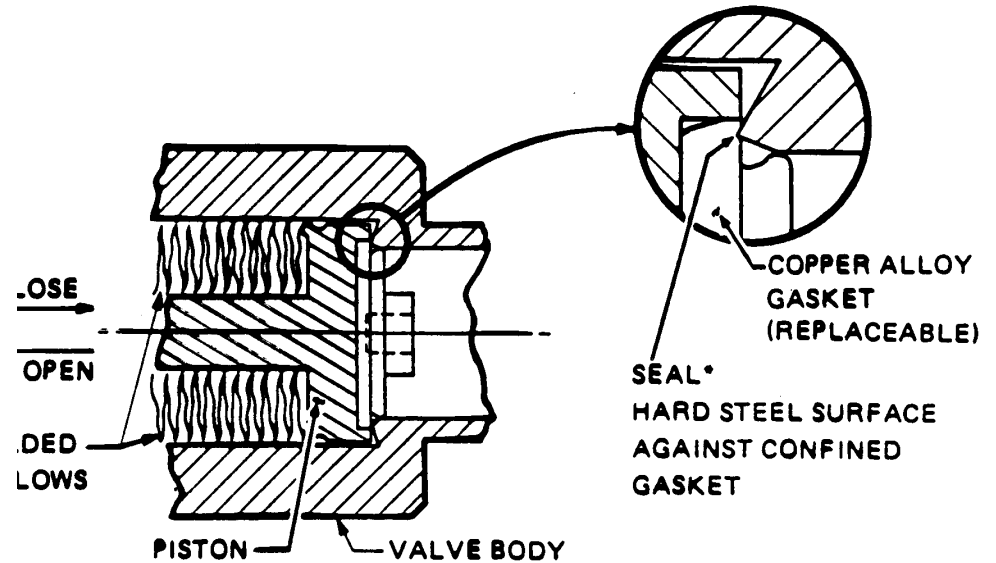
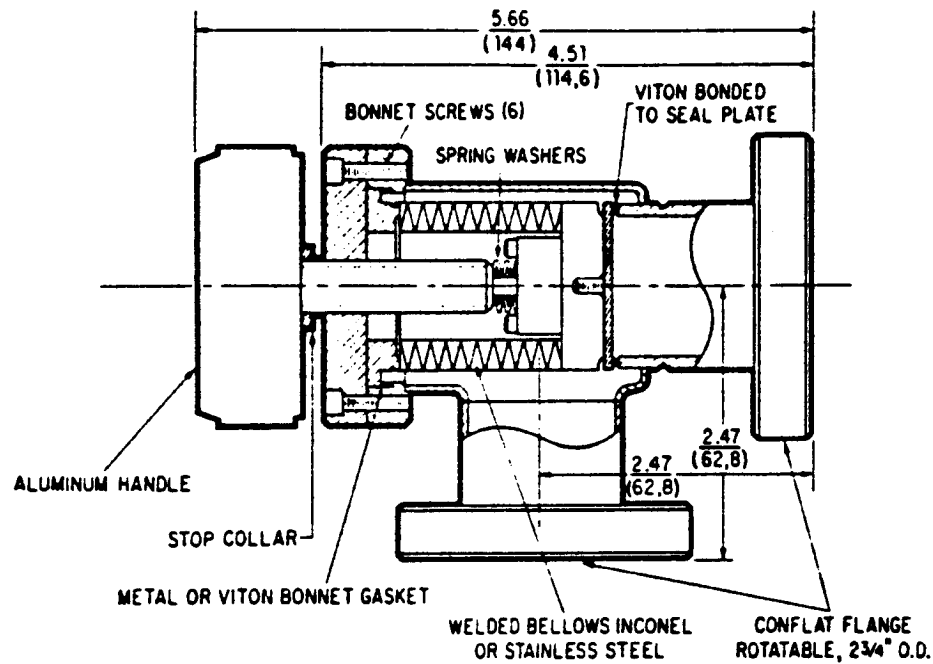


## Valves for high and ultrahigh vacuum

UHV valves use all metal construction (copper seals)

Manual valve with Viton seal for high vacuum applications ( $>10^{-4}$  Pa)

Manual Valves, Viton Main Seal



\* SEAL IS IDENTICAL IN ALL VALVES; PARTIAL VALVE BODY SHOWN IS THAT OF MINI-VALVE.