CERN

Vacuum Systems
Lecture 3

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Outline

1. Cryopumping
2. Adsorption isotherms
3. Crysorber in cold systems: case of LHC
4. Helium leaks in cold systems: case of LHC

1. Cryopumping

Desorption probability

- The desorption probability, P, is a function of the binding energy, E and the temperature, T (first order desorption, Frenkel 1924). The surface coverage, θ, varies like :

$$P = \frac{d\theta}{dt} = -\theta\, \nu_0\, e^{-\frac{E}{kT}}$$

($\nu_0 \sim 10^{13}$ Hz, k = 86.17 10^{-6} eV/K)

- The desorption process is characterized by the sojourn time, τ :

$$\tau = \frac{e^{\frac{E}{kT}}}{\nu_0}$$

- For large E and small T, molecules remains onto the surface : CRYOPUMPING

- For some combination of E and T, the molecule is desorbed (bake out)

Sojourn time at cryogenic temperature

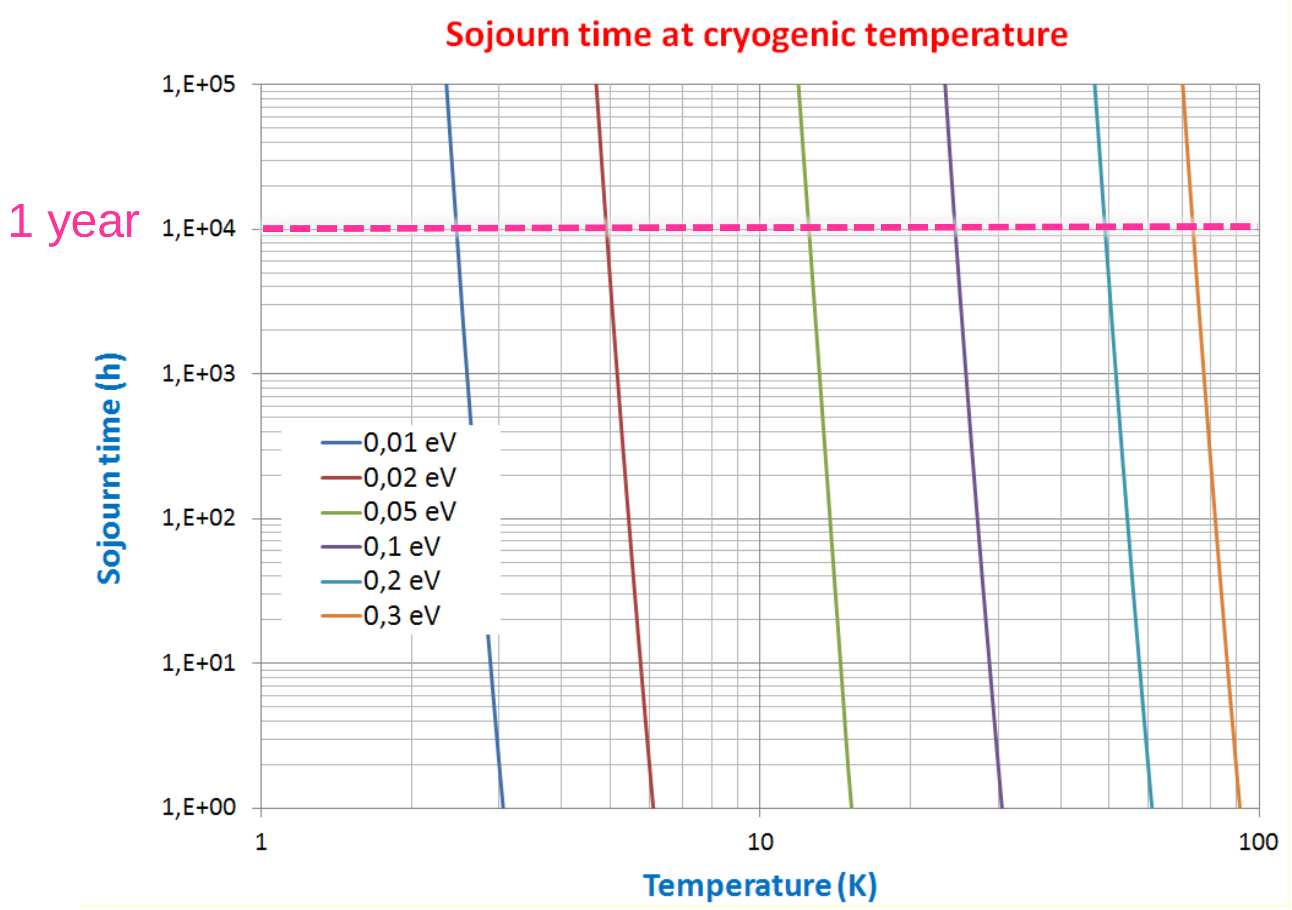
- Cryosorption occurs till ~ 100 k

E(eV)	1.9 K	4.2 K	50 K	70 K
0.01	$1\ 10^6$ years	0.1 s	1 ps	0.5 ps
0.02	∞	$3\ 10^3$ years	10 ps	2 ps
0.15	∞	∞	130 s	6 ms
0.21	∞	∞	5 years	130 s
0.3	∞	∞	$1\ 10^4$ years	12 years

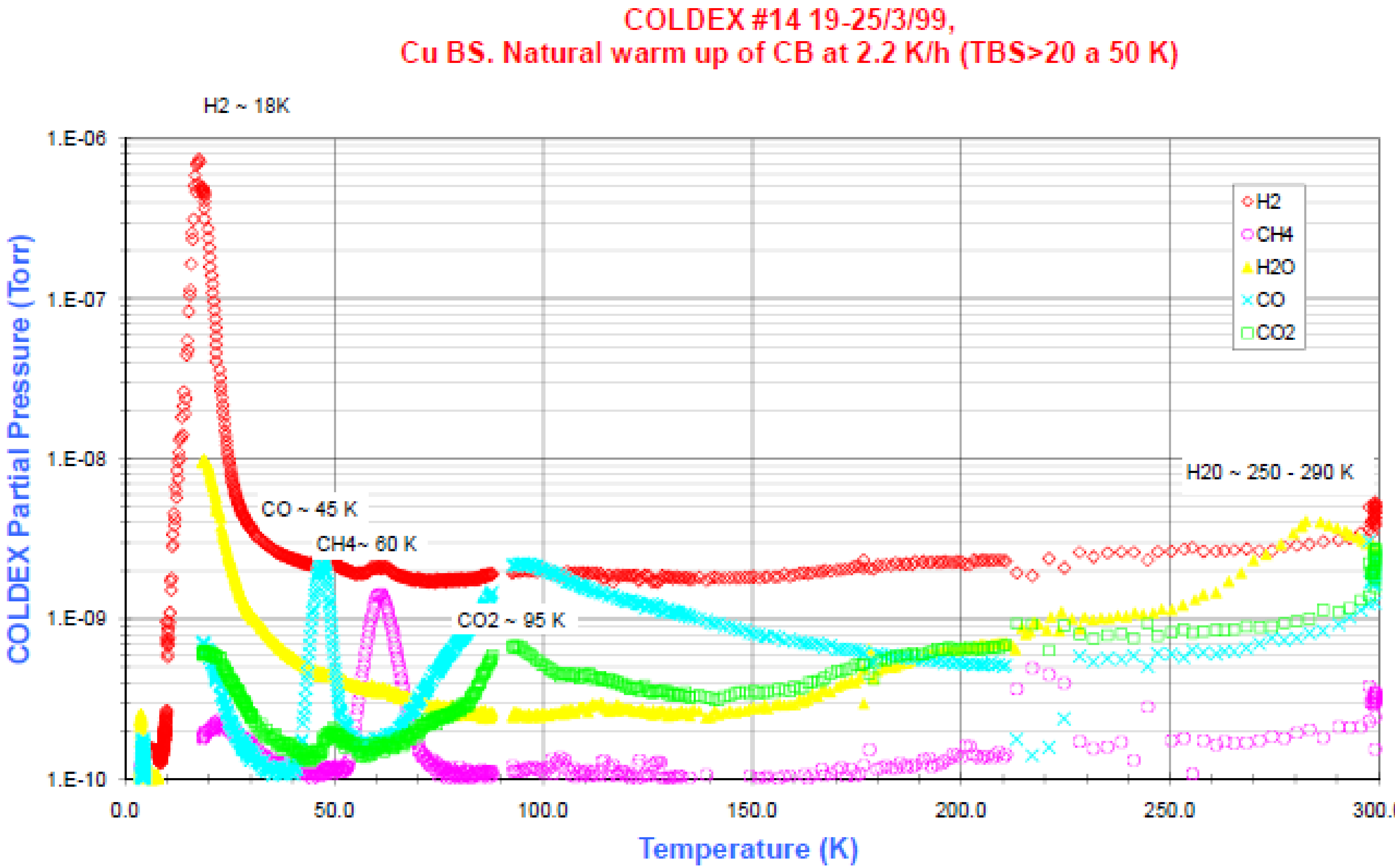
$$\tau = \frac{e^{\frac{E}{kT}}}{\nu_0}$$

Sojourn time - Physisorbed molecules

- Physisorption occurs:
  - below 20 K for binding energies < 0.1 eV
  - below 50 K for binding energies < 0.2 eV
  - below 70 K for binding energies < 0.3 eV



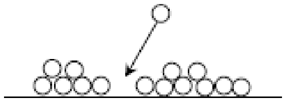
A Natural Warm Up of a St. Steel Cold Bore



Cryopumping regimes

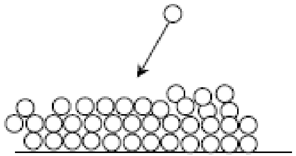
Physisorption

- Sub-monolayer coverage : attractive force (van der Waals) between a gas molecules and a material
- Binding energy for physical adsorption
- H_2 from 20 to 85 meV for smooth and porous materials resp.
- 1 h sojourn time at 5.2 K and 26 K for smooth and porous materials resp.



Condensation

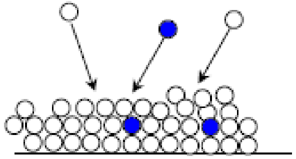
- For thick gas coverage, only forces between gas molecules
- Energy of vaporisation 9 to 175 meV for H_2 and CO_2 resp.
- 1 h sojourn time at 2.8 K and 53.4 K for H_2 and CO_2 resp.



➔ sub-monolayers quantities of gas can be *physisorbed* at their boiling temperature (ex : H_2 boils at 20.3 K and a bake-out above 100 ºC removes water)

Cryotrapping

- Use of a easily condensable carrier (*e.g.* Ar) to trap molecules with a high vapor pressure gas (*e.g.* He, H_2)



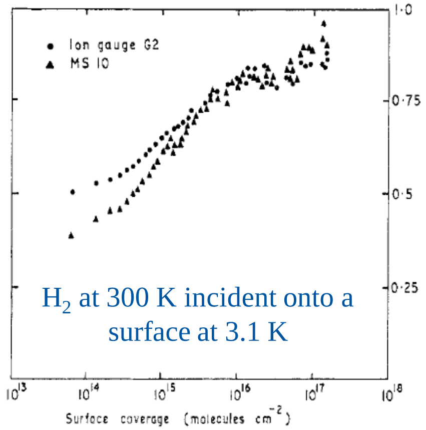
Sticking probability/coefficient

- Probability : $0 < \sigma < 1$

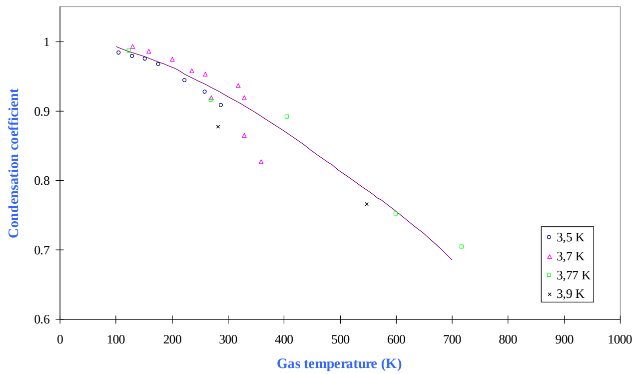
  ν collision rate (molecules.s^{-1}.cm^{-2})

$$\sigma = \frac{\nu_{\text{incident}} - \nu_{\text{departing}}}{\nu_{\text{incident}}} = \frac{\nu_{\text{sticking}}}{\nu_{\text{incident}}}$$

- Function of gas, surface, surface coverage, temperature of gas and surface temperature



J.N. Chubb *et al*. J. Phys. D, 1968, vol 1, 361



J.N. Chubb *et al*. Vacuum/vol 15/number 10/491-496

- Pumping speed

$$S = \frac{1}{4}\sigma\left(1 - \frac{P}{P_{sat}}\right)A\bar{v} \approx \frac{1}{4}\sigma A \bar{v}$$

$$S\left[l.s^{-1}.cm^{-2}\right] = 3.63\,\sigma\sqrt{\frac{T}{M}}$$

i.e : σ times the conductance of a surface

- H_2 and CO at 4.2 K :

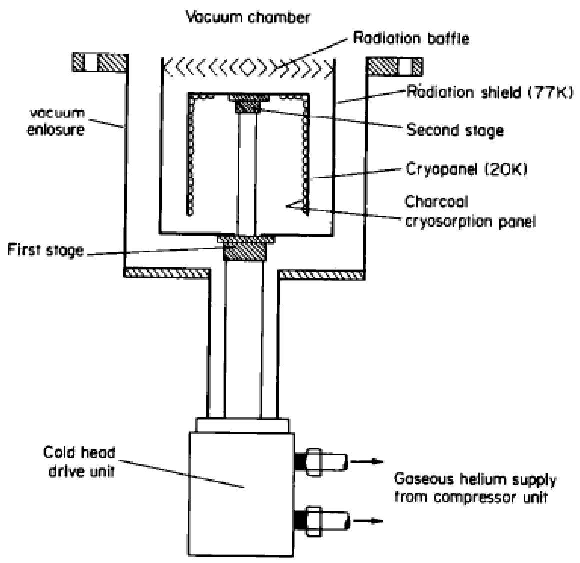
  $S_{H2} = 5.3$ l.s^{-1}.cm^{-2}

  $S_{CO} = 1.4$ l.s^{-1}.cm^{-2}

Capture factor, C_f

- The capture factor Takes into account the geometry of the system :

Baffle in a cryopump



$C_f \sim 0.3$

R. Haefer. J. Phys. E : Sci. Instrum., Vol 14, 1981, 273-288

Holes in the electron shield of the LHC beam screen

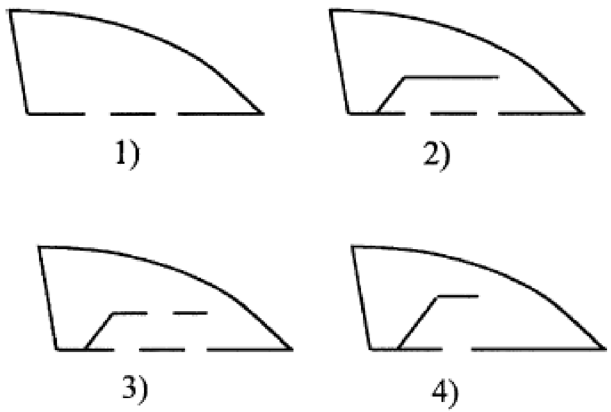


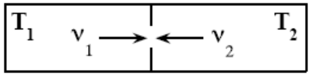
Fig. (1) Two slots in the beam screen, without electron shield, (2) two slots in the beam screen, electron shield without slot, (3) two slots in the beam screen, electron shield with slot, (4) only one slot in the beam screen, electron shield without slot.

σ	1	2	3	4
0.1	0.48	0.26	0.39	0.43
1	0.68	0.36	0.51	0.57

A.A. Krasnov. Vacuum 73 (2004) 195-199

Thermal transpiration

- Vacuum gauges are located at room temperature to reduce heat load
- For small aperture, the collision rate, ν, is conserved at the cold / warm transition



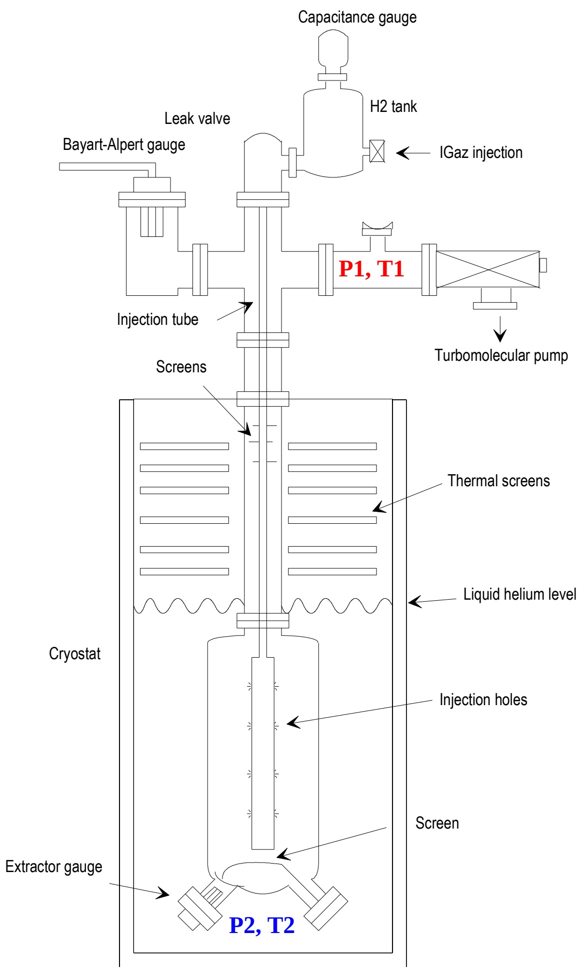
$$\nu = \frac{1}{4} n \bar{v}$$

- Since the average velocity scales like √T

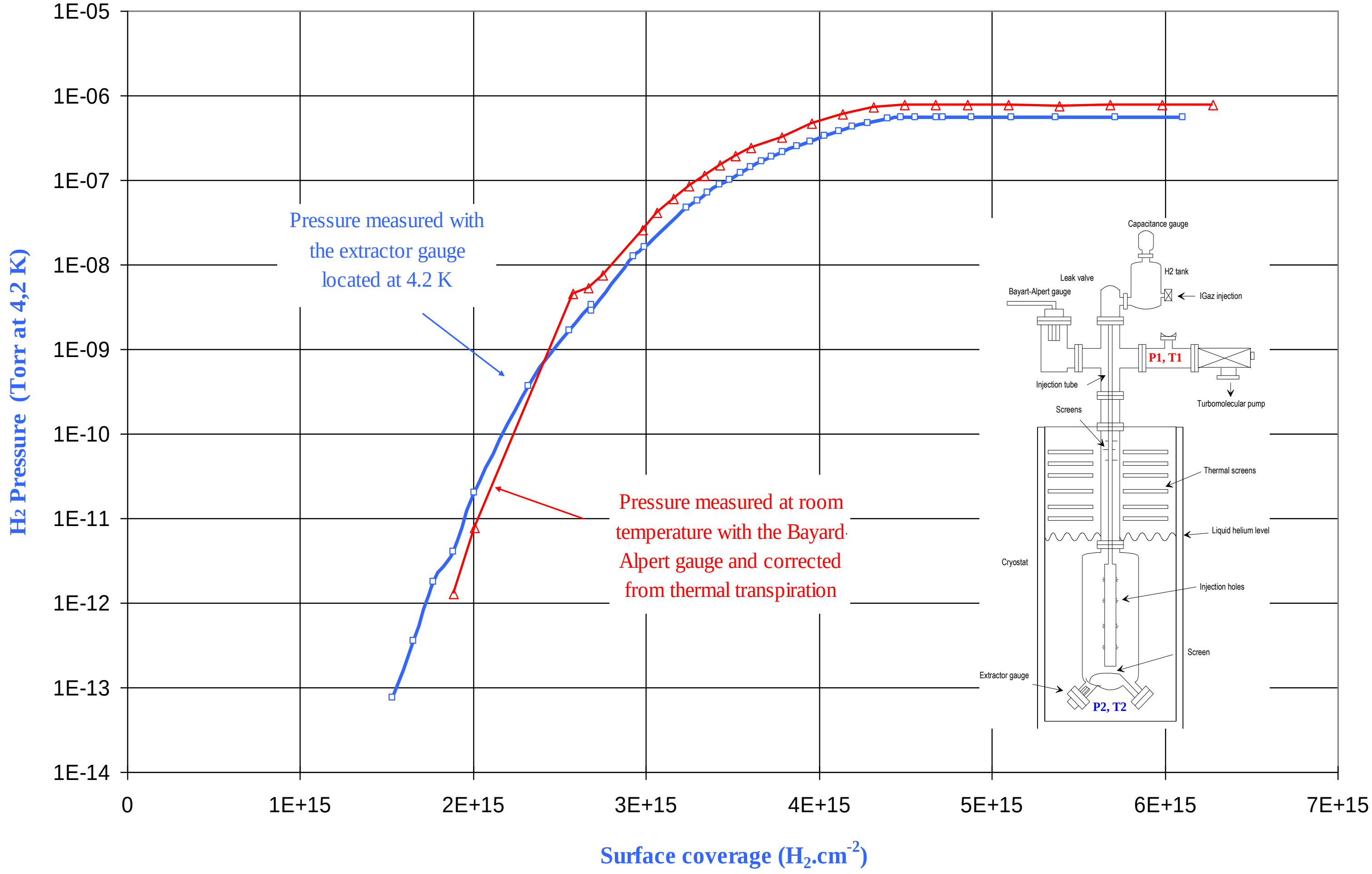
$$\frac{P_1}{P_2} = \sqrt{\frac{T_1}{T_2}}$$

$$\frac{n_1}{n_2} = \sqrt{\frac{T_2}{T_1}}$$

T (K)	4.2	77
P_1/P_2	8	2



Experimental evidence of thermal transpiration
Static conditions



V. Baglin *et al.* CERN Vacuum Technical Note 1995

2. Adsorption isotherms

Adsorption isotherm

- Measurement, at constant temperature, of the equilibrium pressure for a given gas coverage, θ
- Varies with:
  - molecular species
  - surface temperature (under 20 K only H_2 and He)
  - surface nature
  - gas composition inside the chamber
  - ...
- Models :

Henry's law for low surface coverage

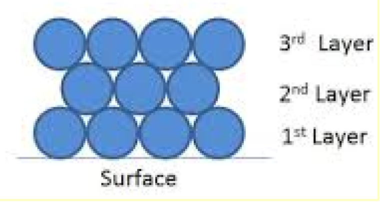
$$\theta = c\,P$$

DRK (Dubinin, Radushkevich and Kaganer) for metalic, glass and porous substrate. Valid at low pressure. Good prediction with temperature variation

$$\ln(\theta) = \ln(\theta_m) - D\left(kT\ln\left(\frac{P_{Sat}}{P}\right)\right)^2$$

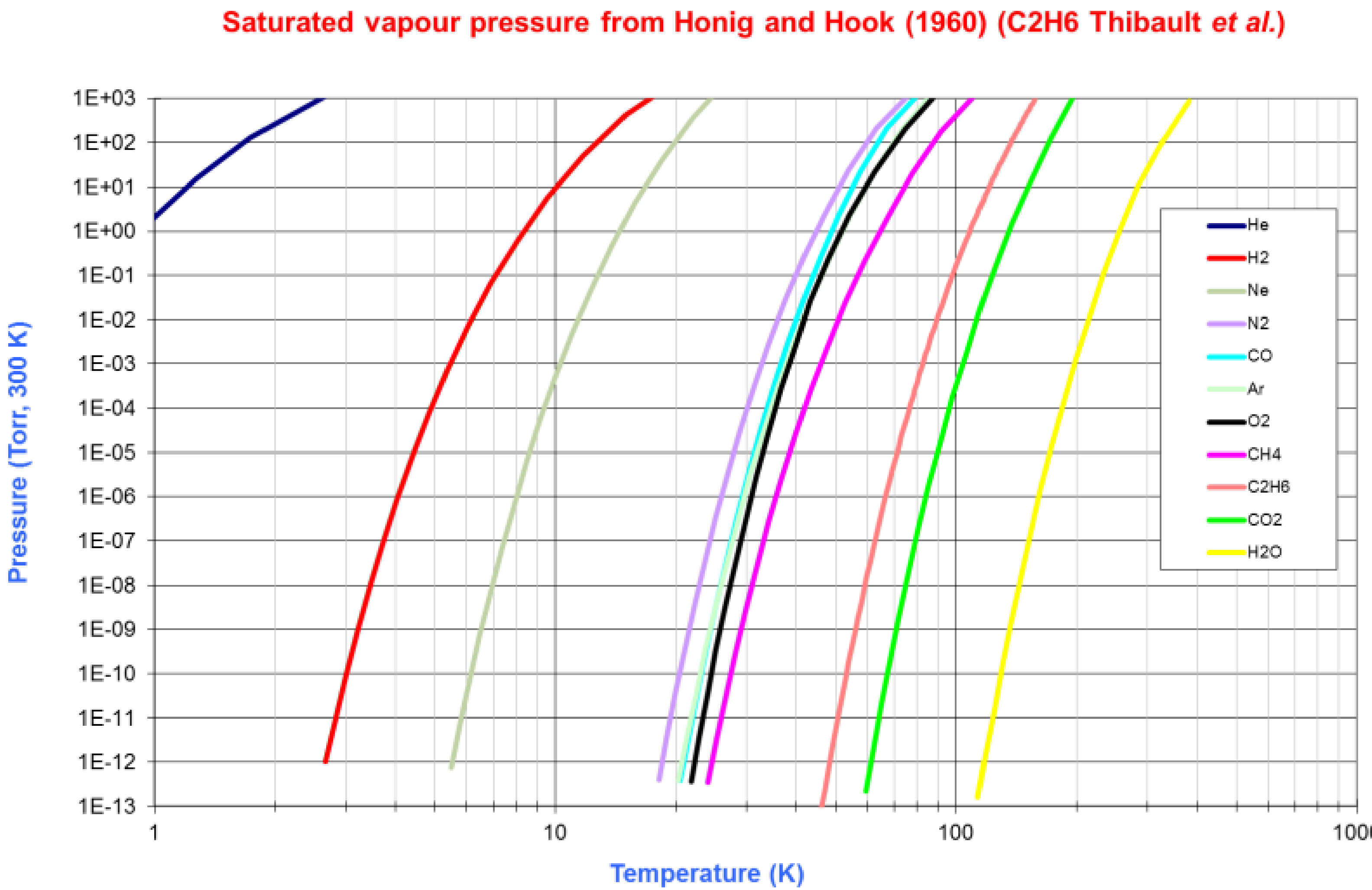
BET (Brunauer, Emmet and Teller). Multi-monolayer description

$$\frac{P}{\theta\,(P - P_{Sat})} = \frac{1}{\alpha\,\theta_m} + \frac{(\alpha - 1)}{\alpha\,\theta_m}\,\frac{P}{P_{Sat}}$$



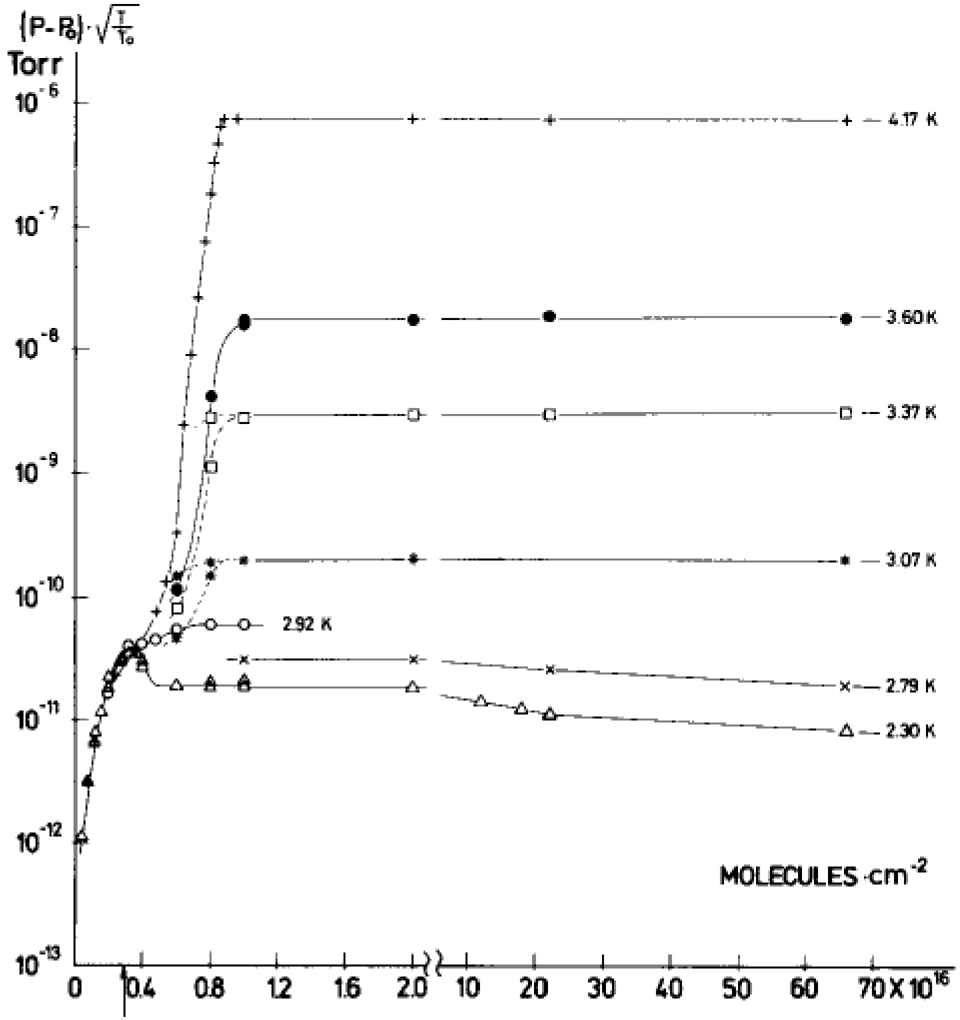
Saturated Vapor Pressure

- Pressure over liquid or gas phase (many monolayers condensed)
- Follows the Clausius-Clapeyron equation: $\log P_{sat} = A - B/T$



H_2 Adsorption Isotherm on Stainless Steel

- The vapor pressure increases when increasing the adsorption of gas up to a few monolayers (~ 10^{15} molecules/cm^2)
- The vapor pressure saturates when several monolayers of gas are adsorbed
- The pressure level of the saturation is a function of the temperature

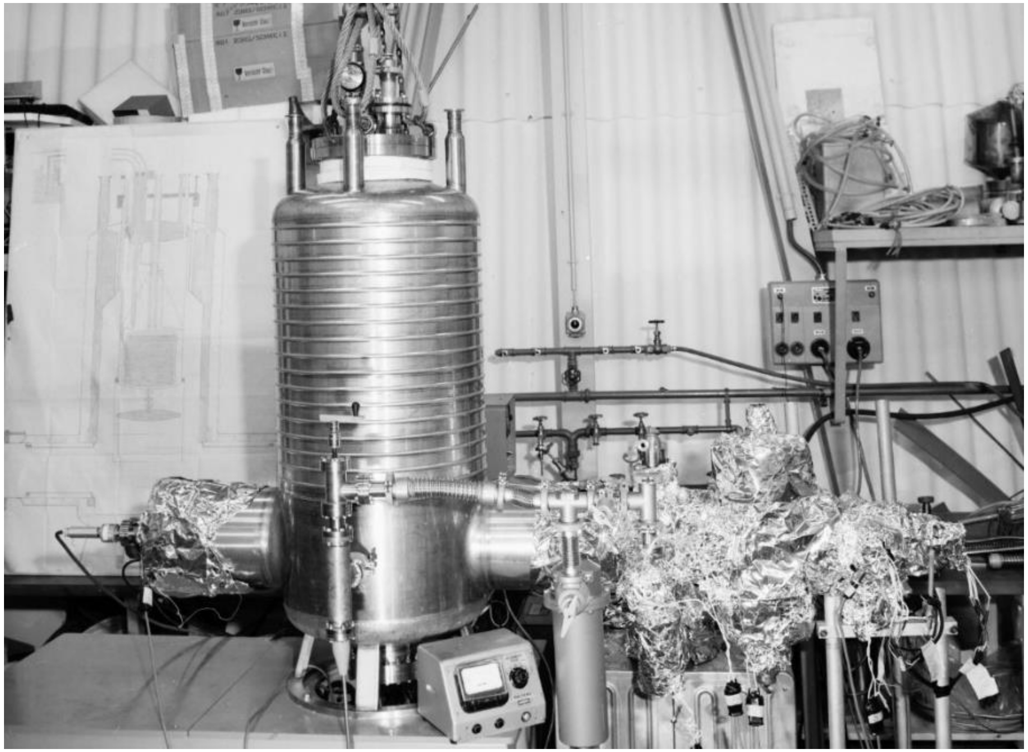


A monolayer

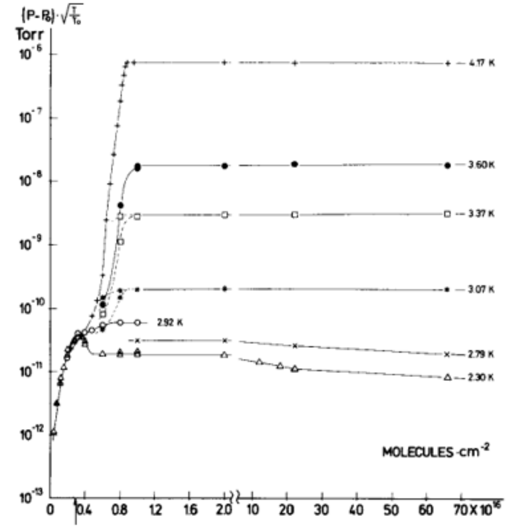
C. Benvenuti, R. Calder, G. Passardi
J.Vac.Sci. 13(6), Nov/Dec 1976, 1172-1182

H_2 adsorption isotherm on stainless steel

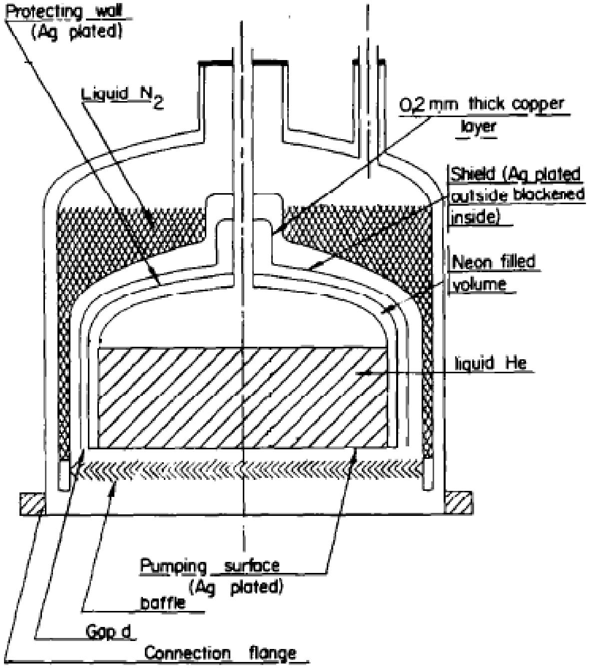
- The condensation cryopumps allows to pump large quantities of H_2
- CERN ISR condensation cryopump operated with liquid He at 2.3 K (50 Torr on the He bath)



C. Benvenuti *et al*. Vacuum, 29, 11-12, (1974) 591



A monolayer



H_2 adsorption isotherms from 8 to 20 K

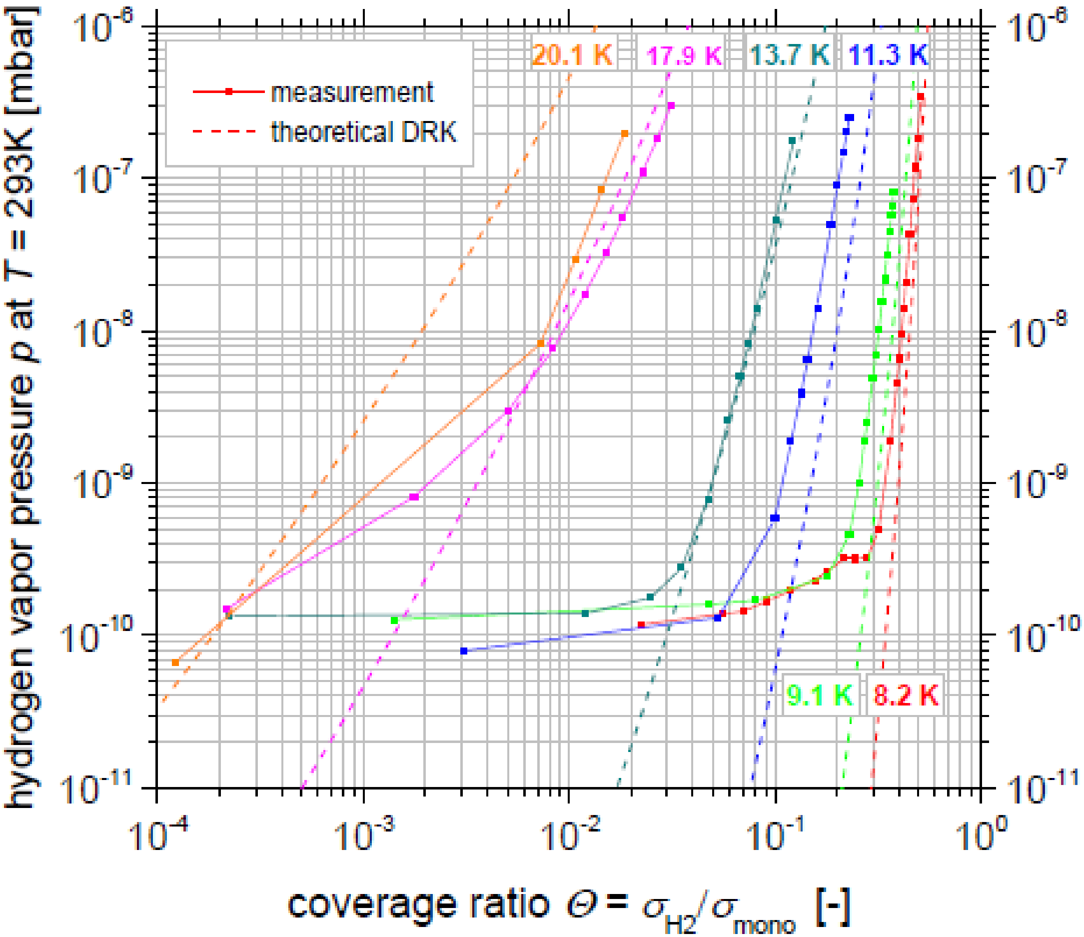
- The surface capacity strongly decreases when increasing the surface temperature

- Stainless steel

- DRK description

$$\ln(\theta) = \ln(\theta_m) - D\left(kT \ln\left(\frac{P_{Sat}}{P}\right)\right)^2$$

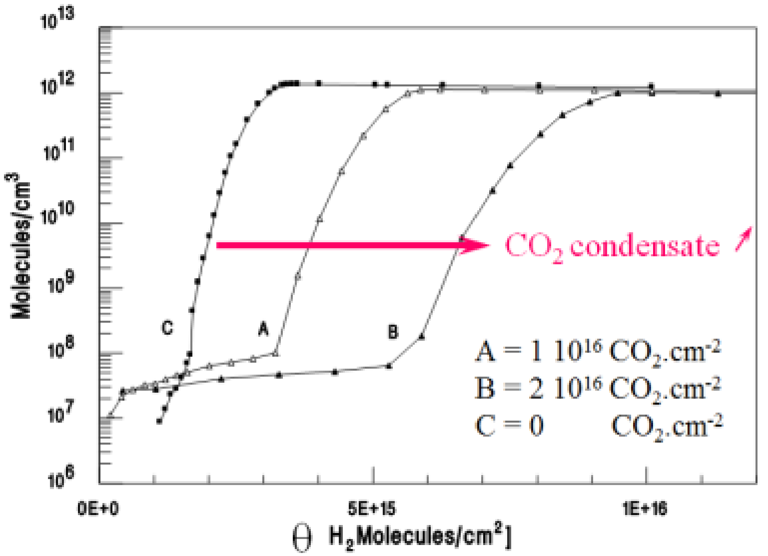
- $D = 3125$ eV^{-2}
- $\Theta_m = 7\ 10^{14}$ H_2/cm^2

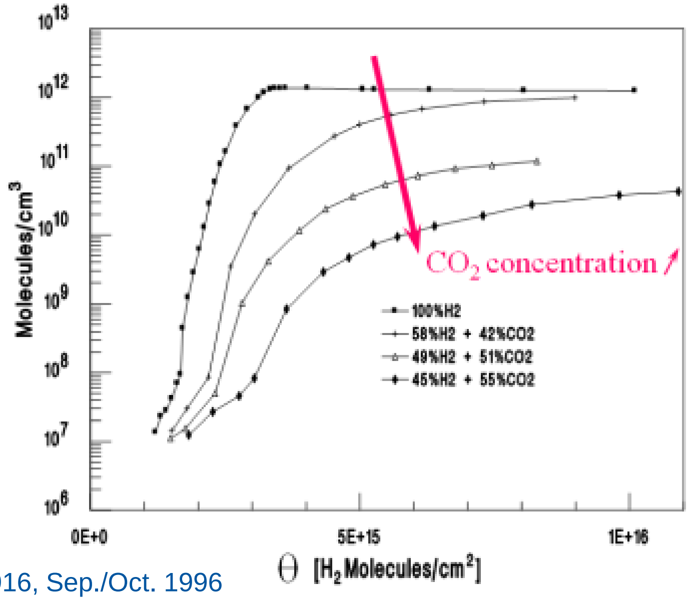


F. Chill *et al.* PAC'2015, Richmond, USA, 2015.

Vapor Pressure in a Machine

- Several types of molecules are present in machine vacuum systems
- The adsorption isotherm is affected by the presence of these molecules
- Condensed CO_2 forms a porous layer increasing the hydrogen capacity
- Co-adsorption of CH_4, CO and CO_2 reduce the vapor pressure of H_2 by cryotrapping





E. Wallén, JVSTA 14(5), 2916, Sep./Oct. 1996

➔ Studies with real machine environments are mandatory

H_2 Isotherms for Industrial Surfaces

- Identification of two categories of adsorption sites with high energy (pores, defects) and low energy (flat surface).

Table 1
Hydrogen adsorption capacity at 4.2 K

	Molecules/cm² at saturation: σ_m	Molecules/cm² at P_{sat} (10^{-6} Torr): σ_{sat}	Ratio σ_{sat}/σ_m
Smooth surfaces			
Copper film unbaked	6.07×10^{15}	1.49×10^{16}	2.45
Electrochemical buffed stainless-steel unbaked	2.36×10^{15}	4.08×10^{15}	1.73
Electrochemical buffed stainless-steel baked	2.68×10^{15}	5.22×10^{15}	1.95
TiZrV film	3.05×10^{15}	6.02×10^{15}	1.97
Porous surfaces			
Al anodised unbaked (USA)	1.23×10^{17}	—	—
Al anodised baked (USA)	1.80×10^{17}	—	—
Al anodised (KEK)	8.1×10^{16}	1.18×10^{17}	1.46

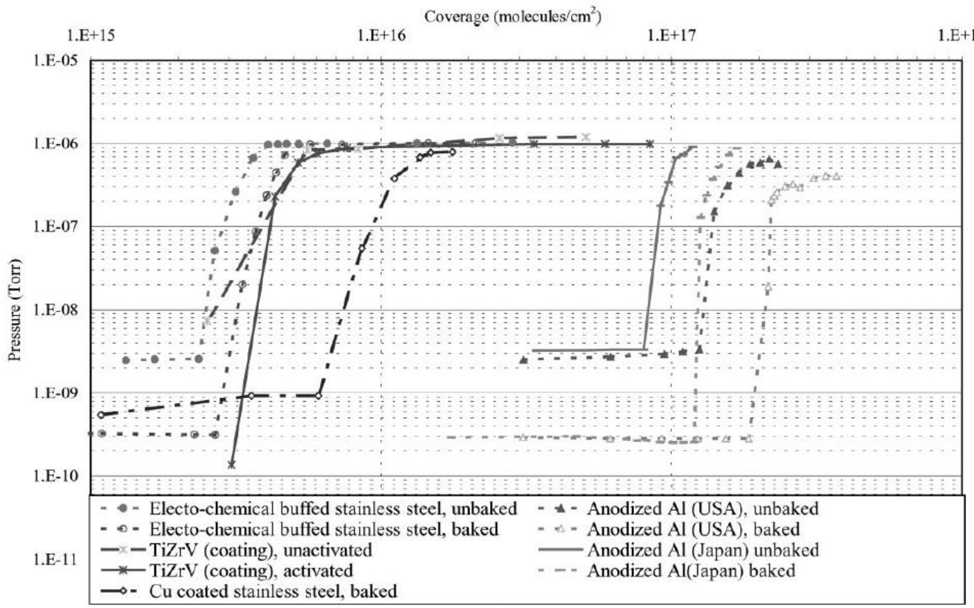


Fig. 3. Hydrogen adsorption isotherm at 4.2 K for various samples.

G. Moulard, B. Jenniger, Y. Saito, Vacuum 60 (2001) 43-60

Cryosorbing materials

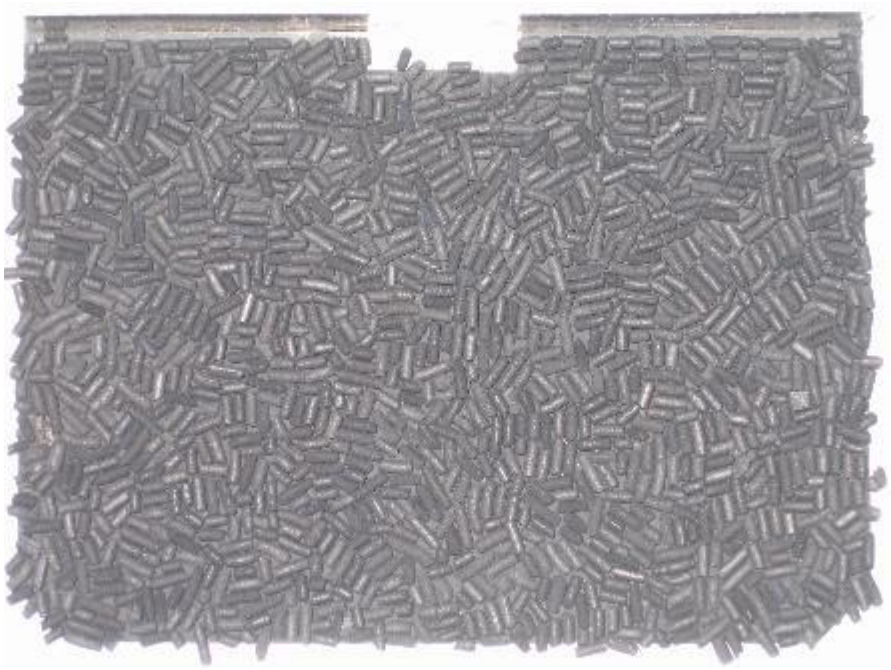
- Large capacity
- Large pumping speed
- Large temperature working range (up to ~ 30 K)

e.g. Activated Charcoal used for cryopumps

Capacity ~ 10^{22} H_2/g i.e. 10^{21} monolayers (P. Redhead, Physical basis of UHV,1968)

Sticking coefficient ~ 30 % at 30 K (T. Satake, Fus. Tech. Vol 6., Sept. 1984)

20 K cryopanels



B.E.T surface area

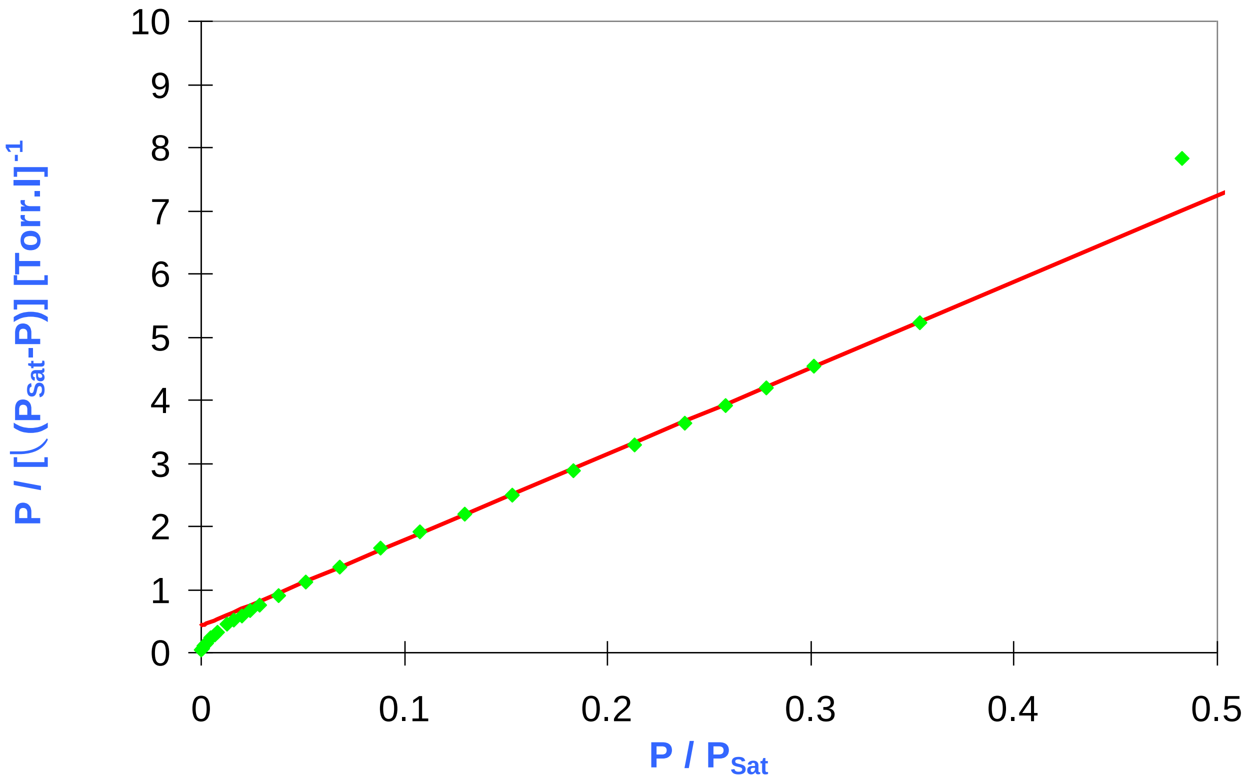
- Xe is an inert gas which can only be physisorbed on a surface
- Xe adsorption isotherms at 77 K are used to derive the roughness factor of surface using the BET multi-monolayer theory

$$\frac{P}{\theta\left(P - P_{Sat}\right)} = \frac{1}{\alpha\ \theta_m} + \frac{(\alpha - 1)}{\alpha\ \theta_m}\frac{P}{P_{Sat}} \approx \frac{1}{\theta_m}\frac{P}{P_{Sat}}$$

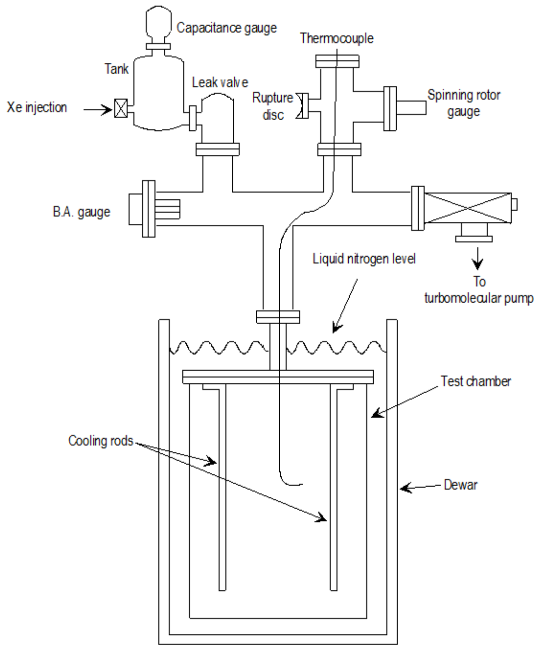
- Valid for $0.01<P/P_{sat}<0.3$
- BET monolayer = θ_m
- $\alpha = \exp(\Delta E/kT) >> 1$

$$R = \frac{A_R}{A_G} = \frac{A \times \theta_m}{A_G}$$

A for Xenon ~ 25 Å^2

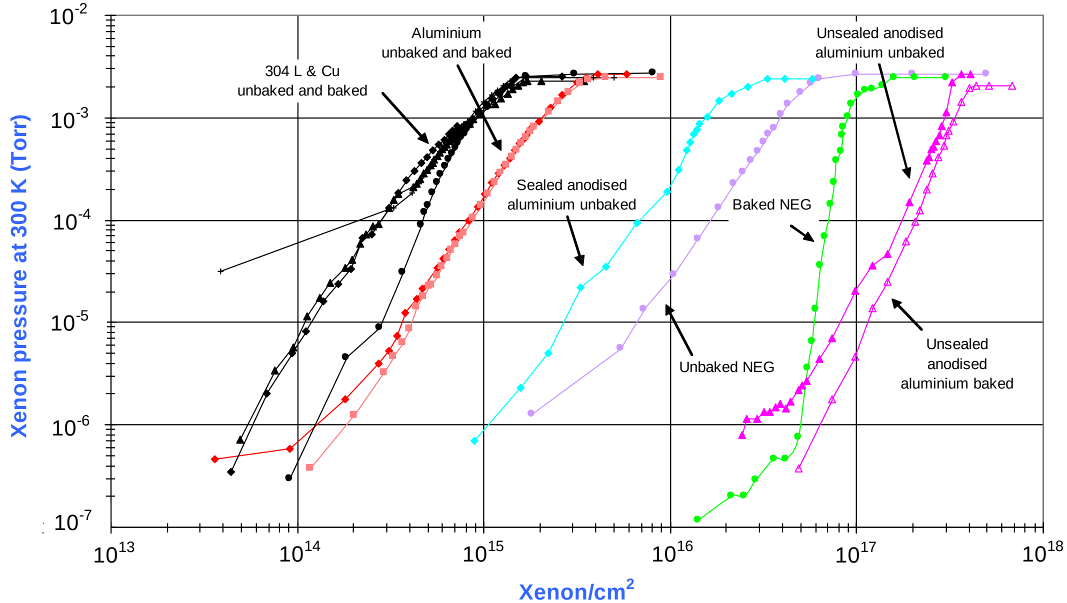


BET set-up


Capacitance gauge
Thermocouple
Tank
Leak valve
Xe injection
Rupture disc
Spinning rotor gauge
B.A. gauge
Liquid nitrogen level
To turbomolecular pump
Test chamber
Cooling rods
Dewar

Roughness factor

- Xe saturated vapour pressure ~ 2.6 10^{-3} Torr at 77 K



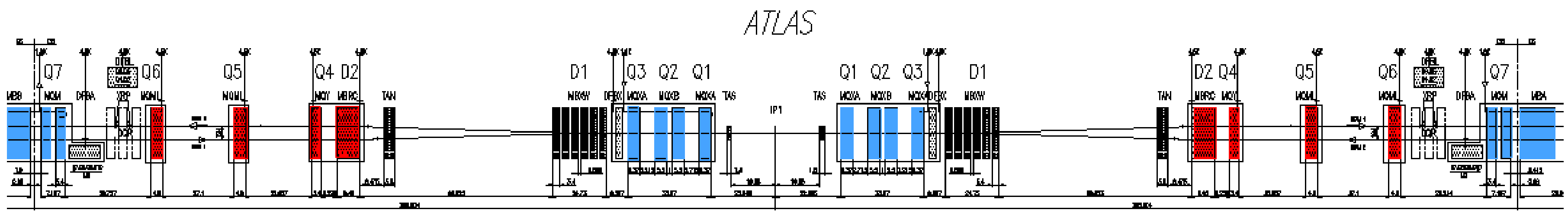
Technical surface	Unbaked	Baked at 150 ºC
Copper Cu-DHP acid etched	1,4	1,9
Stainless steel 304 L vacuum fired	1,3	1,5 (at 300 ºC)
Aluminium degreased	3,5	3,5
Sealed anodised aluminium 12 V	24,9	not measured
Unsealed anodised aluminium 12 V	537,5	556,0
NEG St 707	70,3	156,3

V. Baglin. CERN Vacuum Technical Note 1997

3. Cryosorbers in cold systems.

Case of the LHC superconducting magnets operating at 4.5 K

LHC Long straight section vacuum system



- Focusing inner triplets located around experiments operate at 1.9 K
- Matching sections operate at 4.5 K

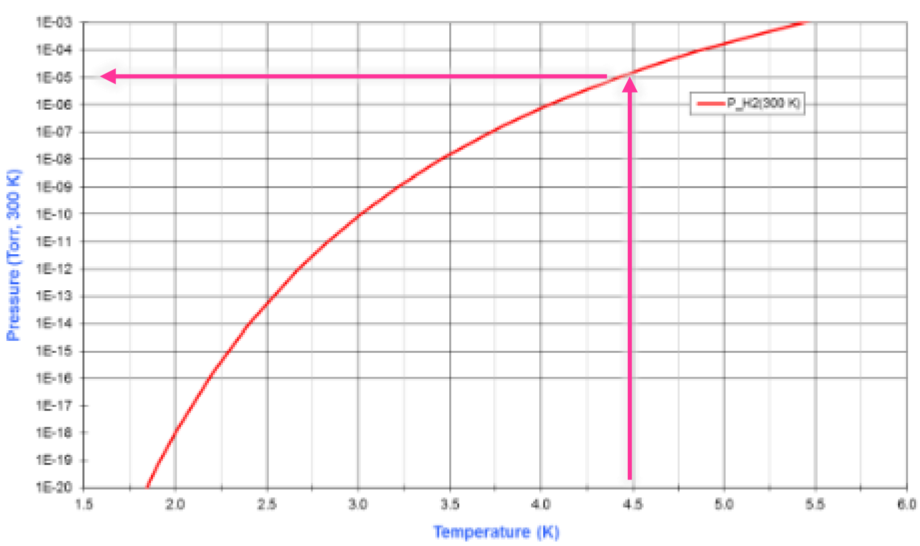
Perforated beam screens

- 1.9 K cold bore (~660 m, arc beam screen technology)
- ~ 4.5 K cold bore (~ 740 m)

With a 4.5 K Cold Bore

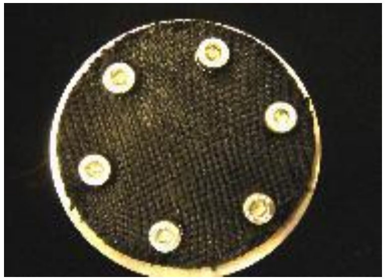
- Saturated vapour pressure equals 2 10^{-5} mbar
- Cryosorbers are needed to provide a porous surface

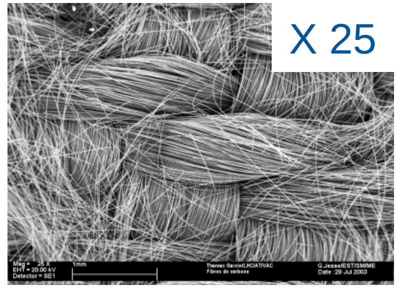
Hydrogen saturated vapour pressure from Honig and Hook (1960)

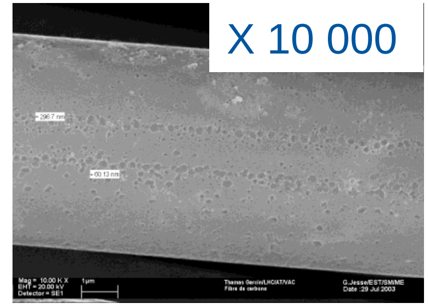


H_2 adsorption isotherm on cryosorbers

- Woven carbon fibers are used in LHC as cryosorbers in 4.5 K magnets
- Beam screen operates in the 5-20 K range





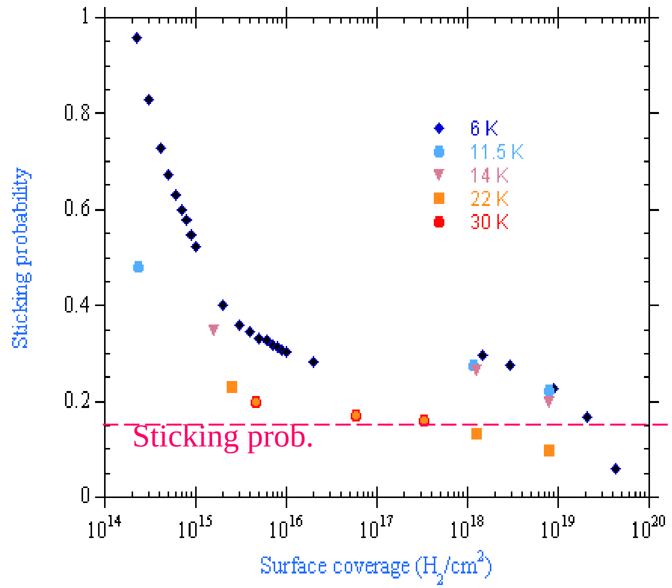


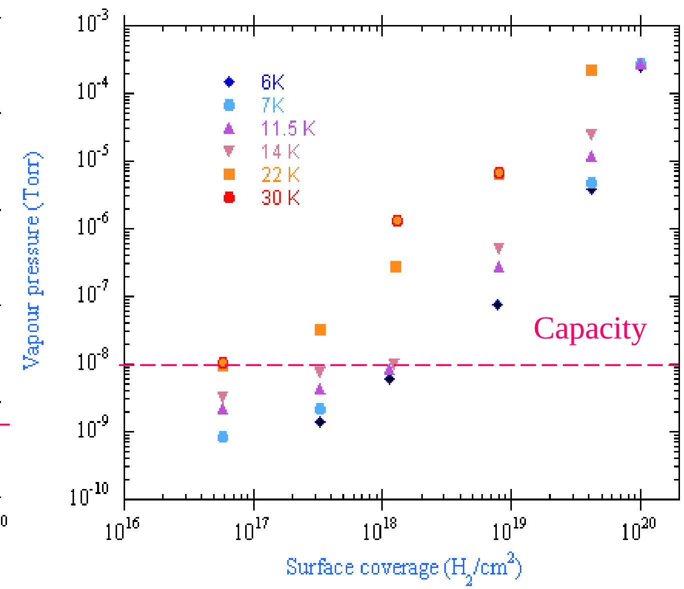
V. Anashin *et al.* Vacuum 75 (2004) 293-299

- Sticking probability at 10^{18} H_2/cm^2 :
  - 15 % at 22 K
  - > 15 % below 22 K

- Capacity at 10^{-8} mbar :
  - 10^{18} H_2/cm^2 at 6 K
  - 10^{17} H_2/cm^2 at 30 K

$R \sim 10^3 R_{Cu}$





V. Baglin *et al.* EPAC'04, Luzern 2004.

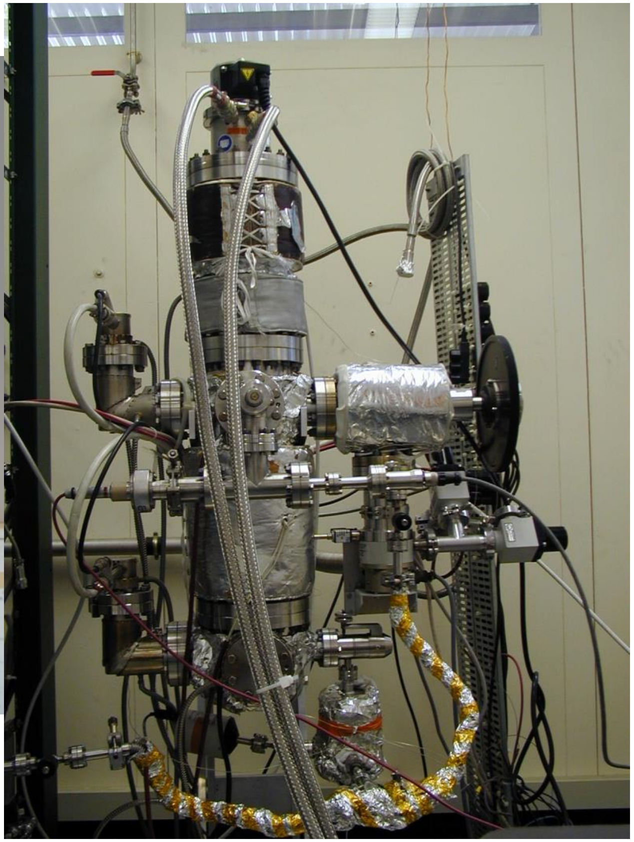
Fisher-Mommsen set Up

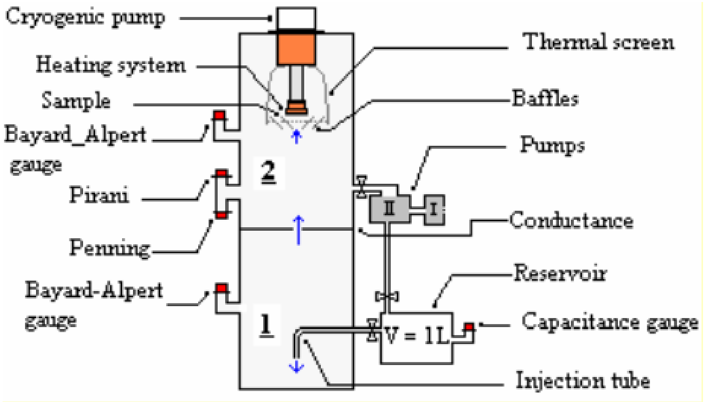
- The pumping speed is measured during the gas injection

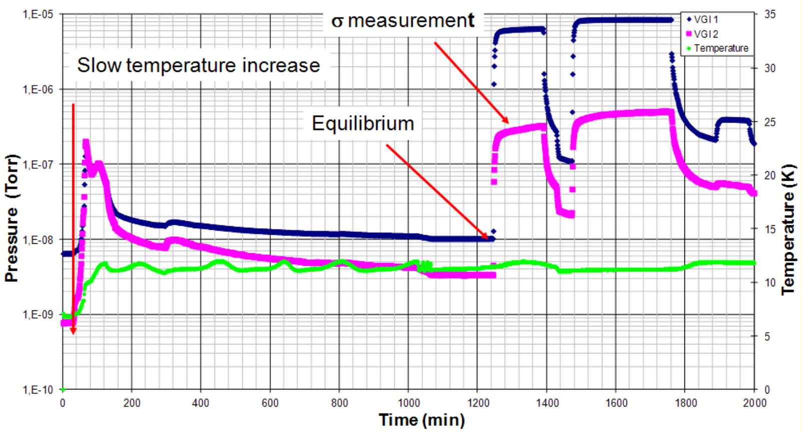
$$S = \frac{C\,(\Delta P_1 - \Delta P_2)}{\Delta P_2} \qquad \sigma = \frac{S}{S_{max}}$$

- The vapor pressure is measured after injection, when dP/dt = 0

C = 10.5 l/s for H_2

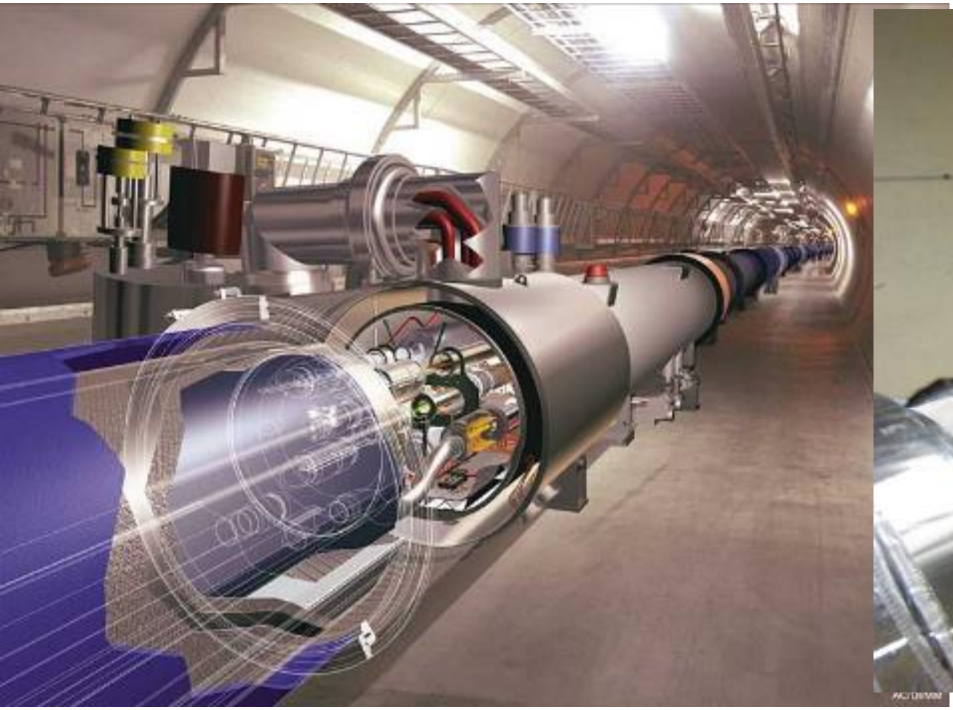


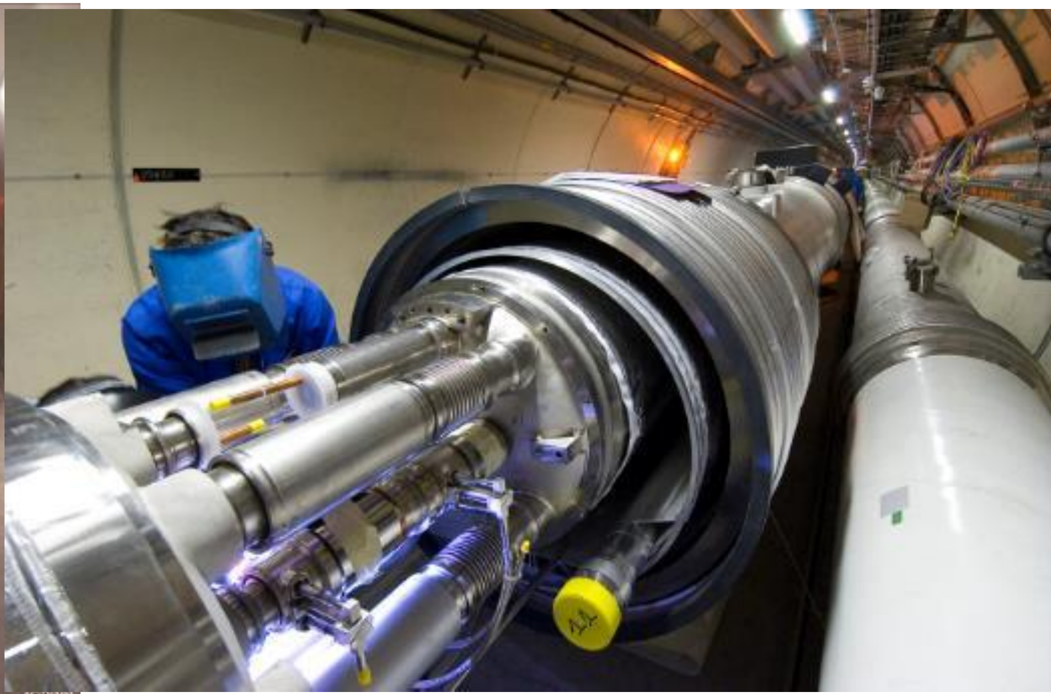




4. He leaks in cold systems

LHC : Superconducting technology

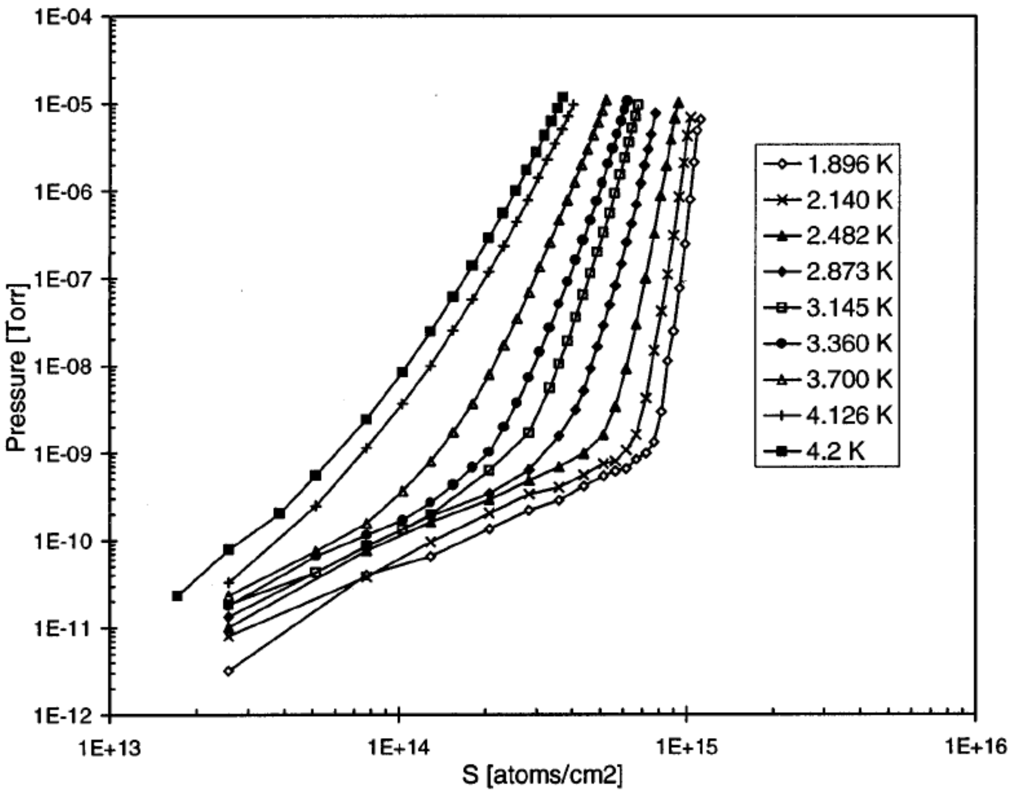




- Air leak or He leaks could appear in the beam tube during operation : the consequences are risk of magnet quench, pressure bump and radiation dose

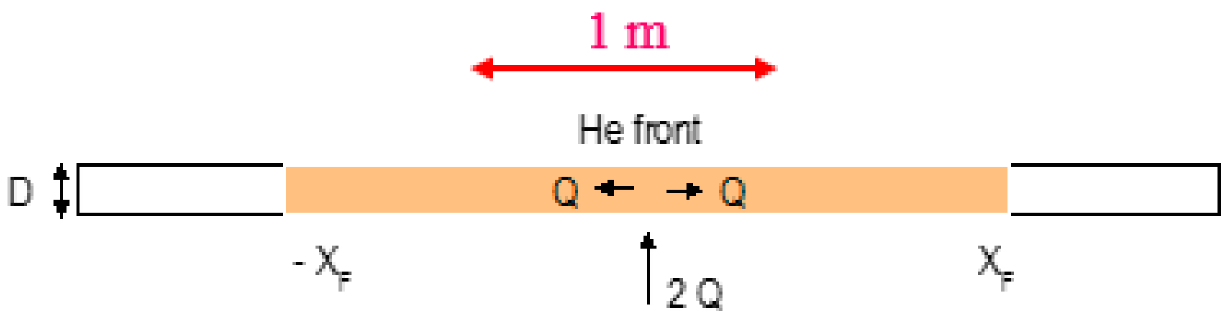
He adsorption isotherm from 1.9 to 4.2 K

- Sub-monolayer range
- Approaches Henry's law at low coverage
- The isotherms are well described by the DRK model
- θ_m ~ 1.3 1015 H2/cm2
- Stainless steel

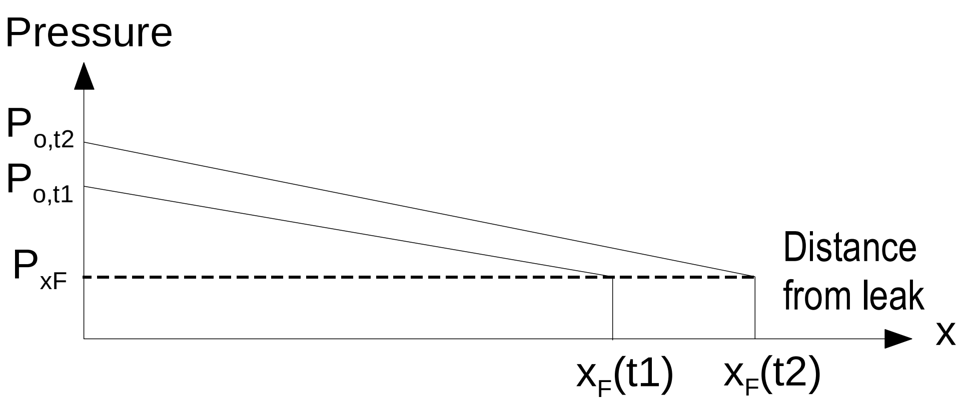


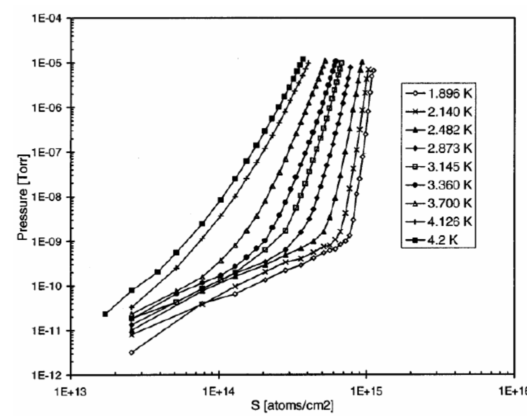
E. Wallén. J.Vac.Sci.A 15(2), Mar/Apr 1997, 265-274.

He leaks at 1.9 K



P. Hobson *et al*. J.Vac.Sci. A. 11(4), Jul/Aug 1993, 1566-1573





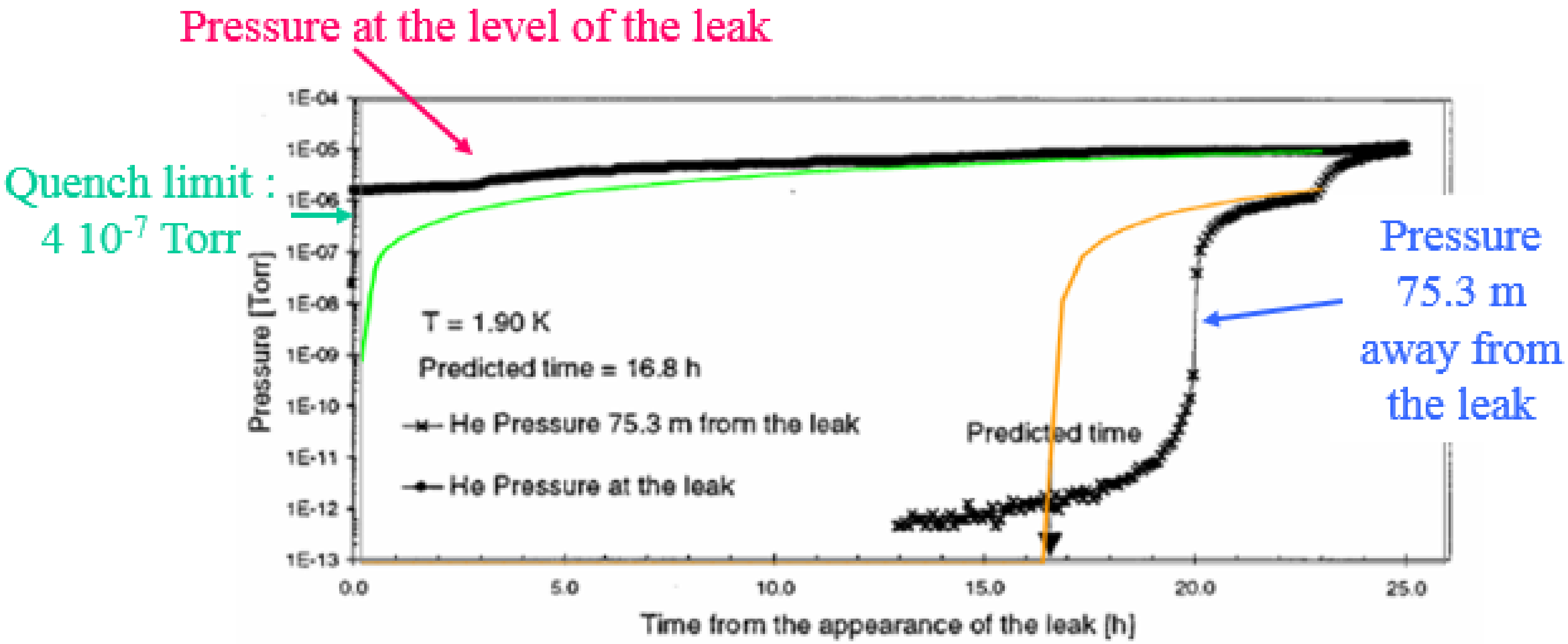
E. Wallén. J.Vac.Sci.A 15(2), Mar/Apr 1997, 265-274.

- A He pressure wave is developed with time along the beam vacuum chamber
- The He wave can span over several tens of meter without being detected
- The local pressure bump gives a local proton loss (risk of quench)

Example: LHC test string

Example : LHC Test string
Leak rate 6 10^{-5} Torr.l/s
Distance 75.3 m

20h to be detected 75 m downstream!



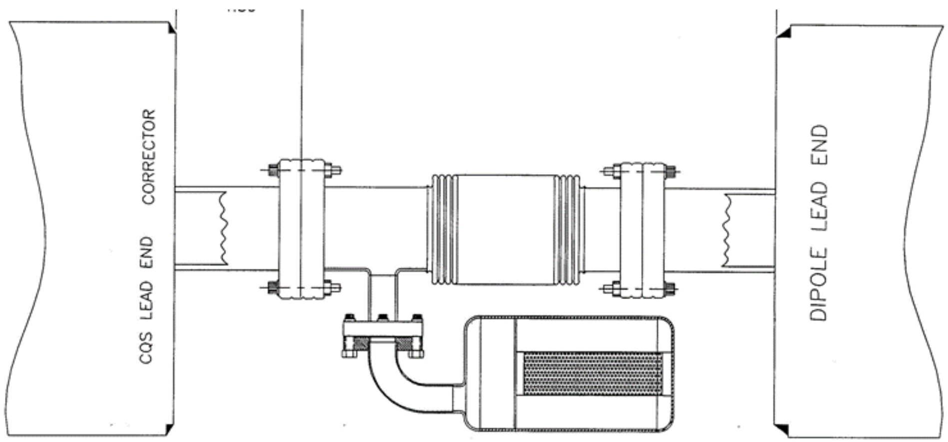
E. Wallén, JVST A 15(6), Nov/Dec 1997

RHIC, Brookhaven, USA

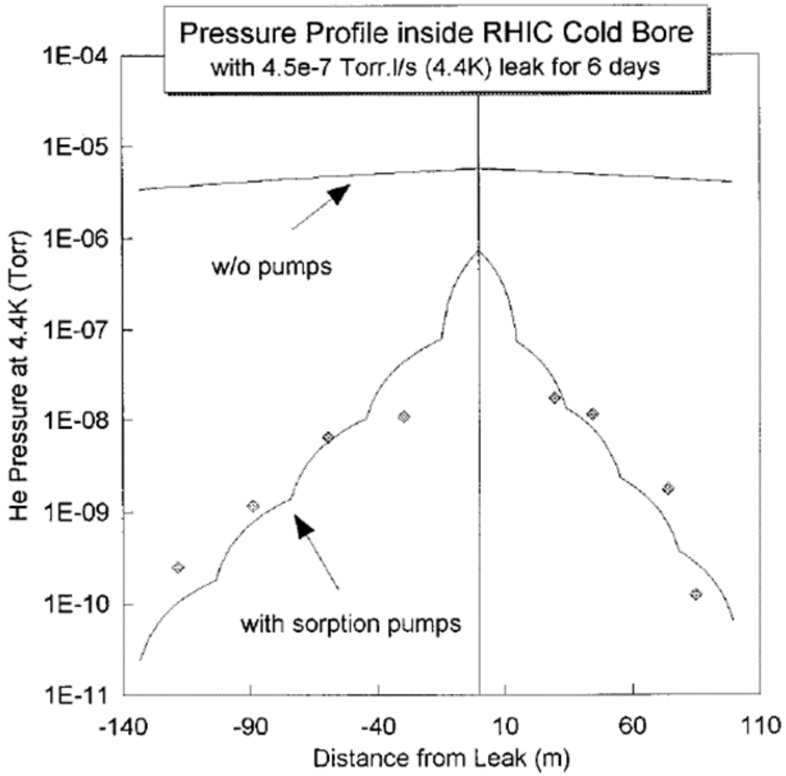
- RHIC's solution: anticipate leaks !!
- RHIC use sorption pumps based on 300 g of activated charcoal.
- They are located every 30 m to mitigate He leaks and to pump H_2

Test in a 480 m long sector at 4.4 K

RHIC interconnect



H.C. Hseuh, Proc. PAC 1999



H.C. Hseuh, E. Wallén.
J.Vac.Sci.A 16(3), May/Jun 1998, 1145-1150.

Lecture 3 summary

- Gas can be physisorbed for very long period on cryogenic surface
- The sticking coefficient characterise the pumping speed of a surface
- The capture coefficient characterise the pumping speed of a device
- At cryogenic temperature, thermal transpiration correction shall be applied
- The vapour pressure is the equilibrium pressure as a function of gas coverage
- When saturated (many monolayers), the vapour pressure follows the Clausius Clapeyron law
- Adsorption isotherms vary very much with the conditions
- Some material can be porous so to adsorb many monolayers of gas without reaching the saturated vapour pressure: cryosorbers
- He leak can be difficult to detected at cryogenic temperature

Some References

- Cern Accelerator School, Vacuum technology, CERN 99-05
- Cern Accelerator School, Vacuum in accelerators, CERN 2007-03
- Cern Accelerator School, Vacuum for particle accelerators, Glumslov, June 2017
- The physical basis of ultra-high vacuum, P.A. Redhead, J.P. Hobson, E.V. Kornelsen. AVS.
- Capture pumping technology, K. Welch, North Holland.
- Cryopumping, theory and practice, R. Haefer, R. Clarendon press

Some Journals Related to Vacuum Technolgy

- Journal of vacuum science and technology
- Vacuum

Thank you for your attention !!!

CERN

Complementary information

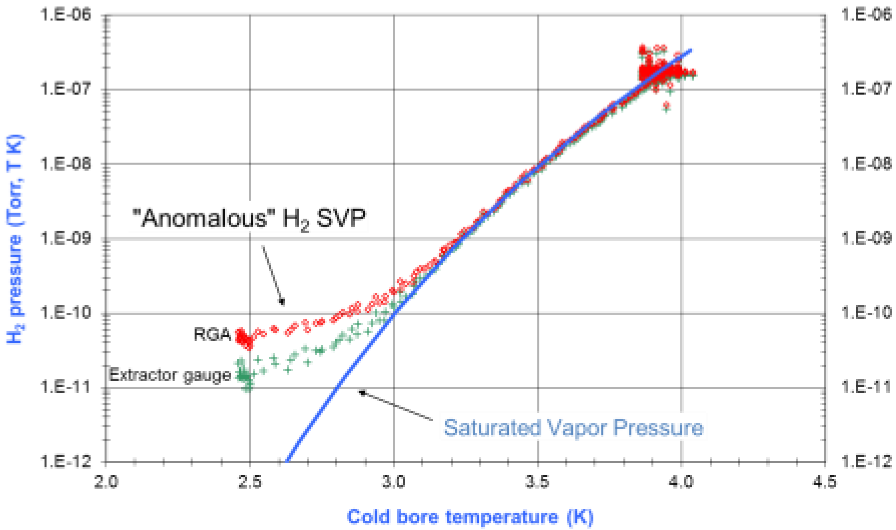
2. Adsorption isotherms

"Anomalous" Saturated Vapor Pressure

Complementary information

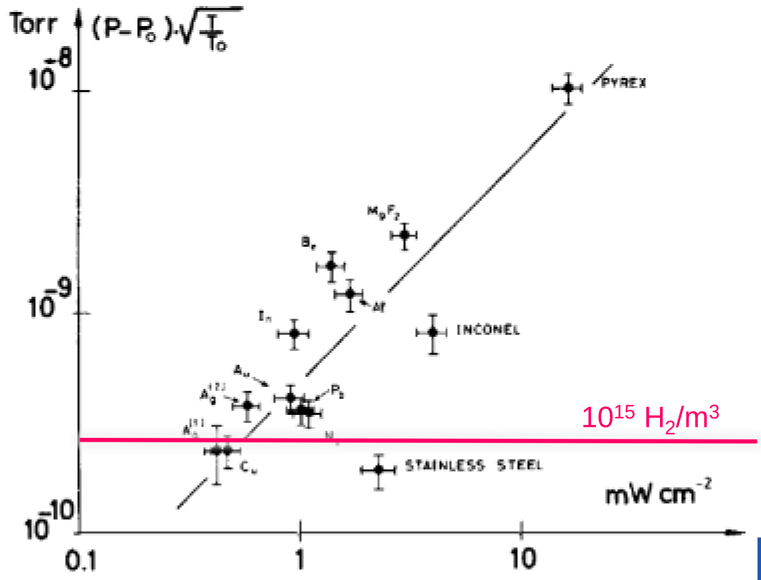
Thermal radiation induced desorption:

- <u>In a "LHC type" mock-up (COLDEX):</u>
- After condensation of 10 monolayers of H_2, the pressure follows the Clausius-Clapeyron equation while the cold bore temperature is decreased from 4 to 3 K
- Below 3 K, a deviation is observed due to the thermal radiation coming from the room temperature parts located at the extremities of the 2 m long system.
- Increasing the beam screen temperature from 20 K to 100 K has no impact on the observed deviation while the cold bore is held at 2.7 K



V. Baglin, B. Jenninger, COLDEX Run 24, September 1999

- <u>Cryopump optimisation:</u>
- 10 monolayers of H_2 is condensed at 2.3 K
- The different cryosurface types are fully exposed to 300 K radiation
- Linear dependence with the absorbed power (incident radiation x substrate emissivity)
- The pressure, measured at 2.3 K, varies from 10^{-10} to 10^{-8} Torr => gas density 5 10^{14} to 5 10^{16} H_2/m^3



C. Benvenuti, R. Calder, G. Passardi J.Vac.Sci. 13(6), Nov/Dec 1976, 1172-1182

H_2 adsorption isotherm at 4.2 K on CO_2 condensat

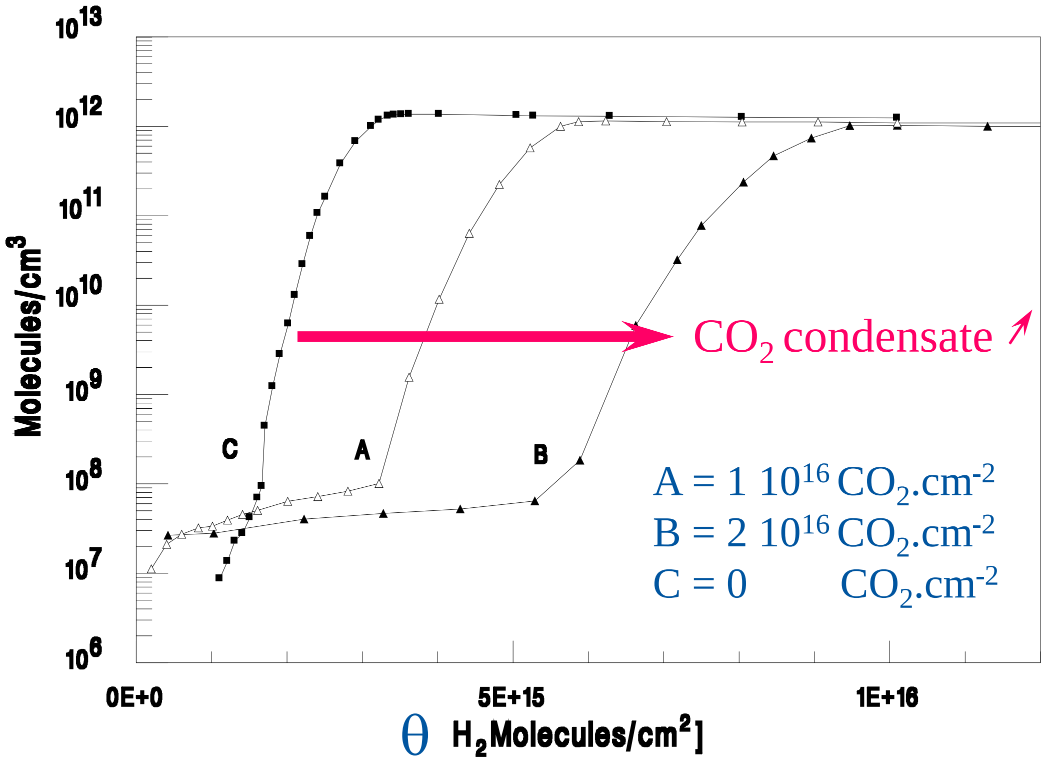
Complementary information

- Growth in packing form for CO_2 films

  ➔ Porous layer

- DRK adsorption capacity :
  0.3 H_2/CO_2

- Electroplated Cu



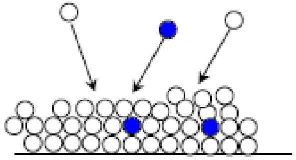
E. Wallén, JVSTA 14(5), 2916, Sep./Oct. 1996

H_2 adsorption isotherm at 4.2 K in co-adsorption with CO_2

Complementary information

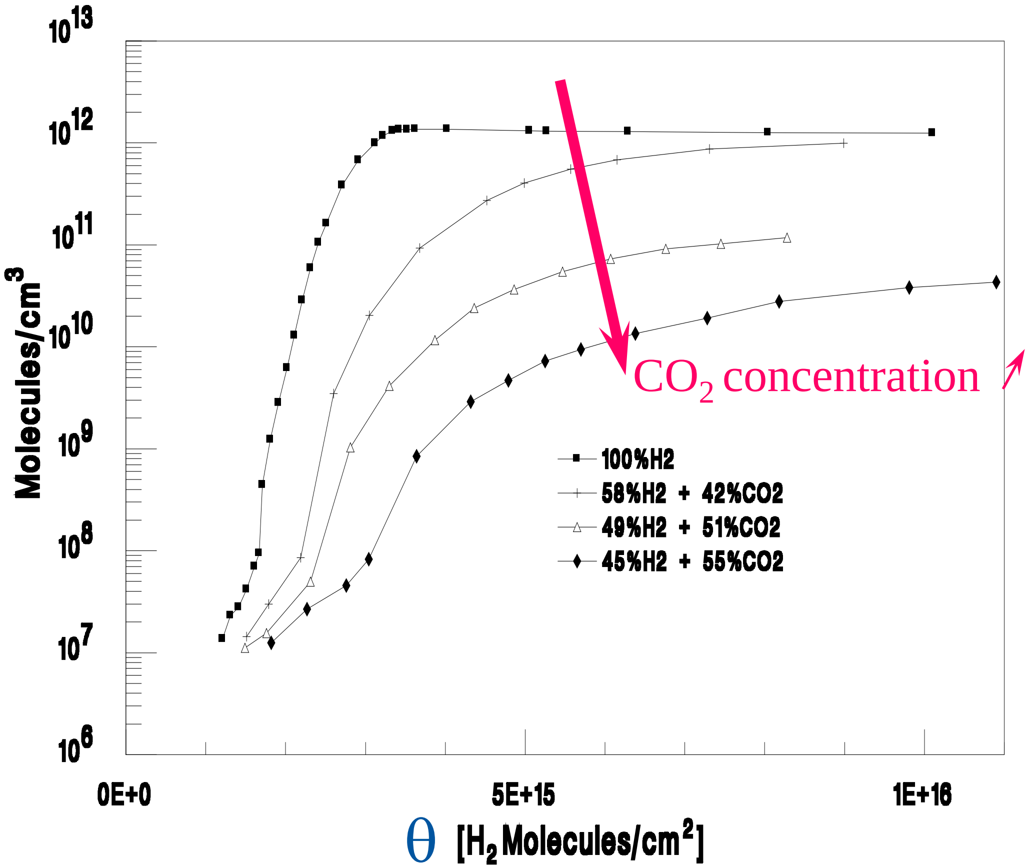
- Reduction of the saturated vapour pressure by orders of magnitude

➔ Cryotrapping



- Electroplated Cu

- In cryopumps CO_2 is admitted to enhance the pumping of H_2 and He



E. Wallén, JVSTA 14(5), 2916, Sep./Oct. 1996

CO_2 adsorption Isotherm at 77 K

Complementary information

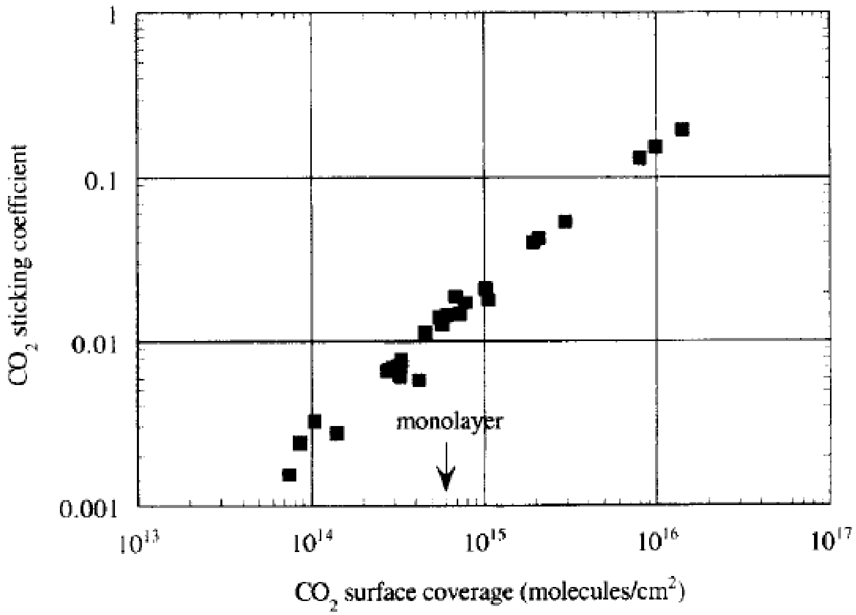


Figure 2. The sticking coefficient for CO_2 at 77 K as a function of the surface coverage.

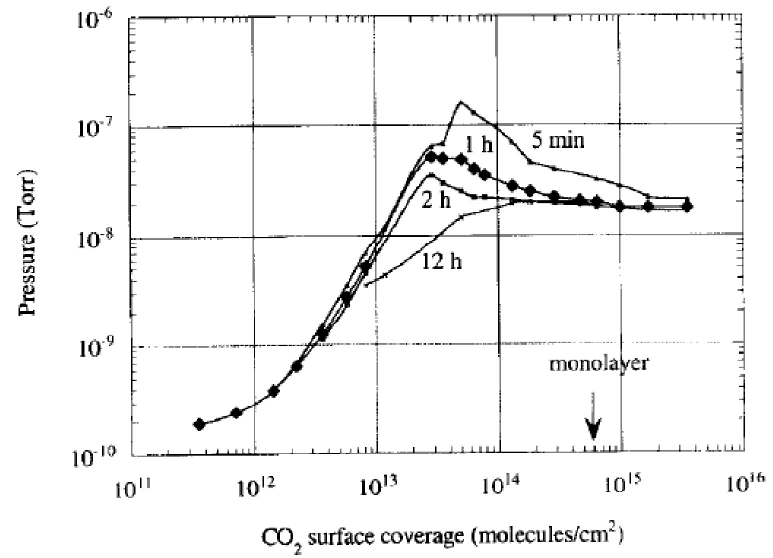


Figure 3. Adsorption isotherm for CO_2 at 77 K as a function of surface coverage. The curves refer to measurements for different waiting times for pressure stabilisation.

V.V Anashin et al, Vacuum 48 (1997) 785-788

- Metallic surface
- Below a monolayer, the equilibrium pressure of the isotherm is obtained after several hours
- Due to the low sticking coefficient and the molecular adsorption by cluster.

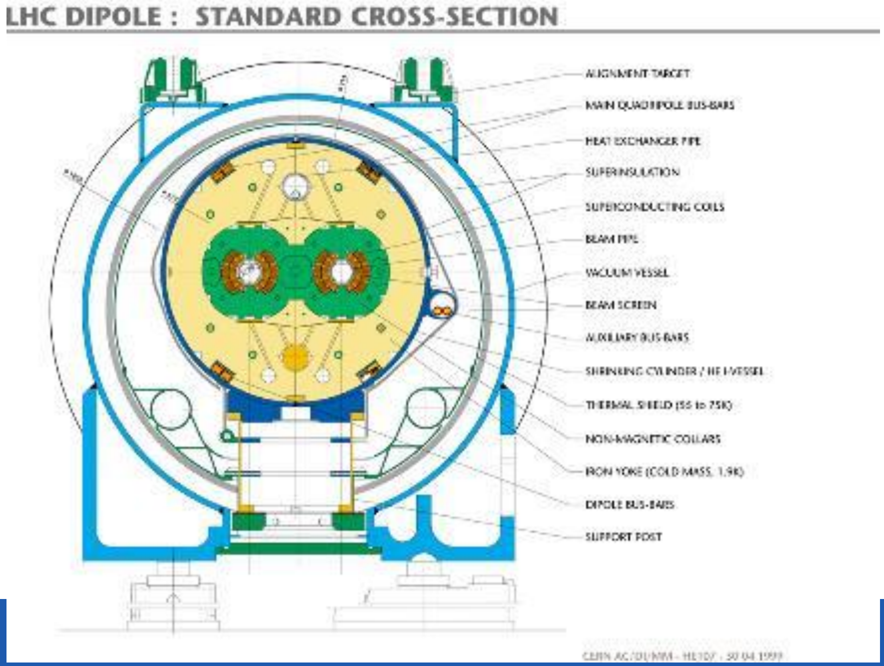
3. Cryosorbers in cold systems.

Case of the LHC superconducting magnets operating at 4.5 K

LHC dipole vacuum system

Complementary information

- Cold bore (CB) at **1.9 K**
- Beam screen (BS) at 5-20 K (intercept thermal loads)



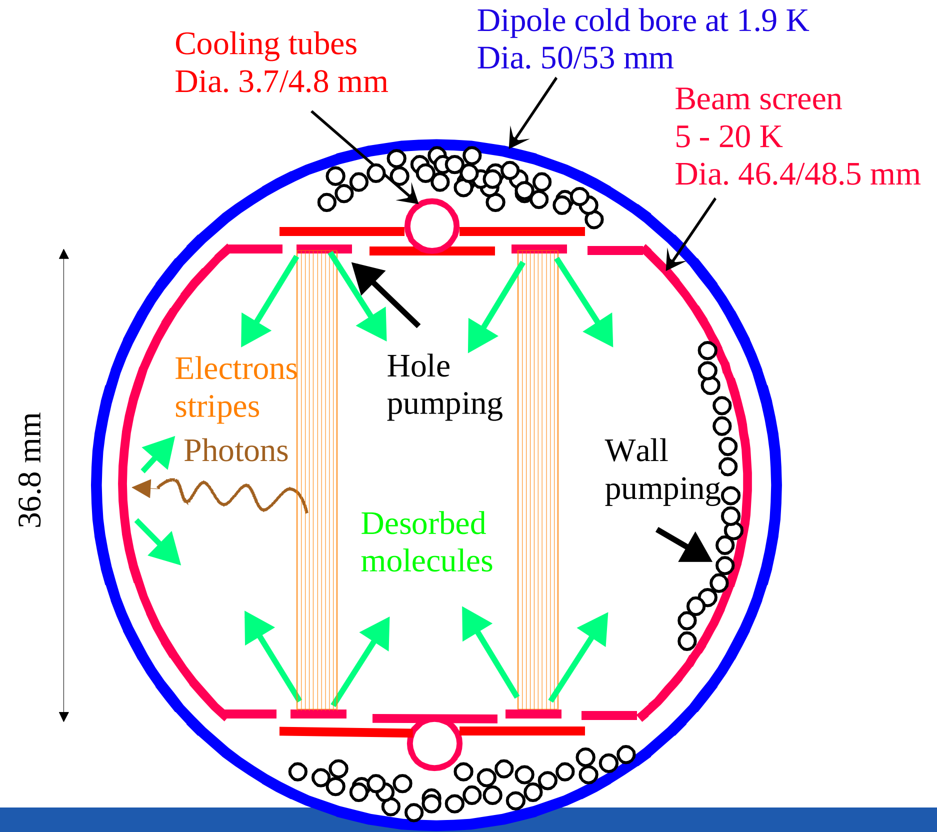
LHC vacuum system principle

Complementary information

- Molecular desorption stimulated by photon, electron and ion bombardment
- Desorbed molecules are pumped on the beam vacuum chamber
- 100 h beam life time (nuclear scattering) equivalent to ~ 10^{15} H_2/m^3 (10^{-8} Torr H_2 at 300 K)

In cryogenic elements

- Molecular physisorption onto cryogenic surfaces (weak binding energy)
- Molecules with a low recycling yield are first physisorbed onto the beam screen (CH_4, H_2O, CO, CO_2) and then onto the cold bore
- H_2 is physisorbed onto the cold bore

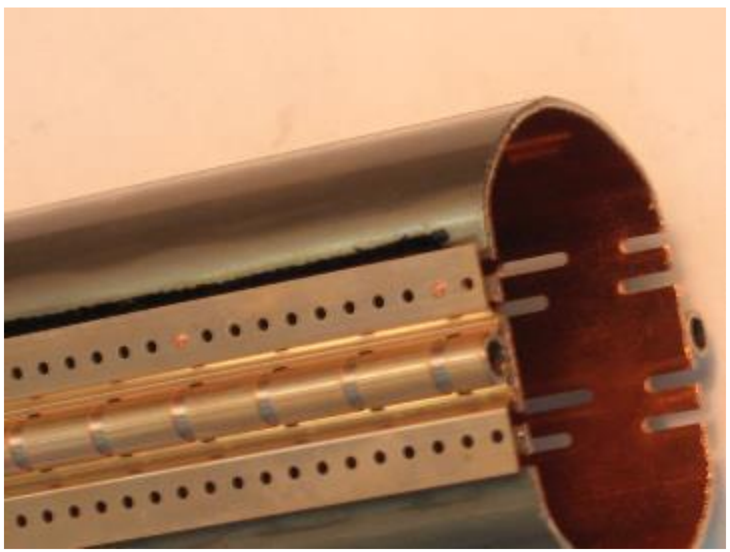


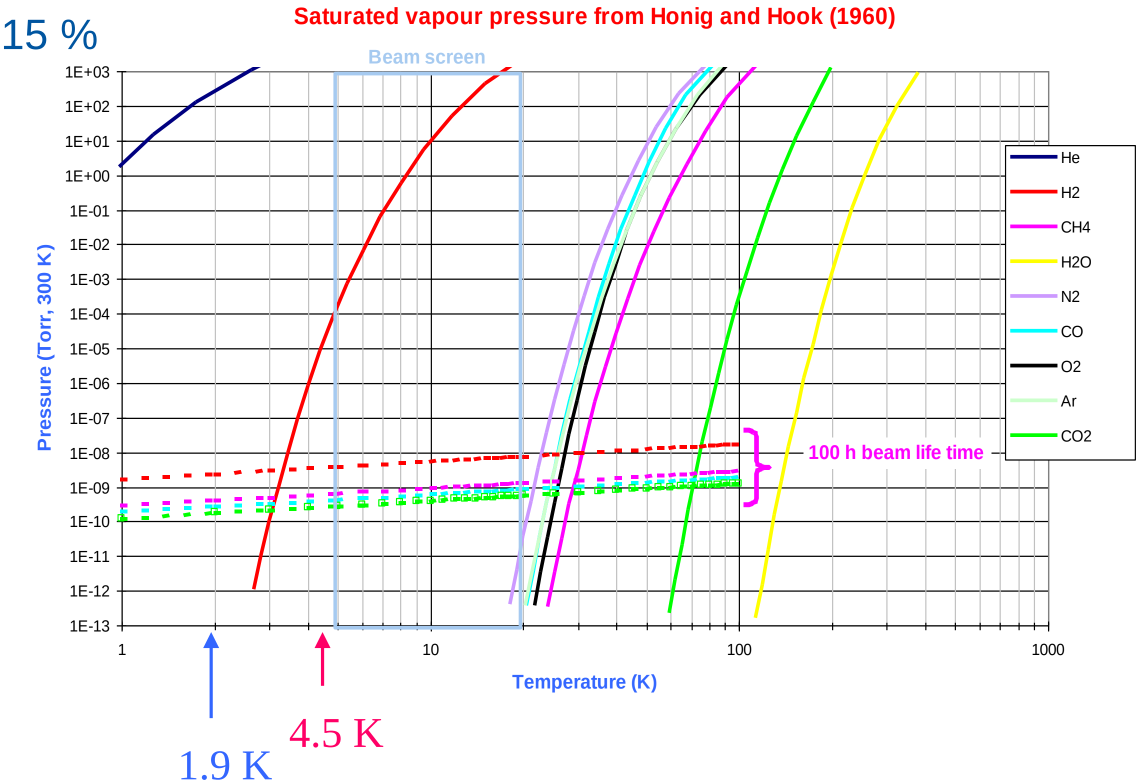
LHC cryosorbers requirements

Complementary information

- The Design requires > 100 h life time with 4.5 K cold bore and thick surface coverage
- Taking into account the gas loads due to the beam stimulated molecular desorption

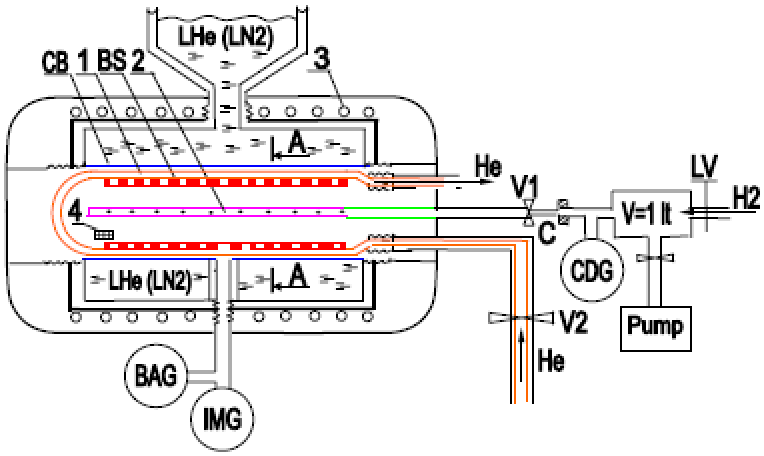
- Required performances (for installation of 200 cm^2/m):
  - Operates from 5 to 20 K
  - Capacity larger than 10^{18} H_2/cm^2
  - Capture coefficient larger than 15 %





LHC Cryosorber Validation By Gas Injection

Complementary information



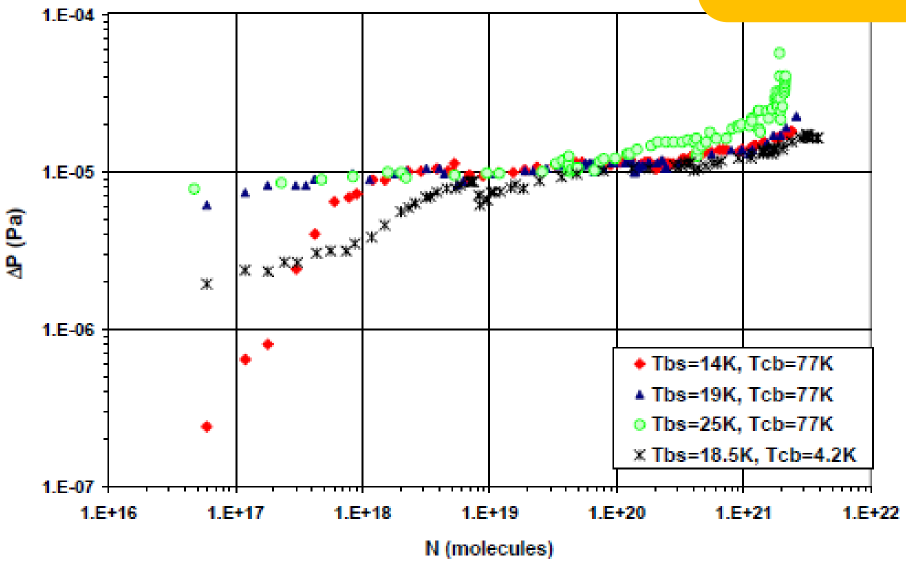


Fig. 3. Dynamic pressure measured by the gauge at room temperature in 1-m long LHC vacuum chamber prototype with a beam screen at different temperatures. Dynamic pressure is normalised to the injecting H_2 flux 10^{15} molecule/s.

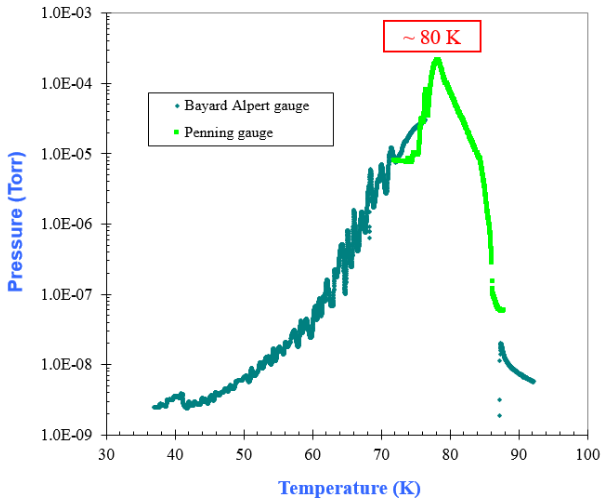
V.V Anashin et al, Vacuum 75 (2004) 293-299

- The dynamic pressure is below design value (*i.e.* 10^{-8} mbar) for a molecular flux less than 10^{14} H_2/s
  - When η_{photon} ~ 10^{-4} H_2/ph, then maximum photon flux is 10x LHC SR
  - When $\eta_{electron}$ ~ 10^{-3} H_2/ph, the corresponding electron cloud heat load is ~ 1 W/m

Operation of Cryosorbers in LHC

Complementary information

- The cryosorbers installed onto the back of the BS provide the required capacity and pumping speed for H_2. They are located in cryoelements operating with 4.5 K cold bores
- The cryosorbers installed in D2, D3, D4, Q4, Q5 and Q6 of the LSS require a regeneration during the shutdown for removing the H_2
- During normal operation of the LHC machine, a regeneration is not foreseen
- The cryosorber is regenerated at ~ 80 K
- While regenerating, the beam is OFF and the BS should be warmed up to more than 80 K and the CB held at more than 20 K (emptying cold mass)
- While the H_2 is liberated from the cryosorbers, it is pumped by an external pumping system.



- Activation energy Ed = 236 meV

$$\frac{E_d}{kT_p^2} = \frac{1}{\tau_0 \beta} \exp\left(-\frac{E_d}{kT_p}\right)$$

V. Baglin *et al.* EPAC'04, Luzern 2004.

4. He leaks in cold systems

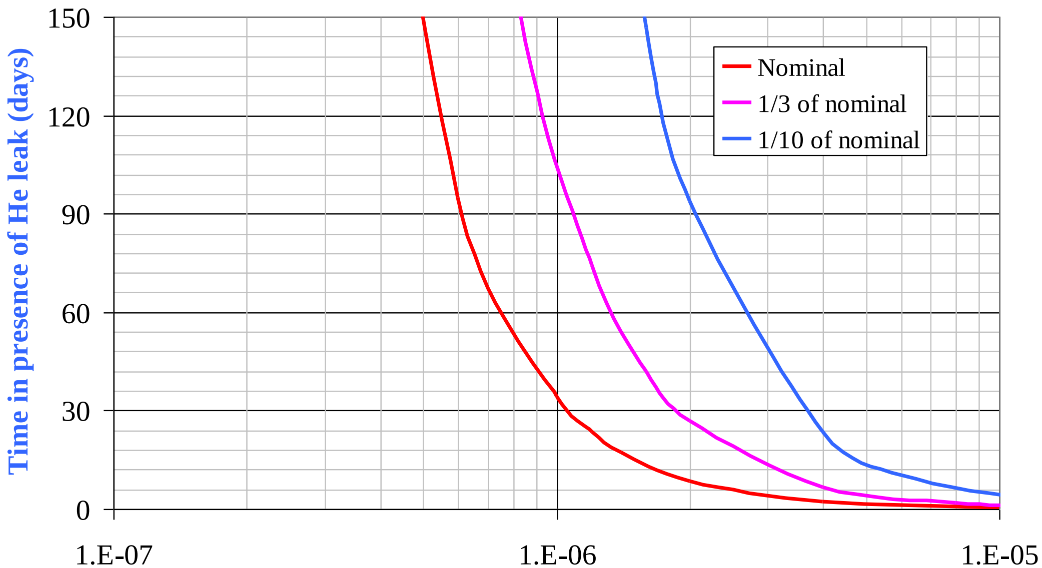
He leak rate with risk of quench

Complementary information

- 1 year of operation ~ 150 days

Helium leak rate above 5 10^{-7} Torr.l/s shall be detected to avoid the risk of a quench

Time to provoke a quench



V. Baglin, Vacuum 81 (2007) 803-807

- Lower leak rate :
  Require a pumping of the beam tube on the yearly basis (cold bore >~4K)

- Larger leak rate will provoke a magnet quench within :
  30 to 100 days beam operation for He leak rate of 10^{-6} Torr.l/s
  A day of beam operation for He leak rate of 10^{-5} Torr.l/s