Cryopumping – Basics and Applications

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Outline

- Introduction.
- Cryopumping mechanisms (condensation, sorption).
- Cryopump sub-systems and issues in their design.
- Commercial cryopumps.
- Tailor-made cryopumps.
- Examples.
Site of Forschungszentrum Karlsruhe

Institute of Technical Physics

FZK features three Research Areas:
- Structure of Matter (neutrino mass, ANKA)
- Key Technologies (micro, nano, grid)
- Energy (fusion, fission, biomass)

FZK: ~ 3500 people

FUSION Programme:
~ 220 FTE

FZK is responsible for 2/3 of the EU Contributions to ITER.
Pump classification (1)

VACUUM PUMPS (METHODS)

Gas Transfer Vacuum Pump

Positive Displacement Vacuum Pump
- Reciprocating Displacement Pump
- Rotary Pump
  - Diaphragm Pump
  - Multiple Vane Pump
  - Sliding Vane Pump
  - Rotary Plunger Pump

Rotary Pump
- Dry Pump
- Roots Pump

Reciprocating Displacement Pump
- Axial Flow Pump
- Radial Flow Pump

Molecular Drag Pump

Turbomolecular Pump

Kinetic Vacuum Pump
- Drag Pump
- Fluid Entrainment Pump

Ion Transfer Pump
- Molecular Drag Pump
- Diffusion Pump
- Ejector Pump

Entrapment Vacuum Pump
- Adsorption Pump
- Cold Trap

Capture vacuum pumps: Non-continuous → ion, getter, cryo

Mechanical pumps: Rotary vane, screw, scroll, roots

Momentum transfer: Diffusion, turbo

CAS – Vacuum in accelerators, Platja d’Aro, May 2006  ‘Cryopumps’  Chris Day  slide # 4
For a cryopump in the applicable pressure range (molecular flow regime) holds: Pumping Speed $S = \text{constant}$.  

But:
Typical pumping speed curve shape

At constant temperature, but variable Gas flow

log (Pressure inside the pump)

Typical pumping speed curve shape

At constant temperature, but variable Gas flow

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Typical pumping speed curve shape

At constant temperature, but variable Gas flow

log (Pressure inside the pump)
Definition according to ISO:

A cryopump is a vacuum pump which captures the gas by surfaces cooled to temperatures below 120 K ....

Cryopumps exploit the most elementary form of producing vacuum by lowering the temperature. They are capture pumps which remove gas molecules by sorption or condensation/sublimation.

Pumping of helium, hydrogen?
Limits of Cryocondensation: Hydrogen as example

Thermodynamic equilibrium
→ Net pumping speed of Zero

Rule of thumb in design: oversaturation by two decades in pressure

Example H\textsubscript{2}:
Achievement of p=10\textsuperscript{-4} Pa (T\textsubscript{eq}=4.2 K) requires 3.6 K (p\textsubscript{eq}=10\textsuperscript{-6} Pa)

→ For D\textsubscript{2}: p\textsubscript{eff}(T=4.2 K) < 10\textsuperscript{-4} Pa

Efficiency of cryocondensation is close to the theoretical maximum, i.e. practically all particles hitting the pumping cryosurface are pumped.
Use of Cryosorption to pump $H_2$ and He @ 4.2 K

Pumping of the gas via physisorption at the cold cryosorbtent. The pumping effect is given by the porosity of the material (pore size distribution rather than BET surface).

- Zeolites (molecular sieve)
- Activated charcoal
- Sintered metal
- Porous ceramics
- Condensed gas frost (most commonly argon)

The porous materials are bonded to the cooled surfaces (by means of a glue/cement/braze/…).

Additional design parameter: Not only pressure and temperature, but also the gas load $\rightarrow$ saturation effects.
Operation point of the cryosorption pump is given by the sorption isotherms

The efficiency of cryosorption is lower than for condensation (you need more hits before the particle becomes immobilized), but the achievable equilibrium pressures are always lower, e.g. $H_2$: $p = 10^{-4}$ Pa; $T(\text{eq}) = 20$ K.
Cryosorbent Material Assessment

Qualification of new Materials and Quality Assurance of different Manufacturing Batches

Refrigerator cold head allows for all temperatures between 3.5 K and 90 K

Sample holder with activated charcoal
Microscopic views

Granular, highly activated, hydrophobic activated charcoal (1.2 mm)

Cryosorbent Material Assessment – Example charcoal

Pressure (Pa)

Gas Load ((Pa·m³)/cm²)

10 µm

100 µm

Type I sorption isotherm

H₂, D₂

He

d(V_ads)/dw (cm³/Å/g)

this work, 3 samples

Belgian Army NBC, 4 samples

Pore Width (Å)

0.00

0.02

0.04

0.06

0.08

0.10

0.12

0.14

0.00

0.02

0.04

0.06

0.08

0.10

0.12

0.14

0.00

0.02

0.04

0.06

0.08

0.10

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0.08

0.10

0.12

0.14

0.00

0.02

0.04

0.06

0.08

0.10

0.12

0.14

1 mm
Choice for the ‘best’ cryosorption material: He @ 5K

The optimum material is not only given by sorption issues, but the complete panel set-up has to be taken into account. The coating may lead to different conclusions for powders/ granules/ pellets.

What counts is the surface-related capacity, not the mass-related value.

<table>
<thead>
<tr>
<th>material aspects</th>
<th>application aspects</th>
<th>optimum panel</th>
</tr>
</thead>
<tbody>
<tr>
<td>sorption data, equilibria, pore distribution</td>
<td>compatibility with bonding agent, thermal cycling, coating technique</td>
<td>best (dynamic) pumping performance for H2, D2, He and their mixtures at 5K</td>
</tr>
</tbody>
</table>
The elementary features of a cryopump set-up: Baffles + shields, pumping panels, and cryosupply

- **Tailor-made in-vessel cryopump**, 4.4 m x 1.5 m
  LHe @ 4.2 K
  S=400 m³/s (H₂)

- **Tailor-made ITER Torus cryopump**, With integrated valve.
  Valve DN 800,
  SCHe (4 bar) @ 4.2 K
  S=100 m³/s (all gases)

- **Commercial Refrigerator-Cryopump**, Up to about 60 m³/s,
  DN 1200,
  Cryogen-free
Cryopump elements are given by cryogenic issues

\[ \dot{Q}_{\text{tot}} = \dot{Q}_F + \dot{Q}_G + \dot{Q}_{\text{Rad}} + \Delta H = \text{min}. \]

1. Solid heat conduction
\[ \dot{Q}_F \sim \lambda / L \cdot A \cdot \Delta T \]
2. Residual gas heat conduction
3. Thermal radiation
\[ \dot{Q}_{12} = C_{12} \cdot A_1 \cdot (T_1^4 - T_2^4) \]
\[ C_{12} = \frac{\sigma \cdot \varepsilon_1 \cdot \varepsilon_2 \cdot \varphi_{12}}{1 - (1 - \varepsilon_1) \cdot (1 - \varepsilon_2) \cdot \varphi_{12} \cdot \varphi_{21}} \]
\[ \dot{Q}_{\text{Rad}} = t \cdot \dot{Q}_{12} \]
4. Phase change enthalpy

Gas Heat Conduction

Source: Haefer

CAS – Vacuum in accelerators, Platja d’Aro, May 2006
Consequences

Cryopumps can in principle provide the maximum theoretical (‘black hole’) pumping speed $S_{id}$, if the cold surfaces are installed directly in the vacuum recipient (i.e. without any conductance limiting flanges).

However, the baffles which are needed due to cryogenic reasons reduce the pumping speed to the practically achievable value. That reduction is being described by the capture probability $c < 1$:

$$S = c \cdot S_{id} = c \cdot \sqrt[2]{\frac{R \cdot T}{2 \cdot \pi \cdot M}} \cdot A$$

The ideal pumping speed is reduced for a real pump
- first by the limited transmission probability $w$ of a particle on the way from the vessel volume on the pumping surface $\rightarrow$
- then by a non 100% sticking probability $\alpha$ of a particle at the pumping surface.

That means, in addition to the usually given values for molecular mass $M$ of the gas being pumped, temperature $T$, and the inlet cross-section $A$:

The essential design task is to tailor the capture probability function $c \rightarrow \max$; Different pump designs should be judged via the parameter $c$, and not directly via $S$. 
Contributions to \( c \)

Separation of the contributions for a simple geometry:

\[
\frac{1}{c} \approx \frac{1}{\alpha} + \frac{1}{w} - 1
\]

- Influence of the pumping mechanism (big or small values, for sorption a complicated function of the pumping history)

- Geometry influence (constant)

Transmission probability \( w \) of the particle from pump inlet to the pumping surface

Sticking coefficient \( \alpha \)

Strong gas species dependency

Source: Haefer
Liquid He bath cryopump

LHe Bath

Pumppanel 1

Pumppanel 2
(Ar frost Spraysystem)

Inlet-baffle
(blackened)

JET
The commercial refrigerator cryopump

2-stage Gifford McMahon refrigerator with closed compressed helium circuit.

See your hand-outs of the talk
On Cryogenics by Philippe Lebrun
The commercial refrigerator cryopump: set-up

- Vacuum chamber
- Inlet baffle (80 to 100K), in contact with the first cold head stage
- Pumping surface (15 K), in contact with the second cold head stage, with condensation on front and sorption on back
- Thermal radiation shield
- Connection to the forepumps (only for regeneration)

Source: Müller, Leybold
The commercial refrigerator cryopump: parameters

**Water**

\[ S_{id} = 14.9 \text{ l/(s}\cdot\text{cm}^2) @ 300 \text{ K} \]

\[ w=1, \alpha=1 \rightarrow c=1 \]

\[ S = S_{id} = 14.9 \text{ l/(s}\cdot\text{cm}^2) \text{ (Black hole pumping speed)} \]

**Nitrogen**

\[ S_{id} = 11.9 \text{ l/(s}\cdot\text{cm}^2) @ 300 \text{ K} \]

\[ W=0.25, \alpha=1 \rightarrow c=0.25 \]

\[ S = 0.25 \cdot S_{id} = 3 \text{ l/(s}\cdot\text{cm}^2) \]

**Cryosorbed Gas**

\[ H_2: S_{id} = 44.6 \text{ l/(s}\cdot\text{cm}^2) @ 300 \text{ K} \]

\[ He: S_{id} = 31.5 \text{ l/(s}\cdot\text{cm}^2) @ 300 \text{ K} \]

\[ H_2: w=0.25, \alpha=0.6 \rightarrow c=0.21 \]

\[ S = 0.21 \cdot S_{id} = 9.3 \text{ l/(s}\cdot\text{cm}^2) \]

\[ He: w=0.25, \alpha=0.3 \rightarrow c=0.16 \]

\[ S = 0.16 \cdot S_{id} = 5 \text{ l/(s}\cdot\text{cm}^2) \]
Standardised criteria for refrigerator-cooled cryopumps

1. Pumping speed
2. Maximum throughput
3. Pumping capacity:
   - The pumping speed decreases with an increasing amount of pumped gas. Especially for adsorbed gases, the pumping speed asymptotically reaches zero. To have a comparable and reproducible measure, the pumping capacity is defined as the quantity of gas, which has been pumped until pumping speed has been reduced to 50% of the initial value. The test for determining the capacity shall be run at constant throughput, so that a decrease of 50% in pumping speed is indicated by doubling the pressure.
4. Ultimate pressure (of the baked pump, after 24 hours).
5. Cool-down time (from ambient to 100/20 K).
6. Crossover:
   - The crossover is defined by the maximum amount of nitrogen gas, which can be admitted into the pump in a time interval of 3s, while the second stage remains at temperatures below 20 K.

Example: DN 320
- 10000 L/s (water)
- 3000 L/s (air)
- ~ 2 Pam³/s (Ar)
- 10⁵ Pam³ (Ar)
- 100 Pam³ (H2)
- 10 Pam³ (He)
- 10⁻¹⁰ Pa
- 2h
- 40 Pam³
What a refrigerator cryopump system comprises

- The cryopump itself.
- The He compressor (one compressor may serve several cryopumps).
- The cooling system of the He compressor.
- The helium lines (to connect compressor and pump, forward and back).
- Power supply (for compressor and pump).
- Sensors (Temperature, pressure).
- Controller (automatic regeneration etc.).
- A backing pump system for regeneration.

COOLVAC ClassicLine Single System Configuration
For both, high and low voltage region

Typical Cryo Pumps
Coolvac 1500 CL
Coolvac 2000 CL
Coolvac 3000 CL
Coolvac 5000 CL
Coolvac 10000 CL

LEVICO Engineering GmbH
Regeneration of the cryopumps:
- becomes necessary, when the pumping speed has decreased to an unacceptable level (which indicates the achievement of the saturation limit).
- may become necessary even before that point, due to safety reasons when flammable gas has been pumped (hydrogen for example). An air inbreak may cause explosive gas mixtures (oxy-hydrogen explosion), which has to be avoided by a strict inventory limitation so that even under the safety event the concentrations stay below the explosion limit.

Regeneration means:
- Automatic heating or purging with warm gas, **Gasrelease** (Sublimation, Desorption) and pump-down of the gas via the backing system, or:
- complete exchange of the cold surface unit.
Regeneration: Sublimation vs. desorption → re-adsorption

Example $CO_2$

![Graph showing gas release characteristic](image)
Regeneration: Typical desorption curves

![Graph showing typical desorption curves for various gases. The x-axis represents panel temperature in Kelvin (K), ranging from 10 to 300. The y-axis represents released gas as a percentage (%). The graph indicates different gases including H₂, D₂, N₂, CO, CH₄, CO₂, He, Ne, and Ar, with their respective desorption curves. Each curve peaks at different temperatures, indicating the typical desorption behavior of these gases.]
Other operational effects in cryosorption pumping

- A poisoning effect of the sorbent may appear, i.e. a reduction of the nominal sorption pumping speed due to accumulation of gas species which are not regenerated within the normal regeneration pattern (all strongly sorbed gases such as heavy hydrocarbons). This effect can be mitigated by a suitable choice of the cryosorbent (Ar frost) or by provision of ‘extra’ regeneration methods.

- Mobility of the sorbed particles plays a role. The capacity of the pump can be increased by defined temperature peaks, which give the sorbed particle at the surface enough energy to migrate deeper into the pores.

- Fractionation of the components when a mixture is being pumped is possible due to the different pumping mechanisms.
Effects 1: Poisoning

Graph showing the relationship between accumulated poisoning gas load and pumping speed for different gas mixtures.

- D2-Base/He=90/10 + water
- D2-Base/He=90/10 + hexane
- D2-Base/He=90/10 + octane
- H2-Base/He=95/5 + water
- H2-Base/He=95/5 + hexane
- H2-Base/He=95/5 + octane

The graph indicates a decrease in pumping speed as the accumulated poisoning gas load increases.
Other operational effects in cryosorption pumping

- A **poisoning effect** of the sorbent may appear, i.e. a reduction of the nominal sorption pumping speed due to accumulation of gas species which are not regenerated within the normal regeneration pattern (all strongly sorbed gases such as heavy hydrocarbons). This effect can be mitigated by a suitable choice of the cryosorbent (Ar frost) or by provision of ‘extra’ regeneration methods.

- **Mobility** of the sorbed particles plays a role. The capacity of the pump can be increased by defined temperature peaks, which give the sorbed particle at the surface enough energy to migrate deeper into the pores.

- **Fractionation** of the components when a mixture is being pumped is possible due to the different pumping mechanisms.
Effects 2: Mobility

Example $D_2$

![Graph showing temperature and pressure relationships with sublimation, mobility, and desorption markers.]

$dT/dt = 0.116 \text{ K/s}$

$q(0) = 0.0025 (\text{Pa} \cdot \text{m}^3)/\text{cm}^2$
Other operational effects in cryosorption pumping

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- Fractionation of the components when a mixture is being pumped is possible due to the different pumping mechanisms.
Effects 3: Fractionation (1)
Effects 3: Fractionation (2)

\[ \frac{dT}{dt} = 0.052 \text{ K/s} \]

\[ q(0) = 0.0025 \text{(Pa·m³)/cm²} \]
Principle pumping speed curves in molecular flow as a function of temperature

At constant throughput/pressure
Advantages of Cryopumping (1)

- Cryopumps produce the biggest pumping speeds of all vacuum pumps:
  - up to 60 m³/s (nitrogen) via DN1200 for commercial pumps
    (compare with maximum 3 m³/s via DN 300 using turbopumps)
  - custom-made cryopumps can provide very high pumping speed via
    the smallest connection area needs (e.g. 100 m³/s (Helium) via DN750).
  - custom-made cryopumps can be installed in situ.

- Cryopumps are absolutely oil-free and generate a clean vacuum completely free of
  oil or hydrocarbons → optimal for clean applications.

- Cryopumps do not have any movable parts →

- No bearing and shaft seal problems (maintenance aspects, lubrication aspects).
  → Stable against electrical blackouts (if cryogen-based).
  → High reliability and no problem with dust and particles.
  → Compatible with strongest safety requirements, cryogen-based cryopumps
    are excellent for areas with difficult maintenance access (‘build in and forget’).

- Cryopumps can be installed in all directions.
Advantages of Cryopumping (2)

- No backing pump system is needed during operation of the cryopump.
- Cryopumps are the pumps of choice for pumping of water/steam or wet gases (extremely high pumping speeds and no corrosion problems).
- The pumping speed of cryopumps increases for light gases.
- Cryopumps are operable over a wide pressure range.
- Cryogen-based cryopumps do not need any electrical power supply at all (which is good for operation under high magnetic fields) and are completely vibration-free.
- ...
Combination of Refrigerator/LN2

- Three stage cryopump.
  - Inlet shield cooled to 77 K with liquid nitrogen.
  - Inside the pump there is a two-stage GM-refrigerator, which provides 50 K on its first stage for the condensation stage,
  - and 20 K on its second stage for the sorption stage (charcoal).
- The regeneration can be done with electrical heaters or purging with warm gas.
- Inlet diameter of 1.25 m
- Nominal pumping speed of 50 m³/s for nitrogen (capture coefficient $c=0.35$) and hydrogen (capture coefficient $c=0.09$).
- Developed for space simulation chambers ($10^{-4}$ Pa).

Source: Air Liquide
A modular panel approach allows an optimal design for the given geometry.
Panel manufacturing

- Initial quilted panel (stainless)
- Copper coating for temperature homogeneity (100 µm)
- Panel equipped with heaters
- Panel charcoal coated
- Quality control: Thermal Cycling
- Equipped with temperature sensors
A modular square-shape cryopump with liquid cryogens

Dimension: 4.4 m x 1.5 m
Supply with 35-40 g/min LHe
And 50 l/h LN2.

Nominal pumping speed for hydrogen is 400 m³/s.
The three cryopump elements

Starting requirement:
Replace a titanium sublimation pump, stay in the available space and increase the Pumping speed by factor 10.
ITER Vacuum Systems - Outline

3 Large Cryopump systems

- Cryostat HV pumping system
- Neutral Beam HV pumping system
- Torus exhaust HV pumping system

Cryo-mech cross-over pressure is 10 Pa
### ITER Large High Vacuum Cryopump Systems - Overview

<table>
<thead>
<tr>
<th></th>
<th>Torus</th>
<th>Beam Heating (NBI)</th>
<th>Cryostat</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong># Pumps</strong></td>
<td>8</td>
<td>2 (3) +1</td>
<td>2</td>
</tr>
<tr>
<td><strong>Pumping mode</strong></td>
<td>Dynamic</td>
<td>Dynamic</td>
<td>Transient pump-down</td>
</tr>
<tr>
<td></td>
<td>= maintain the pressure (1-10 Pa) inside the NBI volume (150 m³/H-NBI) at a throughput of 36 Pa·m³/s (protium operation) + (33 Pa·m³/s (impurities)); Base pressure for hydrogens: $10^{-5}$ Pa.</td>
<td>= maintain the pressure (0.01 Pa) inside the NBI volume (150 m³/H-NBI) at a throughput</td>
<td>(closed cryostat volume of 8400 m³) + (33 Pa·m³/s (impurities)); Base pressure for hydrogens: $10^{-5}$ Pa.</td>
</tr>
<tr>
<td><strong>Gases</strong></td>
<td>Hydrogen (all six isotopes), helium, impurities</td>
<td>Hydrogen ($H_2$, $D_2$)</td>
<td>Nitrogen, outgassing gas</td>
</tr>
<tr>
<td></td>
<td>Depending strongly on the operation mode (burn &amp; dwell, conditioning, leak detection..)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- **High gas throughput at relatively high pressures**
- **High pumpings speeds**
- **Pumps operated in transitional range**

These systems as well as all the Rest of ITER will be covered in a special Talk by Mike Wykes on Tuesday.
Design Issues in the Development of the ITER torus

Cryopumps

1. Pumping port size is limited → Maximize pumping speed / Capture probability at constant entrance area.

2. Worst case to design the cryosorbent panels → reference design shall include a pure helium/protium shot (which means 100% sorption pumping).

3. The composition of the gas mixture being pumped may vary significantly → Minimize the dependency of pumping speed on gas species.

4. The gas throughput is variable and must be controllable → include an inlet valve for control.

5. The distance between the pumps’ location and the divertor is given and big → Maximize conductance of the pumping port.

6. Compatibility with the operating conditions → Magnetic and electric fields, seismic, tritium-compatibility, Safety (Hydrogen explosion), Remote handling…

7. Find and characterise the ´optimum´ cryosorbent.
Step 1: Operational Scheme for the Torus Cryopump

Staggering interval of ??? s

With this trick, we provide to the torus a quasi continuous pumping speed with batch regenerating cryopumps.

Cyclic cryopump regeneration during long pulse, 4 pumps open

- Warmup & cold He recovery
- Desorb
- Evacuate
- Cooldown
- Pumping

Pressure ranges:
- \( p \rightarrow \text{some 100 Pa} \)
- \( p = \text{some kPa} \)
- \( p \rightarrow 10 \, \text{Pa} \)
- \( p \rightarrow 10^{-6} \, \text{Pa} \)
- \( p = 10^{-2} \, \text{Pa max.} \)
Staggering Interval given by Inventory Limitation

Cropump operational pattern is determined by safety considerations, not by saturation effects of the sorbent.

Result #1:
- Staggering interval of 150 s
- Duty cycle = 4*150 s = 600 s
- Volume of 6 m³
Step 2: Complete System Flow Analysis

A sound design has to consider not only the cryopump itself but also the conductance of the port (10 m long, very complex geometry) → system analysis under transitional flow conditions (ITERVAC code)

Cell model of half ITER
Minimum 50 m³/s as target nominal pumping speed per (pump+inlet bellow) ➔ This defines the requested pump size!
Design of the ITER Pump with integrated inlet valve

**Geometry**
- Valve Diameter: 700 mm
- Valve Stroke: 400 mm
- Pump Length: 1350 mm
- Pump Diameter: 1200 mm
- Coated Cryopanel Surface Area: 16 panels, 4 m²
- Panel Length: 870 mm
- Pump volume: 1 m³ (+5)
Experimental Basis: Test of a Torus Model Cryopump in the TIMO Test Bed at FZK
Pumping speed results

Installed pumping surface of 4 m² → 1.2 l/s cm² = 50 m³/s

Molecular flow regime was predicted by Monte-Carlo simulation:

1.0 l/(s·cm²) @ 100% open
0.45 l/(s·cm²) @ 35% open

Pumping with transitional flow conditions inside the pump.
Almost insensitive against the type of gas being pumped, although big differences in $\alpha$(He) = 0.2 vs. $\alpha$(D$_2$) = 0.9. Achieved by a sophisticated interior design.
Saturation capacities @ 5K

![Graph showing saturation capacities at 5K, with lines for Helium and Deuterium. The graph includes a formula: \( q = 0.8 \times 10^{-4} \text{(Pa·m}^3)/(\text{s·cm}^2) \).]
4000 m³/s cryopump set-up with its three elementary parts

- Inner panel: GHe ‘Chevron’ panel
- Intermediate panel: ScHe quilted panel
- Outer panel: GHe quilted panel
..If you want to read about cryopumps

- *The Bible:*


Thank you for your attention