

## TIPS AND TRICKS FROM THE EXPERTS

### Measures to reduce pump-down times in high vacuum

For rapidly reaching the final vacuum in a vacuum chamber, the material and the surface texture are very important. In addition, the gas load that is to be pumped down is relevant as to how quickly the desired final pressure can be realized. This gas load, before reaching the desired base pressure, consists substantially of the leakage rate of the chamber and the gases and vapors adhering to the chamber walls and fixtures. They desorb gradually from the surface under vacuum. This desorption has a great influence on the pump-down times and therefore on the length of time needed to reach the designated final pressure. The interaction of all parameters described above is reflected in the so-called pump-down curve, in which the pressure loss is shown as a function of time.

To increase the pump-down speed of a vacuum chamber, different measures are possible. The use of materials which gas and water vapor molecules adhere to less strongly reduces the pump-down time. An overview of the desorption rates for dry air from the surfaces of various materials is given in Table 1. Due to the continuous removal of adhering gas particles during the pumping process, the gas delivery rates decrease with increasing pump-down time. In general, if the condition of the chamber is good and the pump design suitable, the ultra-high vacuum range can be achieved after a pump-down time of ten hours. From then on, the desorption of gases from the surfaces becomes less significant in comparison to the diffusion of gases from the materials and is therefore no longer relevant for the pump-down time.

Material	Surface quality	Surface condition	Desorption rates <sup>1)</sup> $q_{des}$ [hPa · l s <sup>-1</sup> cm <sup>-2</sup> ]		
			1 h	4 h	10 h
Steel, stainless	blank	cleaned	$2.7 \cdot 10^{-7}$	$5.4 \cdot 10^{-8}$	$2.7 \cdot 10^{-8}$
Steel, stainless	polished	cleaned	$2 \cdot 10^{-8}$	$4 \cdot 10^{-9}$	$2 \cdot 10^{-10}$
Steel, stainless	stained	baked out 1 h flooded with normal air	$1.4 \cdot 10^{-9}$	$2.8 \cdot 10^{-10}$	$1.4 \cdot 10^{-10}$
Steel, stainless	shot-peened	baked out 1 h flooded with normal air	$3 \cdot 10^{-10}$	$6.5 \cdot 10^{-11}$	$4 \cdot 10^{-11}$
Stahl, Ni-plattiert	polished	cleaned	$2 \cdot 10^{-7}$	$1.5 \cdot 10^{-8}$	$5 \cdot 10^{-9}$
Stahl, Cr-plattiert	polished	cleaned	$1.3 \cdot 10^{-8}$	$2.2 \cdot 10^{-9}$	$1.2 \cdot 10^{-9}$
Steel		rusted	$6 \cdot 10^{-7}$	$1.6 \cdot 10^{-7}$	$1 \cdot 10^{-7}$
Steel	blank	cleaned	$5 \cdot 10^{-7}$	$1 \cdot 10^{-7}$	$5 \cdot 10^{-8}$
Steel	shot-peened	cleaned	$4 \cdot 10^{-7}$	$8 \cdot 10^{-8}$	$3.8 \cdot 10^{-8}$
Aluminium		cleaned	$6 \cdot 10^{-8}$	$1.7 \cdot 10^{-8}$	$1.1 \cdot 10^{-8}$
Brass		cleaned	$1.6 \cdot 10^{-6}$	$5.6 \cdot 10^{-7}$	$4 \cdot 10^{-7}$
Copper		cleaned	$3.5 \cdot 10^{-7}$	$9.5 \cdot 10^{-8}$	$5.5 \cdot 10^{-8}$
Porcelain	glazed		$8.7 \cdot 10^{-7}$	$4 \cdot 10^{-7}$	$2.8 \cdot 10^{-7}$
Glass		cleaned	$4.5 \cdot 10^{-9}$	$1.1 \cdot 10^{-9}$	$5.5 \cdot 10^{-10}$
PMMA			$1.6 \cdot 10^{-6}$	$5.6 \cdot 10^{-7}$	$4 \cdot 10^{-7}$
CR			$4 \cdot 10^{-5}$	$2.2 \cdot 10^{-5}$	$1.5 \cdot 10^{-5}$
NBR			$4 \cdot 10^{-6}$	$1.7 \cdot 10^{-6}$	$1.3 \cdot 10^{-6}$
FKM			$1.2 \cdot 10^{-6}$	$3.6 \cdot 10^{-7}$	$2.2 \cdot 10^{-7}$
FKM		baked out 4 h at 100°C	$1.2 \cdot 10^{-7}$	$5 \cdot 10^{-8}$	$2.8 \cdot 10^{-8}$
FKM		baked out 4 h at 150°C	$1.2 \cdot 10^{-9}$	$3.3 \cdot 10^{-10}$	$2.5 \cdot 10^{-10}$
PTFE		degassed	$8 \cdot 10^{-7}$	$2.3 \cdot 10^{-7}$	$1.5 \cdot 10^{-7}$

<sup>1)</sup> Thank to various pre-treatments, the desorption rates can be enhanced (i.e. burning up without hydrogen).

Table 1: Overview of the desorption rates for dry air from the surfaces of various materials

Compared with plastics, the outgassing rates of metals are much lower. Exceptions are, for example, copper alloys: they also have high outgassing rates. Plastics and metals with higher desorption rates are used in seals, as O-rings made of elastomers up to the high vacuum range or as metal seals in the ultra-high vacuum region.

The outgassing rate of elastomers can be reduced with different chemical compositions. Fluorocarbon compounds, for example, have a desorption rate an order of magnitude lower than nitrile rubber compounds.

### Reducing the use of flanges

Since the sealing materials used in flanges have higher desorption rates than the materials of the rest of the chamber, the number of flanges should be reduced to a minimum. In this way, the risk of the formation of leakages can be reduced.

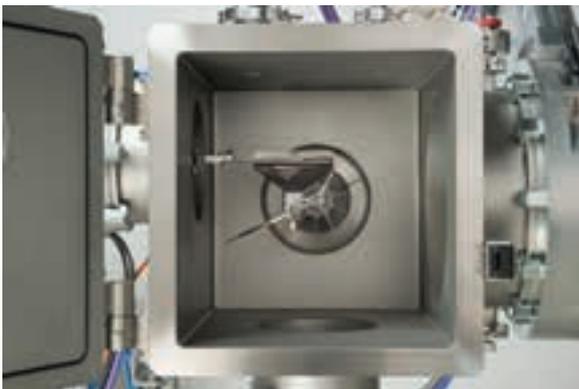


Fig.1: The setup for the examples consists of the vacuum chamber, fore-vacuum pumping station WH 950 and the turbo pumping station HiPace 2300.

## INFOBOX

Figure 2 shows two typical pump-down curves, determined with different desorption rates. They were intended for the following setup (Figure 1):

### Vacuum chamber:

Chamber volume: 1 m<sup>3</sup>

Internal surface: 6 m<sup>2</sup>

Leakage rate of the chamber: < 1·10<sup>-4</sup> Pa m<sup>3</sup>/s

Gas to be pumped down: Air at 20 °C

Intake line: 1 m DN 100 pipe between fore-vacuum pumping station and chamber

### Vacuum pumps:

Fore-vacuum pumping station WH 950, consisting of Okta 1000 Roots piston pump and Hepta 300 P dry pump. HiPace 2300 turbopump on high vacuum side with 15% pumping speed reduction through a protective screen and flange on the chamber (effective pumping speed 1615 l/s).

The pump-down curves were determined empirically based on the desorption rates of a clean and dry stainless steel chamber and a stainless steel chamber slightly soiled by the coating processes. Figure 2 shows the pressure curve for the first hour of pump-down time. Towards the end, an order of magnitude lower desorption rate leads to an order of magnitude lower chamber pressure. It is quite clear that for the desired reduction in the desorption rate, the same prevailing pressure in one chamber is reached after six minutes of pump-down that is reached only after 45 minutes with a higher desorption rate.

Info box 1: Exemplary representation of the pump-down curves

### Reducing the pump-down time by heating the chamber walls

The desorption of gas and water particles from surfaces very much depends on temperature. To overcome the adhesion forces, energy is necessary. The desorption rate  $q_{\text{des}}$  is determined by the ratio of the desorption energy  $E_{\text{des}}$  to the kinetic energy of the particles  $E_{\text{kin}} = kT$ . The temperature dependence is determined by the so-called „Boltzmann factor“:

$$Q_{\text{des}} \sim e^{-E_{\text{des}}/kT}$$

where the Boltzmann constant is  $k = 8.6210^{-5}$  eV/K and the absolute temperature  $T$  is in Kelvin.

The desorption of water is of the order of 0.5 eV. The system is supplied with 0.025 eV at room temperature. Accordingly, the desorption rate is of the order of 0.5 eV. At room temperature, the system is supplied with 0.025 eV. Thus, the desorption rate is  $e^{-20}$ , thus  $2 \cdot 10^{-9}$ . If the temperature is now

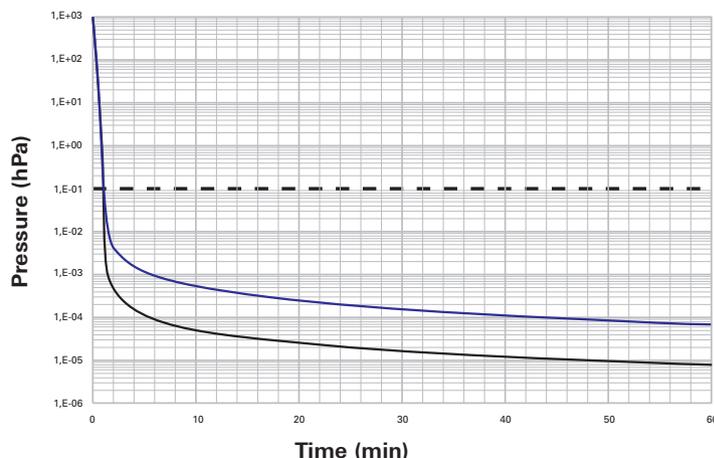


Figure 2: Pump-down curves for desorption rates  $2 \cdot 10^{-6}$  hPa·l s<sup>-1</sup>cm<sup>-2</sup> (upper curve, lightly soiled stainless steel chamber) and  $2 \cdot 10^{-7}$  hPa·l s<sup>-1</sup>cm<sup>-2</sup> (lower curve, clean and dry stainless steel chamber). Dashed line: Change-over pressure, when switching from the fore-vacuum to the turbo pumping station.

increased, for example to approximately 100 °C, the system will be supplied with about 0.032 eV. The desorption rate is correspondingly in the order of  $e^{-15.6}$ , thus  $2 \cdot 10^{-7}$ . At this two decade higher outgassing rate, molecules that adhere to the surface can be removed much more easily. Thus, the desired base pressure in the chamber is also reached more quickly.

However, even if such devices for heating the chamber walls are not available, the pump-down time can be easily shortened: If the vacuum chamber in batch systems is opened frequently, it often results in increased adhesion of water molecules to the chamber walls, depending on the time of year and the prevailing ambient humidity. This automatically results in a longer pump-down time, which can be shortened by flooding the chamber with nitrogen. In this process, the chamber walls are covered with nitrogen molecules. Thus, after opening the chamber, penetrating water vapor cannot adhere so tightly to the chamber walls.

In processes in which water vapor is always present, the use of cryogenic condensers is recommended. With their help, the water vapor is frozen on the cold walls. The high vacuum pump then only has to pump out the so-called permanent gases that cannot be cryotechnically frozen. This results in a considerable reduction in the pump-down time.

Desorption in a high vacuum range up to  $10^{-6}$  has a significant influence on the pump-down time. As the above examples show, it is possible to help reduce the pump-down time by considering various aspects even in the design of a vacuum system. And even with existing systems, small changes - such as suitable seals or venting the chamber with nitrogen - shorten pump-down times significantly. As a result, the base pressure can be reached quickly and the actual work with the system can begin.

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