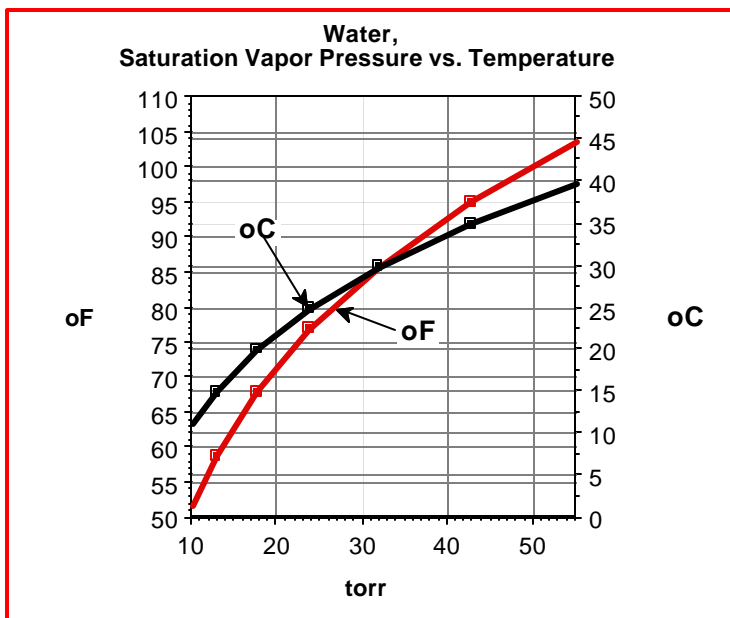


## The Effects of Humidity on Vacuum Systems

The presence of water vapor in vacuum systems is perhaps the most common of all problems that face the practitioners of vacuum technology. Most problems such as leaks or massive contamination can be dealt with on a one time or, at least, recurring basis, but water vapor remains as a daily problem to be faced with every pumpdown. As a pumpdown cycle proceeds to pressures below  $10^{-3}$  torr, water vapor becomes the dominant gas species within the partial pressures of the various residual gases remaining in the chamber. During the pumpdown, the direct source of the water vapor is mainly desorption from internal surfaces and elastomer O-rings. The original source of the water vapor, though, is from the ambient air to which the chamber and work load were exposed prior to the pumpdown cycle.



*The actual partial pressure of water vapor in the air can be easily calculated if the percent relative humidity and the temperature are known by taking the percent of the saturated vapor pressure.*

Water vapor, in the form of humidity, is found in all air. The amount will vary with temperature, atmospheric pressure, and available water sources, but there will always be a certain amount present. A chart, showing the composition of air in terms of partial pressures of its constituents, will list permanent gases such as nitrogen and oxygen. It will also give the partial pressure of water vapor in terms of relative humidity (RH), temperature, and altitude. Consider, for a moment, a weather report that gives RH along with the other weather factors.

That RH number is actually the percentage of water vapor in the air relative to a saturated condition at the temperature given. That relative humidity, for a given

temperature, can be then converted into the partial pressure of water vapor present in the air.

Any material exposed to air for any length of time will reach, or be in the process of reaching, some sort of equilibrium with the water vapor in the air. For example, a porous material will absorb water vapor continually until it's saturated, but there will be a finite period before it's fully saturated. Saturation requires a two-stage process where the surface saturates quickly in the first stage while a slower stage follows where the water vapor slowly migrates from the surface into the bulk. Each material will have its own rate of saturation with its own absorption mechanism. Non-porous materials such as chamber walls will adsorb water vapor in molecular monolayers that remain on the surface. The polar nature of the water molecules not only causes them to adhere to the surfaces with weak bonds, but to adhere to each other in ever weakening bonds as the bed of sorbed molecules becomes thicker and more disordered.

The common factor in all types of water sorption is that the water vapor molecules must come into physical contact with the surface. Since gas molecules are in constant motion resulting in molecule-to-molecule impacts, the amount of sorption will be proportional to the number of impacts of water molecules onto a surface per unit time. As the water molecules are sorbed by the surface, they are instantly replaced, in the air, by others because of the constant interdiffusion produced by the molecular motion. These molecules are sorbed, only to be replaced in turn, and this process continues as long as the surfaces are exposed to air. Obviously, the number of impacts will be lower if the air is less humid or the exposure time is limited. The temperature of the surfaces exposed to water molecules also comes into play. At room temperature, the water will be sorbed in the vapor state, but if the surfaces are below the dew point for a given RH, liquid water will form. This effect is often observed in cases where water-cooled chambers or devices are exposed to humid air by letting the chamber up-to-air while they are still cold. A good way to avoid this problem is to switch the flow from cold to hot water well before air-release. Observing a pumpdown from humid air can supply a good mental picture of the behavior of water vapor during a pumpdown. As the pressure drops during the roughing cycle, a small cloud will form momentarily in the chamber as the gas cools due to rapid expansion. This Wilson cloud chamber effect is often noted in glass bell jar systems. The droplet formation has been reported to cause particle formation on substrates.

Backfilling a chamber with dry nitrogen ( $N_2$ ) instead of ambient air is a well known and accepted procedure to produce a faster pumpdown time after opening the chamber. There is an erroneous, but persistent, belief that the  $N_2$  molecules will sorb to the surfaces and thus provide a barrier layer to reduce the sorption of water molecules onto the surface. There's a tiny grain of truth to  $N_2$  surface sorption effects because it will sorb onto surfaces under ultra-high vacuum conditions where the surfaces are totally clean. In a practical high-vacuum system that's cycled up-to-air

frequently, any N<sub>2</sub> sorption is negligible because there is almost always a sorbed monolayer of water molecules already on the surface of unbaked systems. The fact that N<sub>2</sub> backfilling will result in a faster pumpdown is almost entirely traceable to the N<sub>2</sub> filling virtual leak voids and barring water diffusion into those voids due to the small N<sub>2</sub>-to-air interface area. This effect can be corroborated by backfilling with argon (A) and comparing pumpdown curves. The N<sub>2</sub> and A curves will be identical within the usual ion gauge readout scatter. The only way to prevent water vapor diffusion, after N<sub>2</sub> backfilling, is to restrict the chamber opening to a small area such as demounting a small flange for a short period or maintaining a positive flow of N<sub>2</sub> while the chamber is open. N<sub>2</sub> can be a potential safety problem since the flow will bar oxygen as well as water vapor, and the operator might breathe the N<sub>2</sub> while loading the system. The flow technique is totally impractical with systems that have large doors or require a raised bell jar. To get a feel for the rate at which water vapor will diffuse through air, consider a gas with a sufficiently intense odor to still be detected by the olfactory sense even though it's greatly diluted. For example, a minuscule aliquot of hydrogen sulfide released into a room can be almost instantly detected many feet away.

The elastomer O-rings used to seal the flanges on many vacuum chambers are often the major source of water vapor gas loads within the chamber. Viton O-rings, arguably the most common sealing material, is heavily loaded with water formed during the manufacturing process. If a fresh O-ring in the "as manufactured" condition is used on a system, the Viton will act as a source of water vapor during use until it's been under vacuum for the weeks or months it takes to allow the water

to diffuse to the O-ring's vacuum exposed surface and desorb. This extremely high initial outgassing rate can be avoided by pre-baking the O-rings under vacuum or buying pre-baked O-rings. Once exposed to air, though, the Viton will begin to absorb water vapor from the air. As in metallic surfaces, exposure time and the amount of water molecules due to humidity that impact its surface will control the amount of water vapor it will absorb that will add to the system's water vapor outgassing rate during subsequent pumpdown cycles.

<b>Composition of Atmospheric Air</b>		
Gas	Volume %	Partial Pressure (torr)
N <sub>2</sub>	78.08	5.94 x 10 <sup>2</sup>
O <sub>2</sub>	20.95	1.59 x 10 <sup>2</sup>
Ar	0.93	7
CO <sub>2</sub>	0.033	2.5 x 10 <sup>-1</sup>
Ne	1.8 x 10 <sup>-3</sup>	1.4 x 10 <sup>-2</sup>
He	5.24 x 10 <sup>-4</sup>	4.0 x 10 <sup>-3</sup>
CH <sub>4</sub>	2.10 <sup>-4</sup>	1.5 x 10 <sup>-3</sup>
Kr	1.1 x 10 <sup>-4</sup>	8.4 x 10 <sup>-4</sup>
H <sub>2</sub>	5.0 x 10 <sup>-5</sup>	3.8 x 10 <sup>-4</sup>
N <sub>2</sub> O	5.0 x 10 <sup>-5</sup>	3.8 x 10 <sup>-4</sup>
Xe	8.7 x 10 <sup>-6</sup>	6.6 x 10 <sup>-5</sup>
O <sub>3</sub>	7 x 10 <sup>-6</sup>	5.3 x 10 <sup>-5</sup>
H <sub>2</sub> O*	1.57	1.19 x 10 <sup>1</sup>

\*50% relative humidity at 23°C

*Water vapor makes up a surprising portion of atmospheric air.*

Backfilling the chamber with a dry gas will accomplish much the same results as found with virtual leaks. The O-ring's surface will absorb the dry gas and slow the absorption of water vapor. Additionally, most O-rings are located within trapped areas caused by small flange-to-flange gaps that make them essentially virtual leak sites. They are then partially protected from full exposure to humid air while the system is open to air and the surface sorption of water vapor is, at least, lessened.

Bulk absorption/desorption and surface adsorption/desorption are not the only processes that interact with humidity. Permeation of gases from the ambient air can add a significant gas load to a system that's been pumped down to its customary ultimate pressure. In fact, permeation gas loads are usually larger than the outgassing gas loads from pre-baked Viton. This means that the permeation gas load is the limiting factor in the system's ability to achieve a low pressure. A good example of the effects of permeation is often encountered when leak checking a system by probing with helium as a tracer gas. Helium, directed onto a real leak, will show an almost immediate indication on a leak detector, but if the helium is allowed to flow over an O-ring for a minute or so, a slow rise in the apparent leak rate will be detected. This high helium background will remain until the helium has completely passed through the O-ring material. This can take hours. An inexperienced vacuum practitioner can mistake the permeation as a real leak, and although a real leak and permeation both allow penetration of the vacuum envelope, they are totally different processes and should not be confused.

Permeation rates through O-rings are controlled by the material's temperature and the partial pressure of gas across the O-ring's cross section. In general, increasing the temperature results in an increased permeation rate. Additionally, each gas has a different permeation rate through any given material. For example buna-N has a relatively low helium permeation rate with an attendant high water permeation rate, but the situation is entirely reversed for Viton. Obviously, then, the permeation process of envelope penetration is not merely a function of the penetrating gas's molecular weight as is true for a real leak. All of the air gases permeate Viton, but water vapor is the only one that presents a normal variation on the air side of a seal. The humidity becomes a problem when we consider that the permeation rate for water vapor from air at 20° C and 50% RH is approximately the same as air. As the humidity increases, usually along with a temperature increase, the predominant permeating gas will be water vapor. Barring the replacement of Viton with metal gaskets, the only way to avoid the permeation of water vapor is to use two concentric O-rings with an evacuated channel between them so that the channel can act as a "guard vacuum." A pressure of only about 10 torr is usually adequate for this pressure to totally stop any permeation.

The unavoidable presence of humidity of any degree is an important consideration in the practice of vacuum technology. Understanding of what its presence means along with ways to avoid its effects can allow better pumpdown performance and attendant increases in product throughput.

*Reprinted with permission by R&D Magazine, all rights reserved. Cahners Business Information. A shorter version appeared in R&D Magazine, June 2001.*