High, Ultra-high & Extreme High Vacuum:
The fundamentals

Leybold

Content

Note: throughout this e-book we refer to mbar where 10^{-12} mbar equates 10^{-10} Pa

Introduction

It is only by examining the differences (at a molecular level) between the various vacuum levels that one can begin to appreciate the challenges associated with achieving and working with high vacuum (HV), ultra-high vacuum (UHV) and extreme high vacuum (XHV).

In rough and medium the major source of gas is from the “bulk” or original gas, whereas in HV and UHV the gas load is dominated by outgassing from surface gas desorption; in XHV the main load is from gas permeation from the chamber walls and other materials.

What is the definition of high, ultra and extreme high vacuum?

- The pressure range of XHV is usually defined as 10^{-12} mbar (10^{-10} Pa) and lower.
- UHV is between 10^{-7} and 10^{-12} mbar (10^{-5} to 10^{-10} Pa)
- HV between 10^{-7} and 10^{-3} mbar (10^{-5} to 10^{-1} Pa).

XHV is associated with the levels found in outer-space in the form of geo-stationary orbiting satellites, UHV with high-energy physics and nuclear research, such as that being conducted at CERN and KATRIN, and HV for industrial and research applications.

As one would expect, the established norms, rules and protocols that define and govern vacuum factors and matters, from how to obtain such vacuum levels, the pump set-up, safeguards, measurement methods, as well as leak detection, must all be thoroughly re-examined and frequently re-engineered.

Key considerations for working in HV, UHV & XHV conditions

Several key considerations for working in HV, UHV and XHV conditions are associated with the system design, including the materials being employed.

In addition, the condition of the system/chamber surface is also important and can be optimized by:

- minimising the chamber's internal surface area
- only welding from the inside
- using materials with low desorption/outgassing rates
- suitable pre-treatment of materials (e.g. electro-polishing)
- making sure there are no internal gaps or trapped volumes (e.g. trapped blind holes)
- reducing the number of seals, feed-throughs etc.
- and employing metallic seals

Pre-treatment of the system is important, and includes heating to a high-temperature (known as "baking"), careful handling using powder-free latex gloves to prevent fingerprint greasing, and thorough cleaning to remove hydrocarbons, fillings and other contaminants (both chemical and physical).

Outgassing for HVs, UHVs and XHVs

At the HV, UHV and XHV end of the vacuum spectrum, the most important contribution to pressure reduction (and vacuum retention) is that associated with outgassing. Outgassing, which is a major problem in HV, UHV and XHV systems, is the result of the desorption of previously adsorbed molecules, by bulk diffusion, permeation and vapourisation.

At room temperature									
Standard values* (mbar • l • s^{-1} • cm^{-2})			Metals 10^{-9}....• 10^{-7}				Non-Metals 10^{-7}....• 10^{-5}		
Outgassing rates (standard values) as a function of time									
Examples:	1/2 hr.	1 hr.	3 hr.	5 hr.	Examples:	1/2 hr.	1 hr.	3 hr.	5 hr.
Ag	$1.5 \bullet 10^{-8}$	$1.1 \bullet 10^{-8}$	$2 \bullet 10^{-9}$		Silicone	$1.5 \bullet 10^{-5}$	$8 \bullet 10^{-6}$	$3.5 \bullet 10^{-6}$	$1.5 \bullet 10^{-6}$
Al	$2 \bullet 10^{-8}$	$6 \bullet 10^{-9}$			NBR	$4 \bullet 10^{-6}$	$3 \bullet 10^{-6}$	$1.5 \bullet 10^{-6}$	$1 \bullet 10^{-6}$
Cu	$4 \bullet 10^{-8}$	$2 \bullet 10^{-8}$	$6 \bullet 10^{-9}$	$3.5 \bullet 10^{-9}$	Acrylic glass	$1.5 \bullet 10^{-6}$	$1.2 \bullet 10^{-6}$	$8 \bullet 10^{-7}$	$5 \bullet 10^{-7}$
Stainless Steel		$9 \bullet 10^{-8}$	$3.5 \bullet 10^{-9}$	$2.5 \bullet 10^{-8}$	FPM, FKM	$7 \bullet 10^{-7}$	$4 \bullet 10^{-7}$	$2 \bullet 10^{-7}$	$1.5 \bullet 10^{-7}$

*All values depend largely on pretreatment!

Outgassing can occur from two sources: surfaces and bulk materials.
Outgassing from bulk material vaporization is minimized by the selection of materials with low vapor pressures (such as glass, stainless steel, and ceramics) for everything that is inside the system. Materials which are not normally considered absorbent can actually outgas significantly, these include most plastics and some metals. For example, vessels lined with a highly gas-permeable material such as palladium (which is a high-capacity hydrogen sponge) creates its own special outgassing problems.

Many common materials are used sparingly, if at all, due to high vapour pressure or absorptivity which can result in subsequent troublesome outgassing, or high permeability in the face of differential pressure, for example, “through-gassing”.

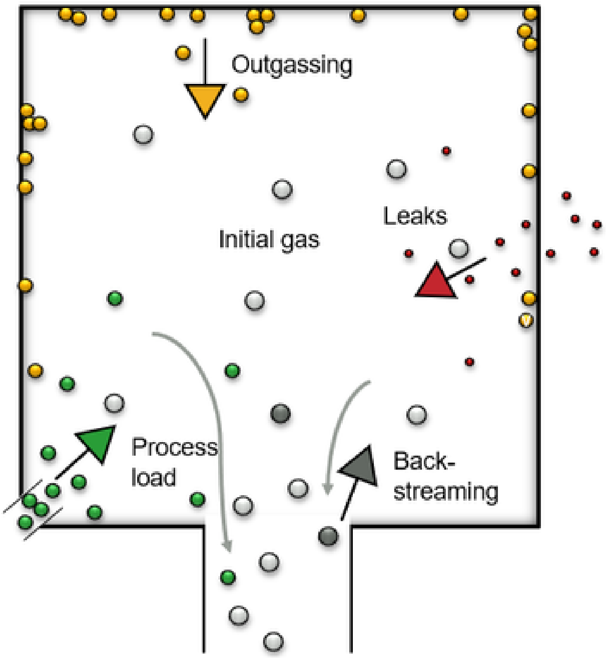
The majority of organic compounds cannot be used. Whilst it would be logical to assume that steels are suitable, the reality is actually not so clear. Due to oxidization of carbon steel, which greatly increases its adsorption area, only stainless steels should be used. Particularly, non-leaded and low-sulfur austenitic grades of steel such as 304 and 316, which include at least 18% chromium and 8% nickel. Other variants of stainless steel that are suitable include those with additives such as niobium and molybdenum, which reduces the formation of chromium carbide. The science and research associated with materials that are compatible with HV, UHVs and XHVs is extensive and all but a mention is beyond the scope of this article.

Whilst there are significant variations in the measured outgassing rates for different materials, such rates are also impacted by pumping speed, time taken to evacuate the chamber, system temperature and the base pressure to be achieved.

To illustrate the importance and significance of outgassing at HV, UHV and XHV ranges, it is worth considering that at a pressure of 10^{-6} mbar there are 500 molecules residing on the walls of the system for every molecule that is free to move around the system. This highlights the fact that at such levels, the pressure is determined by the system's surface gas load. This explains why surface analysis equipment (which is described later) is usually employed at these vacuum levels.

There are six main contributions to the gas load within a system:

- the **initial gas** contained in the system/chamber
- the **gas load due to the pumping process**
- entrapped gases (which will add further to the load)
- **back-streaming**/migration (which can be reduced by using cold traps, anti-suck-back valves etc.)
- **outgassing** (which is a major contributor to gas load and is the hardest to counteract)
- and **leaks**



Finally, on the subject of outgassing, it is important to clarify the difference between the two "sorption" processes: **absorption** and **adsorption**.

- **Absorption** is a physical or chemical process by which a substance enters into the bulk of a second substance, usually in a different phase (in this case, gas molecules into the solid material of the chamber wall).

- **Adsorption** is an accumulation of molecules from the gas phase on/adjacent to the surface of a second substance (in this case, the chamber wall). Adsorbed molecules can simply "desorb", whilst absorbed molecules must first "diffuse to the surface" and then undergo desorption.

High, Ultra & Extreme High Vacuum Generation

What pump types can you use to generate HV, UHV and XHV?

Obtaining HV, UHV and XHV levels can only be effectively and efficiently obtained by using a fore pump that charges the main pump. Fore pumps, (sometimes called “backing pumps”) reduce the pressure to a level where HV, UHV and XHV pumps can take over to operate in a safe, efficient and effective manner. However, pairing different types of vacuum pumps for optimum performance is not straight forward. There are no off-the-shelf pumping systems which cover all applications, eventualities and requirements simultaneously, as there are a myriad of critical factors and impacts to be taken into account. The selection of which pump (both fore and main) depends on a number of factors including noise/vibration, cost (initial and on-going), tolerance to contamination, footprint, maintenance schedules, resilience to shock etc.

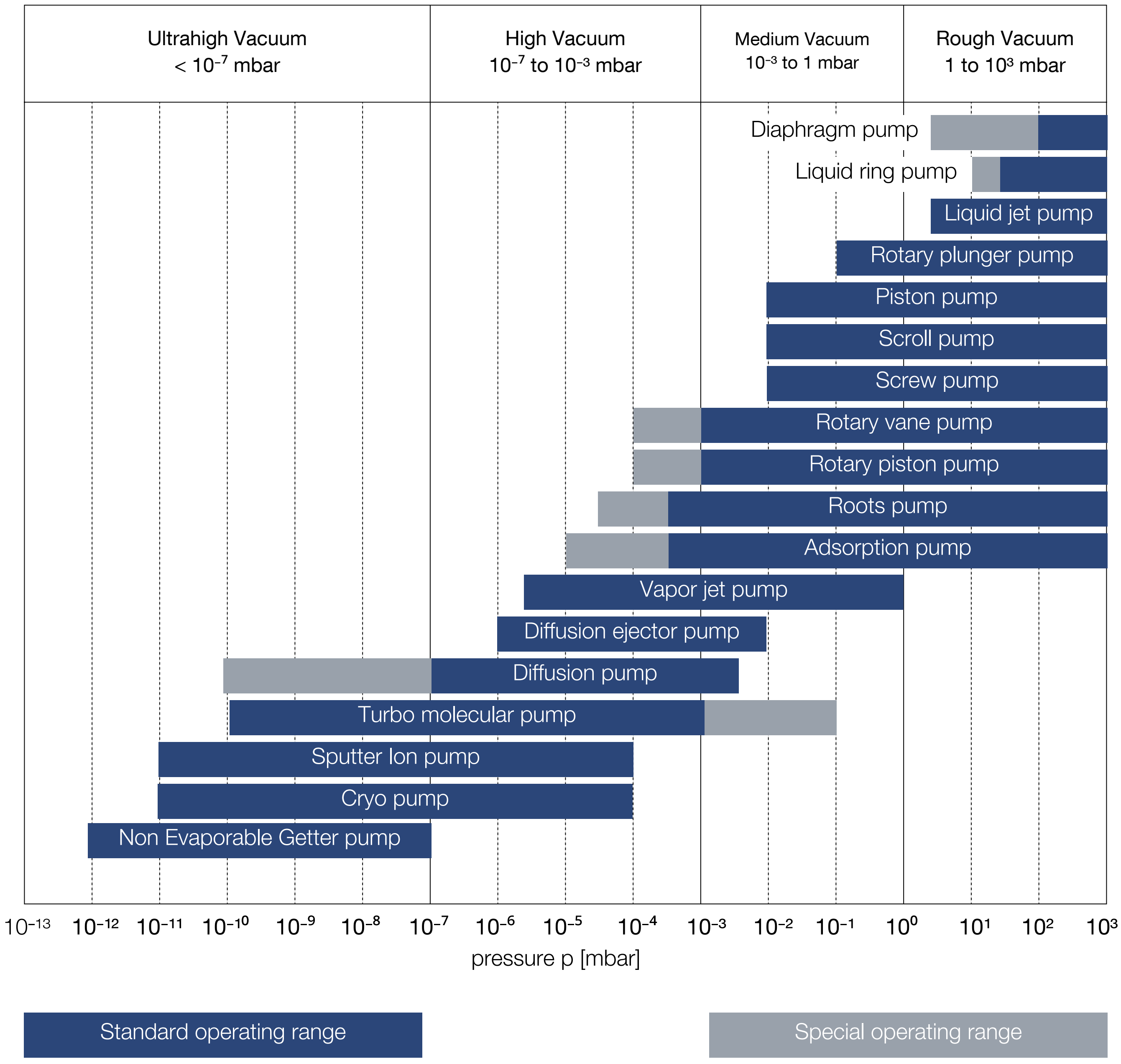
However, there is no single ideal HV, UHV or XHV pump: each type has its own set of advantages and detractions.

The main fore pumps (which preferably are oil-free and dry) include:
- Diaphragm pumps
- Scroll pumps
- Multi-stage roots pumps
- and Screw pumps

The main pumps capable of delivering HV, UHV and XHV, and which can do so in rapid draw-down time include:

- Diffusion pumps
- Cryo pumps
- Sputter Ion Pumps (SIPs)
- Titanium sublimator pumps (TSPs)
- Non-evaporable getters (NEG) pumps
- Turbomolecular pumps (TMPs)

The commonality of these HV, UHV and XHV pumps is their ability to produce vacuum conditions by either rapidly evacuating gas molecules or by capturing/tying them up.



What are the benefits and limitations of these pumps?

Kinetic-type pumps (such as TMPs) have numerous benefits:

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- Easy to operate;
- Low maintenance;
- Provide a hydrocarbon-free operation;
- Require no regeneration;
- Operate at high pumping speeds in the HV, XHV and UHV range.

However, TMPs are not without their disadvantages including:

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- Moving parts mean that they produce vibration and electrical noise;
- Reduced pumping speeds for light gases;
- Sensitive to mechanical shocks;
- Intolerant of particulate contamination.

The capture-type pumps (such as SIPs) also have their own set of benefits:

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- Because they have no moving parts, vibration and electrical noise are eliminated;
- They are built with radiation tolerant materials in excess of $1e^8$ Gray;
- By removing the magnets, capture pumps can be baked up to 450°C (such long hot bakes are critical to every HV, UHV and XHV system);
- Capture pumps require virtually no maintenance.
- Very energy efficient at low pressures.

However, IGPs also have several disadvantages when compared to other pump types:

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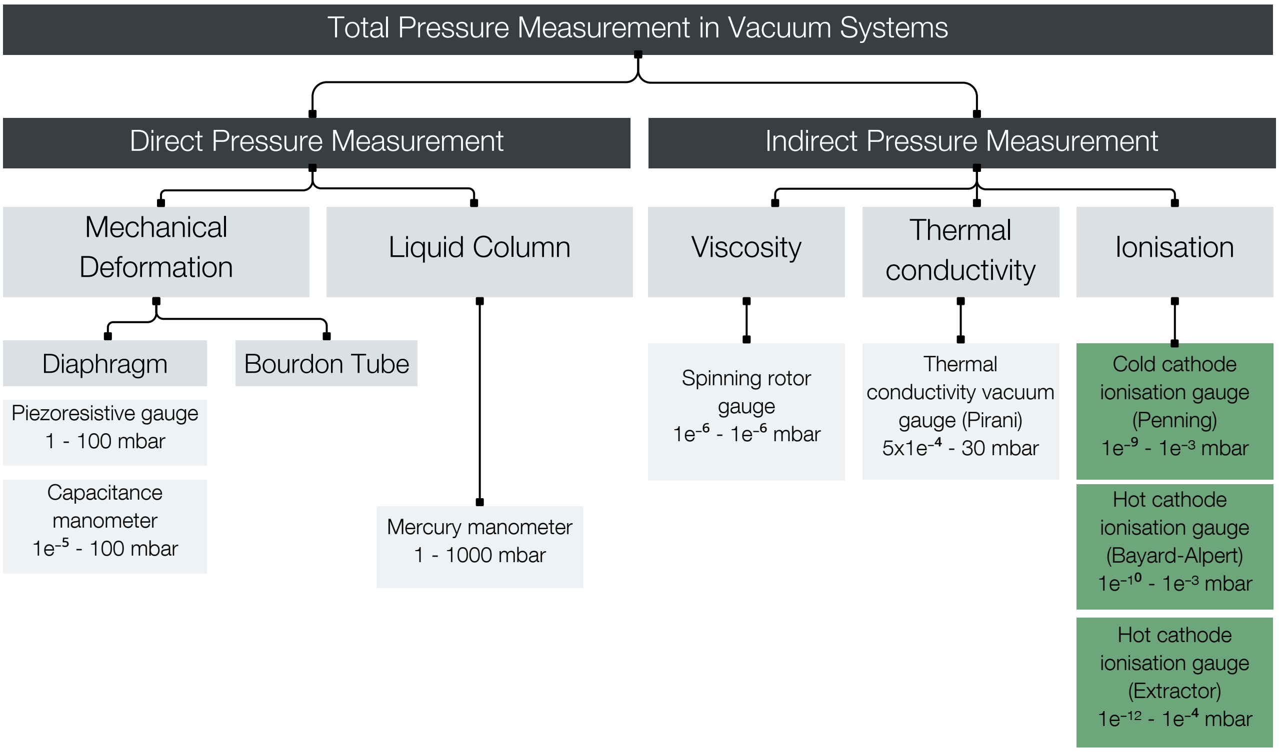
- Low pumping efficiency for noble gases;
- Decreasing pumping speed in the HV, XHV and UHV range;
- Need high voltage and magnetic field;
- Are heavy.

High, Ultra & Extreme High Vacuum Measurement

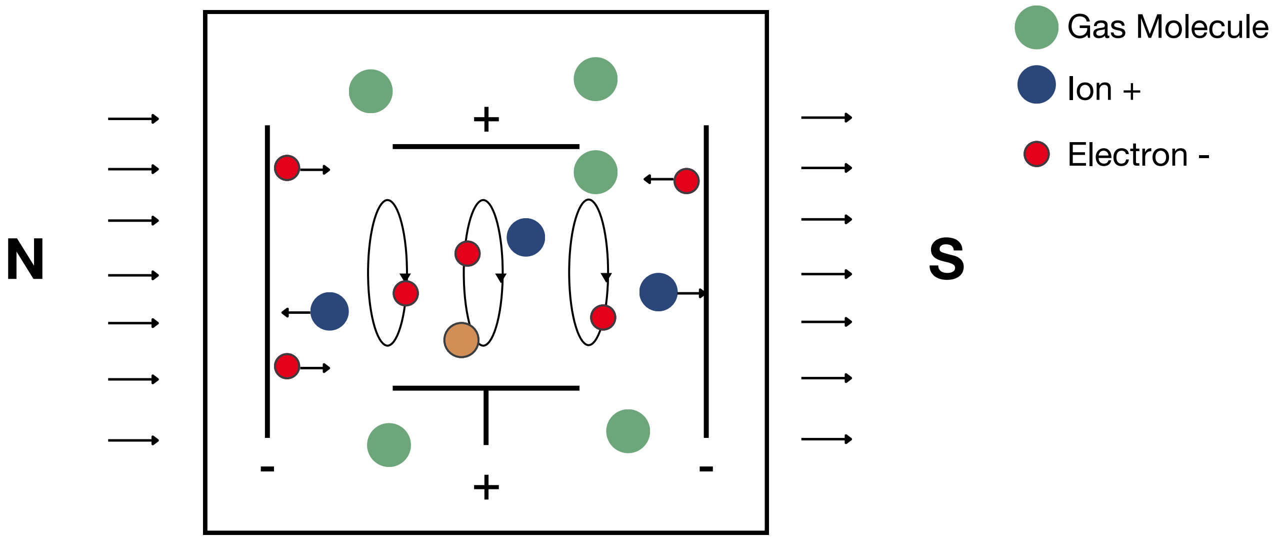
In terms of measuring pressures in HV, UHV and XHV, traditional pressure/vacuum gauges are unsuitable due to their restricted operating range. As a result, **ionization gauges are employed** instead: these use the probability of gas ionization to determine the particle number density.
There are two types: cold and hot cathode ionization gauges.



Cold cathode ionization

A cold cathode ionization gauge, often referred to as a Penning gauge, initiates a plasma discharge between two electrodes. The high voltage difference up to 3 to 4 kV accelerates the electrons from the cathode to the anode, as it does so the electrons hit gas molecules and form positive ions. The electric field lines are crossed by a strong magnetic field which confines the electrons to a spiral path. This extension allows enough collisions to occur to maintain the discharge current. The number of “charge carriers” increases with pressure, and by measuring the charge, one can determine the pressure (or vacuum).
For example, for a PR35 Penning gauge 0.1 mA equates to 10^{-2} mbar, whilst 0.01 mA equates to 10^{-5} mbar.

Cold cathode ionization gauges can measure vacuum conditions from 10^{-2} to 10^{-9} mbar. They are sturdy and easy to service, and although gas type dependent, correction factors are available. However, their measurement uncertainty is between 30 and 50% and, furthermore, cold ionization gauges prefer starting the discharge in a pressure range between 10^{-2} and 10^{-5} mbar, due to ionization probability being reduced as it is dependent on particle density (i.e. pressure).

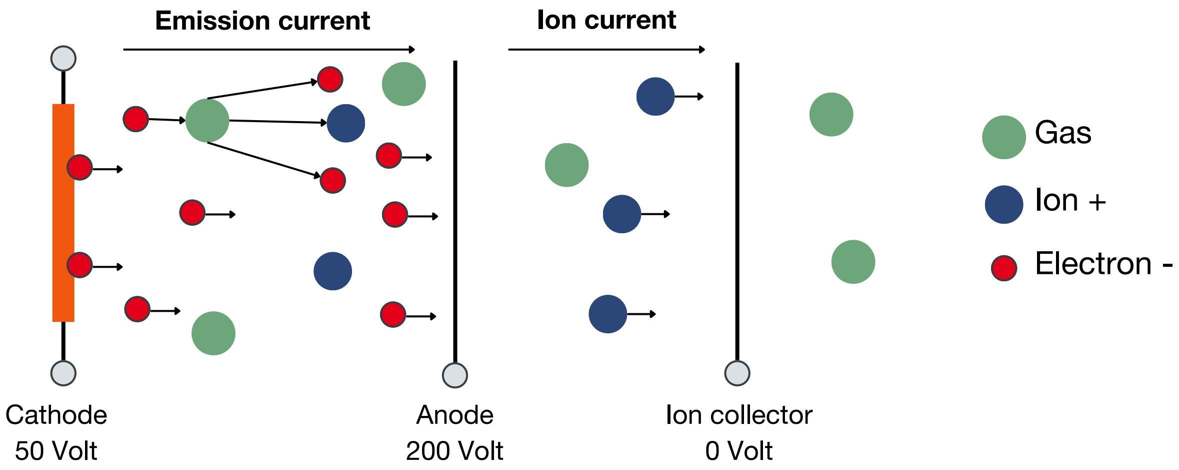


Hot cathode ionization

The hot cathode ionization gauge initiates a constant electron flow from the glowing cathode (electron source) to the anode (electron drain). These electrons hit a pressure dependent quantity of gas molecules, which become positive ions, causing a pressure related current to register on the ion collector.

Although the measuring range of a hot cathode ionization gauge is between 10^{-2} and 10^{-11} mbar, when the particle density is too high, the positive ions cannot predictably reach the ion collector. To extend the range, a Pirani (transducer) gauge is usually employed at the pump start point (when the particle density is high).

Furthermore, the lower limit of hot cathode ionization gauges is due to the X-ray limit. The electrons emitted from the cathode impact on the anode causes a release of photons (soft X-rays) which triggers electrons to be emitted from the ion collector surfaces, causing an offset current. To overcome this an "extractor" system has been developed which reduces the X-ray effect by shielding the ion collector. The extractor system, by focusing the positive ions, maintains the ion current at an acceptable level and, as a result, extends the measuring range from 10^{-4} to 10^{-12} mbar.



Whilst hot cathode ionization gauges display reliability, are gas dependent (although correction factors are available), and have a low measurement uncertainty (only about 10%), they do suffer from two disadvantages: it is necessary to prevent depositions/ vapor/dust contamination, because this will influence a cathode's lifetime and cause increased measurement errors; and, it is likewise necessary to prevent vibrations/shocks/ strong gas flows, as these will stress the filaments and reduce their lifetime.
To further facilitate their usefulness in the HV, UHV and XHV ranges, most hot cathode gauges are equipped with a degassing function. By using a higher potential on the anode and a higher emission current, the desorption rate rises, which means faster degassing when trying to reach an ultimate pressure.

Leak detection in High, Ultra & Extreme High Vacuum

No vacuum device or system can ever be absolutely vacuum-tight and indeed it does not actually need to be. The simple fact is that the leak rate should be low enough so that the required operating pressure, gas balance and ultimate pressure in the vacuum container, are not unduly influenced. In terms of HVs, XHVs and UHVs, it is small leaks that are the main cause of concern, and the only credible method to detect leaks smaller than 10^{-7} mbar.l/s is with a helium leak detector (HLD). A leak diameter-equivalent of 10^{-12} mbar.l/s (which equates to 1Å) is also the diameter of a helium molecule, and is the smallest leak rate that can be detected. This association with helium is one of the reasons why one of the most accurate and rapid leak detection methods employs helium as the tracer gas, and a mass spectrometer for the analysing/measuring.

HLDs work in the following way: the unit being checked is either pressurised from within, or without, with helium. The gases from any potential leaks are collected and pumped into the mass spectrometer for analysing, and any value above the previously determined background trace of helium is evidence of a leak. The mass spectrometer is the most sensitive leak detection unit available, and the one most widely used in the industry. In principle, it is possible to detect all gases using mass spectrometry, however, helium as a trace gas has proved to be especially practical. The reasons for using helium for leak detection are compelling:

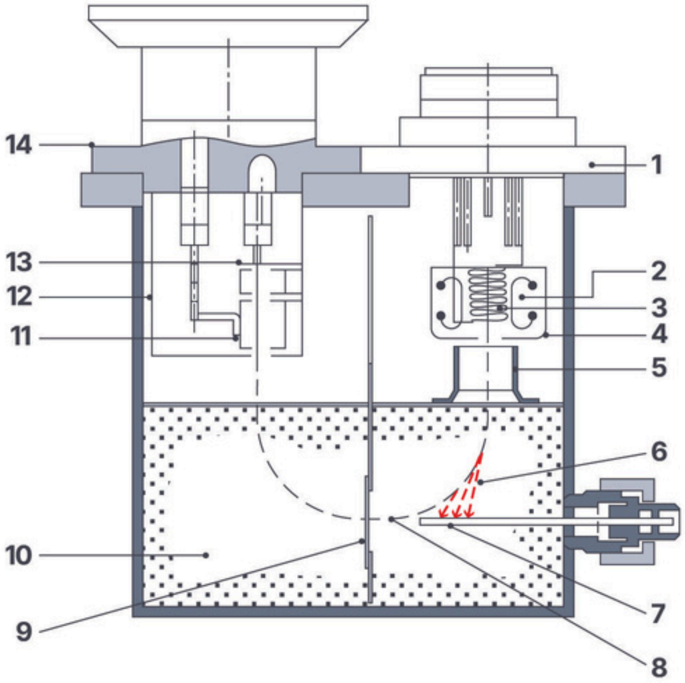
- it is unequivocally detectable by a mass spectrometer
- it is light it is chemically inert
- it is non-explosive
- it is non-toxic (although it is an asphyxiant)
- it is present in normal air in a concentration of only 5ppm
- and it is relatively economical/readily available

As for how helium is actually employed for detection purposes, there are two ways:

- the **“integral” testing method** requires the chamber to be placed inside a gas-proof unit (not always a possibility),
- whilst in the **“local” testing method** the chamber is either internally pressurized with helium or internally evacuated (with helium being generously sprayed onto the surface of the chamber at likely leak points).

In both tests, helium enters via possible leaks and passes to the mass spectrometer for analyzing.

The mass spectrometer works in the following way: any helium molecules entering into the spectrometer will be ionized, and these helium ions will then “fly” into the ion trap where the ion current is measured, allowing the leak rate to be calculated.



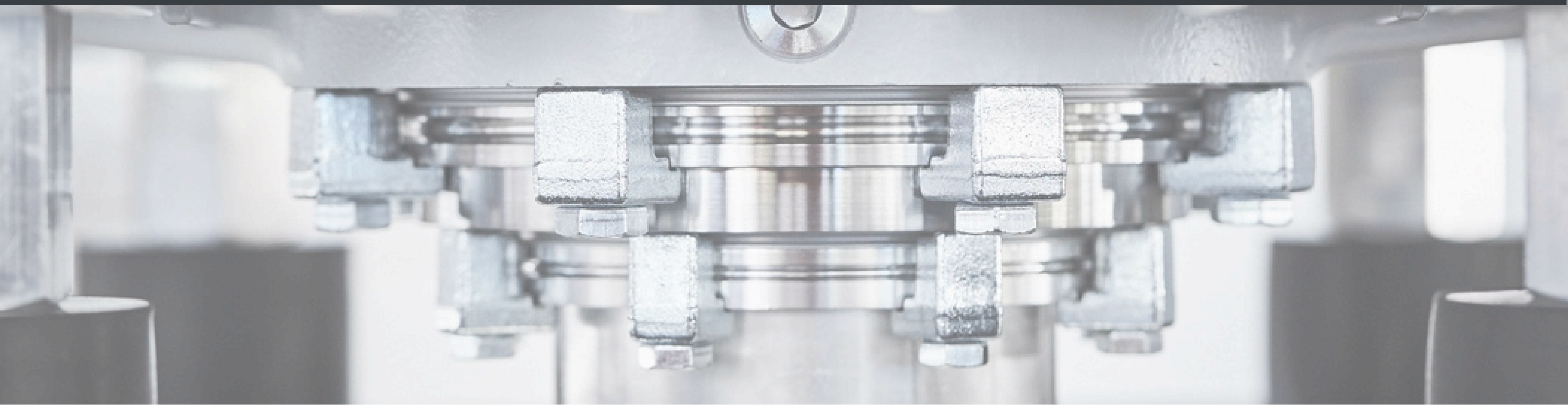
Ion Source (1-4):
1 Ion source flange
2 Cathode (Two Cathodes Ir +Yt_2O_3)
3 Anode
4 Shielding ion source and aperture

Deflection system (5 - 9):
5 Extractor
6 Ion path M > 4
7 Total pressure electrode
8 Ion path M = 4
9 Intermediate aperture

10 Magnetic field (vertical to drawing)
11 Suppressor
12 Shielding of ion trap
13 Ion trap
14 Flange of ion trap with pre-amplifier

However, care needs to be taken to account for any background readings (or from the degassing of helium from metal surfaces) as these will have an inordinate amount of influence (and accuracy) on the results from any small leaks detected. However, these are not the only challenges. HLDs are exceptionally sensitive, and surrounding or entrapped helium can easily impact the accuracy of leak detection and measurement.

Use cases & Applications



HV conditions are typically needed in industrial and research applications, including – but not limited to – composite plastic molding, flight instruments, vacuum tubes, medical applications, coating, mass spectroscopy, electron microscopy and much more.

UHV conditions, on the other hand, are used in different application areas.

A major area of use is related to **surface analytical techniques**, for example, as X-ray photoelectron spectroscopy (XPS), auger electron spectroscopy (AES), secondary ion mass spectrometry (SIMS), and thermal desorption spectroscopy (TPD), angle resolved photo-emission spectroscopy (ARPES), field emission microscopy, field ion microscopy, as well as atom probe tomography (APT). In addition, they are used in thin film growth and preparation techniques where there are very stringent requirements for purity, such as in atomic layer deposition (ALD), pulsed laser deposition (PLD) and molecular beam epitaxy (MBE).
For all these applications UHV conditions are necessary to reduce surface contamination. In this respect, it is important to appreciate that at 10^{-6} mbar, it only takes 1 second to cover a surface with a contaminant, so much lower pressures are needed for longer experiments.

UHV is also required in **research applications** including particle accelerators (like the Large Hadron Collider (LHC) where parts of the baked sections operate at UHV resp. XHV levels down to the 10^{-12} mbar range) and gravitational wave detectors (such as the LIGO experimental apparatus which is housed in a 10,000m^3 vacuum chamber at 10^{-9} mbar). Here UHV conditions will mainly help to reduce beam-gas interactions and limit perturbations from the outer environment.

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