by
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Introduction

The first thing you need to know about vacuum was documented in 16th century Italy:

"Nature abhors a vacuum."
--- Francois Rabelias, *Gargantua* (1534)

Yes, creating a vacuum is a struggle against nature, and the greater or more pristine the vacuum, the more difficult and expensive it is to produce and maintain. That having been said, vacuum science is *crucial* for advances in current scientific, technological and industrial processes.

Figure 1 - An ultra-high vacuum chamber being evacuated with a turbomolecular pump.

What is a vacuum? Looking at Webster's New World Dictionary: *vacuum, n.* **1.** a space with nothing at all in it **2.** a space from which most of the air or gas has been taken. We reserve the first definition for the philosophers and always refer to a vacuum according to the second definition, as a chamber or vessel with as much of the air and residual gasses removed as practical.

Why do we need vacuums? There are tens of thousands of processes that require some type of vacuum but, since you are reading the Advanced Design Consulting, Inc. catalog, you probably need this gas-free environment to avoid absorption of photons, electrons or particles passing through your chamber. Maybe you also need a vacuum to protect your sample from contamination and reaction with atmospheric gasses while you analyze it with some kind of photon, electron or particle probe. Possibly, you need a vacuum to insulate your sample from thermal convective heating or cooling, while containing or processing it at very high or very low temperatures. You may be evaporating metals or other substances onto substrates and require a vacuum so that the vapor stream is not dispersed or reacted by the time it reaches the target. You may be trying to pull volatiles from a solid, or dissolved gasses from a liquid or a melt. There are thousands of uses for vacuums at all levels. The ones mentioned above are just a sampling from the world-wide project list developed for academic, industrial and governmental laboratories by Advanced Design Consulting, Inc.

How do we create a vacuum? I explain to my non-technical friends that the molecules of air are like floating ping-pong balls and a vacuum pump is like a rotary fan. The fan blades are angled so that if a ball comes into contact with a fan blade, it is given a 'whack' and propelled away in a pre-determined direction. Balls that try to come back the wrong way against the fan are given another 'whack' and returned in the downstream direction. If you seal that fan into the doorway of a closed room, blowing outward, pretty soon most of the balls or air molecules are redirected outward from the room. If you make that room air tight — make it out of stainless steel, for example — and you make that fan very powerful — a jet turbine engine, for example — you quickly have an extremely low pressure (low number of ping pong balls) in the room, in other words, a vacuum. On a smaller scale, the stainless steel room with a turbine engine pump is *exactly* what we use to make ultra-high vacuum except that we call the room a vacuum chamber and the jet engine a turbo-molecular pump! (See Figure 1)

Basic vacuum concepts

Pressure

One atmosphere pressure, ~14.7 pounds/in^2 (English units), is not that great in human terms. It is about the floor pressure you feel if you weigh 175 pounds and you stand on the balls of your feet. Considering that submarines and humpback whales routinely ocean dive to 200m, or the equivalent to 20 atmospheres pressure and that we routinely put over 200 atmospheres pressure in those gas bottles that helium balloons are filled from, it is not a long way in pressure terms between 1 and 0 atmospheres pressure where zero pressure is perfect vacuum. (Don't be confused by vacuum terminology, the lower the pressure, the higher the vacuum.)

No, measuring sub-atmospheric pressure or vacuum is more about getting an indication of how many gas *molecules* still remain in the volume of our vessel. The fewer the molecules, the further any one molecule can travel before colliding with another gas molecule. This collision distance is known as the mean free path length.

Pressure Scales

There are at least twenty pressure scales used by various scientific disciplines, engineering specialties and technical trades. Definitions and inter-conversions between the various units can be found in IEEE/ASTM Standards.[i] Conversions are done most easily if you use your PC and download one of the readily available unit conversion programs. You can obtain a low-cost shareware version called *Qvert*[ii] that conforms to the IEEE/ASTM standard or a freeware version called *Convert*[iii]. Table 1 lists conversion factors from the eleven most common units to the three preferred by vacuum scientists; Pascal (Pa), torr and millibar (mbar).

TABLE 1
Conversion Factors Between Common and Preferred Pressure Units

	Multiply by:	*Converting to:*		
		Pascal (N / m2)	torr (1/760 std. atm.)	millibar (mbar or mb)
Converting from:				
S.I. Unit	Pascal		0.0075	0.010
Metric Units Traditional	torr	133.322		1.333
	millibar	100.000	0.750	
	dyne/cm2	0.100	0.00075	0.001
	kgf/m2	9.807	0.07356	0.098
	mm of Hg	133.322	1.000	1.333
	bar	100,000.000	750.064	1000.000
English Units Traditional	atmosphere	101,325.000	760.002	1,013.250
	psi	6,894.757	51.715	68.948
	ksi	6,894,757.00	51,715.073	68,947.570
	inches of H_2O	249.089	1.868	2.491

It is preferable to use the International System of Units (SI) adoption of the Pascal (Pa) but the torr (where 1 atmosphere = 760 torr = 760 mm Hg) has been in use since Galileo's time[iv] and is hard to surrender.

All you need to know to be conversant about the vacuum pressure scales is included in Figure 2. You see that all scales are logarithmic, where each decade indicates a ten-fold improvement in vacuum, roughly a ten-fold decrease in gas density, and roughly a ten-fold increase in molecular mean free path length. Torr and mbar scales are essentially the same. (1 mbar = 0.75 torr) Multiply by 100 to convert to Pascal. Usually, we simply refer to our vacuum systems as rough vacuum, medium vacuum, high vacuum (HV), ultra-high vacuum (UHV) and extreme-high vacuum (XHV), as indicated at the right of the figure. The exact limits of each of these pressure ranges is not well defined but rather, are indicative of a general *quality* of vacuum. The better the vacuum, the greater the expense, the greater the cleanliness and the fewer the choices for system materials there are. Just remember, although it looks quite impressive that we traverse *sixteen orders of magnitude* on the torr scale, we are only actually crossing the pressure range from 1 atmosphere, 14.7 psi, to the vacuum of interplanetary space, effectively the best vacuum we have ever measured, which we'll arguably call 0 psi.

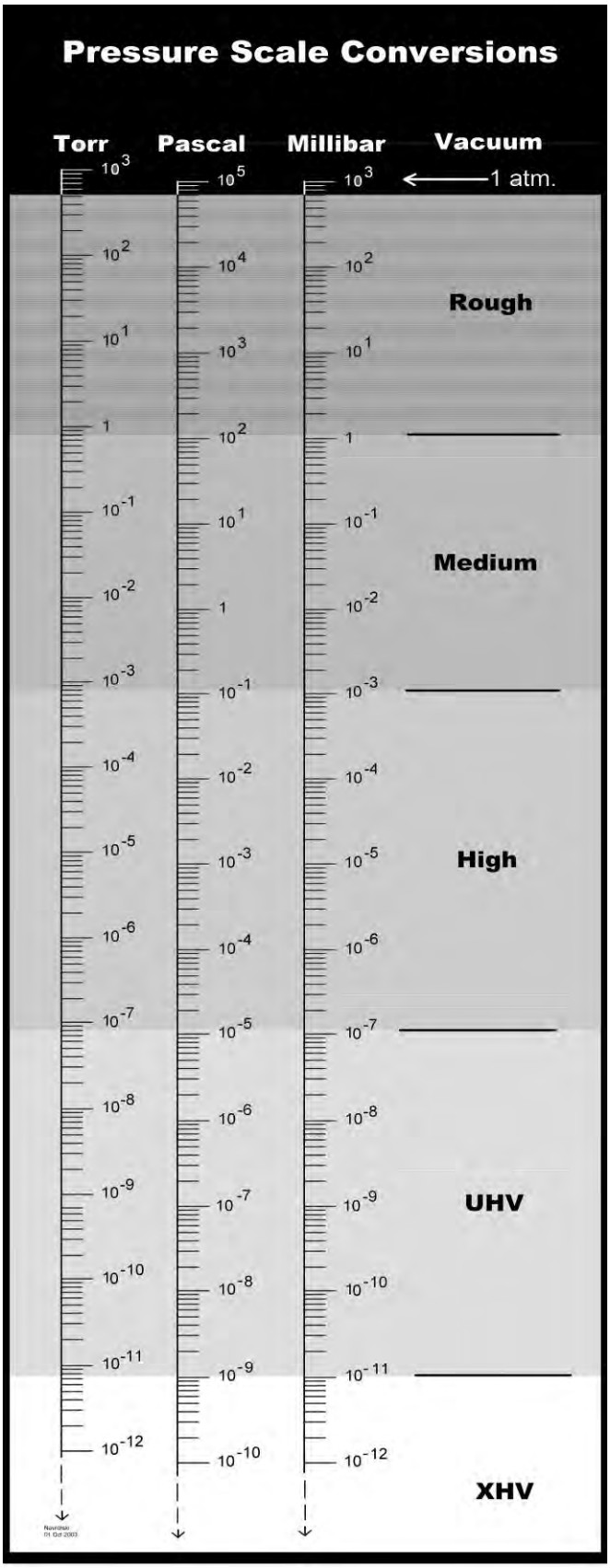


Figure 2 - Pressure Scale Conversions

Pressure Measurement

There are many different ways to measure vacuum levels. Each technique has strengths and weaknesses and each works best over some range of pressures. If you are interested in the nuances of pressure measurement, look at the Leybold technical reference section[v]. As an introduction, it is superior even to the standard text books listed at the end of this primer.

Figure 3 - Mechanical Gauge (Bourdon type)

Here is what you need to know for the vast majority of your vacuum measurement tasks. If measuring atmospheric pressure to rough vacuums; a simple dial gauge is sufficient. These operate by measuring the deflection of a diaphragm or the unwinding of a spiral tube. The physical movement is translated via a mechanical linkage to a dial indicator calibrated for this vacuum range. Fancier gauges measure these mechanical deflections with electronic methods.

Measuring rough and medium vacuums is economically done with thermocouple gauges. These gauges are simply two crossed dissimilar metal wires immersed in the vacuum. Current is passed through two legs of the circuit and two legs measure the temperature of the wire junction. The constant current heats the junction and since heat is removed via thermal conduction, air convection and radiation, any change in air pressure changes the convection loss channel and thus the junction temperature. Temperature and air pressure have a direct correlation. Improved versions of these gauges are known as Pirani Gauges.

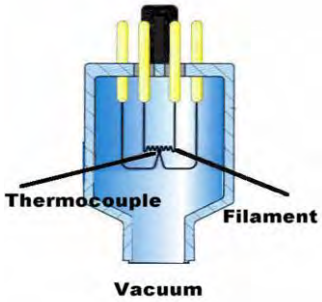


Figure 4 - Thermocouple Gauge

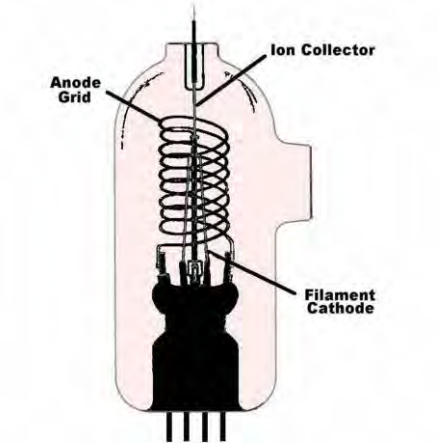


Figure 5 – Hot Cathode Gauge (Bayard-Alpert type)

Measuring HV and UHV is economically done with cold cathode gauges sometimes known as Penning gauges. These are anode/cathode-constructed gauges within a strong permanent magnet. If a cosmic ray or natural radioactive decay event ionizes a gas molecule in the gauge, it spirals into the cathode, ionizing other gas molecules along the way. The current measured in the system is an indication of the molecule density and thus vacuum level.

Measuring UHV and XHV is economically done with hot cathode or Bayard-Alpert gauges. These gauges have a white-hot filament to ionize trace gasses. The resulting ion flow between cathode and anode creates a measurable current indicating vacuum level.

Vacuum Pumping

The first vacuum pump, invented by Otto von Guericke in 1650, was built on the manual piston and bellows assembly used for fire engine pumps of the day. Evangelista Torricelli, a disciple of Galileo Galilei, had already discovered air pressure and vacuum in 1644. Nevertheless, it was von Guericke who changed the pump direction to evacuate air-filled receptacles. His most famous demonstration of vacuum (air pressure) was with his "Magdeburg hemispheres", two tightly fitting brass half-spheres which, when assembled and evacuated, couldn't be pulled apart by sixteen horses. He conducted many other demonstrations of the existence of vacuum, showing that bells will not ring, candles can not burn, animals can not live and that water will flow up into a vacuum chamber. (Figure 6)

All vacuum pumps work by redirecting gas molecules out of the chamber to be evacuated and not allowing them to re-enter. The key physical concept is having a method to *transfer momentum* to the gas molecule and direct it out of the system. Mechanical pumping systems use arms, rotors, pistons vanes, diaphragms, plungers, turbines, interlocking scrolls, rotating cylinders, etc. to accomplish this momentum transfer. When an air molecule comes into contact with the mechanical device, it is redirected out of the system and can not re-enter.

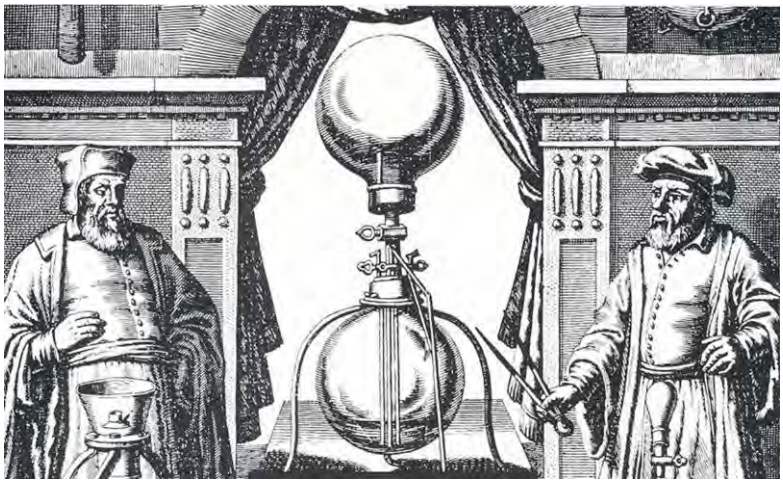


Figure 6 – Illustration from Otto von Guericke's early vacuum experiments, *Experimenta Nova (ut vocantur) Magdegurgica de Vacuo Spatio* (ca. 1672) courtesy AVS archives.

It doesn't have to be a mechanical device initiating this momentum transfer; it can be a fluid or a gas as well. Have you ever used a hose to spray debris off your sidewalk? You can pump air in a similar fashion by using a high pressure stream of steam as the pumping mechanism; this is called a steam ejector pump. For years, the best vacuums were produced by a stream of boiling oil or mercury atoms called a diffusion pump. (see Figure 7)

There is another clever way, using the analogy in Section 1, to remove ping-pong balls (air molecules) from a room. Let's say that I am standing in the room, ping-pong balls flying around creating pressure on the room walls, and I slowly unroll a big piece of duct tape. Balls hitting the sticky tape are trapped. They are still in the room but are no longer free to strike interior surfaces and create pressure. They are just lying on the floor, stuck to the tape. This is the concept for sorption, cryo, getter, sublimation and ion pumps. Sorption and cryo pumps rely on liquid nitrogen cooled, high surface area, pellets or plates as the 'sticky' medium. Getter and sublimation pumps rely on a reactive surfaces that form chemical bonds with gas molecules to trap them. Ion pumps take a gas molecule, strip an electron from it and accelerate it under high potential until it slams into an anode plate where it is physically embedded and trapped. In all cases, the molecules are effectively removed from the chamber volume.

Every variety and variation of pump has strengths and weaknesses. Each pumps the different atomic and molecular species present in air with different efficiencies and speeds. Each operates best in a particular vacuum range and we usually chain different pumps in series or swap them out in parallel to attain the desired vacuum level.

If you are interested in exploring this fascinating area of technology, take advantage of the following resources. First, the American Vacuum Society[vi] and similar societies worldwide[vii] routinely offer multi-day courses on the subject, usually associated with society meetings. Details on the wide variety of pumps and their theories of operation, accompanied by excellent illustrations, are to be found in the catalog reference section of one of the major vacuum vendors, Leybold[v] and in their stand-alone pocket reference, the *Vacuum Vademecum* (Figure 14 of the reference section).

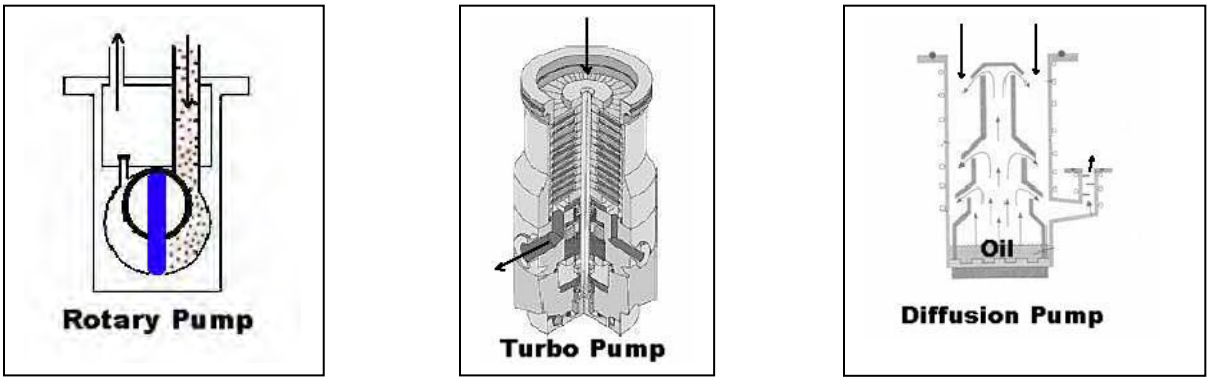


Figure 7 – Three types of vacuum pumps; rotary (mechanical), turbomolecular (mechanical) & oil diffusion

Pumping speed is a concept of which you need to have a firm grasp. As you can guess, each type of pump comes in different sizes. If you spend lots of money and purchase a huge pump to evacuate a small volume, then your system can afford to have many sources of leaks and you will still achieve vacuum levels appropriate for that pump, at least for awhile. Another concept is that of conductance, essentially the size of the tube through which you do your pumping. Imagine trying to pull air through a straw versus through a large pipe. The large pipe allows easier pumping, because it has larger conductance. In addition to the size of the pipe through which you are pumping, its length and number of bends also affect conductance in an easily calculable way. The combination of pumping speed and the system conductance determines how fast your system can be evacuated. There are good examples of manual calculations and programs for automatic calculations of conductance, pump-down, effective pumping speeds and gas loading at Kurt Lesker Company's technical information website.[viii]

Vacuum Design and Fabrication

By good vacuum design, we are talking not only about creating a vacuum chamber without leaks, and that is constructed from proper vacuum materials, but also we are also talking about the mechanical aspects of building vacuum equipment. Figure 8 shows two particularly common mistakes made in poorly designed vacuum equipment. The top example shows how a common screw inserted into a blind tapped hole creates an un-pumpable air pocket that acts like a continuous virtual leak. In this case, your vacuum instrumentation tells you that air is entering your system, preventing it from achieving low pressure. Your vacuum guru will use techniques like helium leak checking (see Section 2.6) to try to find the air leak from outside of the system, but to no avail. All the while, the trapped air volume beneath the screw is slowly creeping along the screw threads, contaminating your system. The bottom example shows a long planar contact between two abutting plates created when the vacuum vessel was outside welded. These adjacent surfaces are filled with trapped adsorbates. If you had cleaned, acid etched or electropolished the inside of this vessel as a good UHV technician should, those solvents wick into this crevice. This kind of virtual leak will continuously contaminate your vacuum and requires heroic measures to remove.

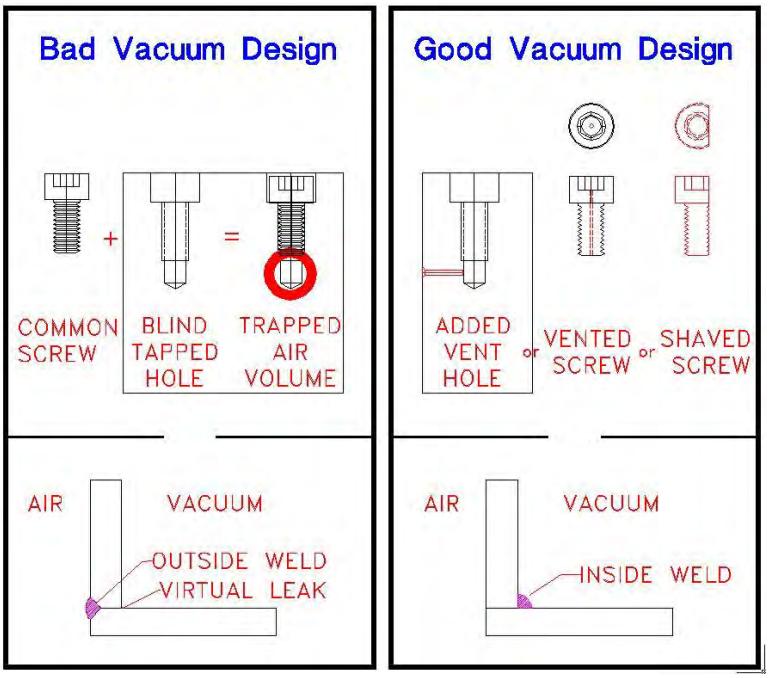


Figure 8 - Examples of bad and good vacuum design practice.

To paraphrase an old saying: good vacuum designs come from experience — experience comes from bad vacuum designs. You must simply look at every part, every hole, every connection, and ask, 'have I trapped un-pumpable contamination in this component?'.

One final tip, for any component that is being made for high vacuum or better: insist that your machinist avoid cutting and tapping fluids containing sulfur (most machining fluids do). Any residue that makes it past your cleaning procedure will outgas and contaminate your chamber. Sulfur is a particularly bad vacuum element. You will find yourself with no other choice but to dismantle and clean everything in your chamber to eliminate this oil residue. Dismantle everything? Yes! As you pump a chamber with contaminants, they release into the vacuum. At low pressures, these molecules have long mean free paths, so they are free to move to every corner of your clean chamber and uniformly contaminate all components. Things in directly line-of-sight have higher contamination levels but, if these molecules do not stick to line-of-sight surfaces with 100% probability (we call this a sticking coefficient of 1), they ricochet around in your chamber until they do stick to something else.

Vacuum Leak Checking

If you are going to do any significant HV, UHV or XHV work, you *must* have access to a vacuum leak checker, because sooner or later, you *will* have a vacuum leak. (Now that these words 'vacuum leak' appear on paper, they seem to be an oxymoron: vacuum can not leak out of your chamber, it is air that leaks into the vacuum. Nevertheless, the term 'vacuum leak' is commonly used everywhere.) The leak checker is one of two indispensable tools for diagnosing chamber pump-down and pressure problems. (The other will be discussed in Section 2.8.)

Here is how it works: the leak checker consists of a vacuum pumping system and a special sensor that looks for the presence of helium in the chamber gasses. As you evacuate your leaky system with the leak checker's pump, you take a hose and gently direct a small stream of He against suspect areas on the outside of your chamber. If there is a leak path, the He is drawn into the vacuum chamber, pumped out by the leak checker, the sensor detects the presence of He and alerts you, usually with an audible signal. You have pinpointed the source of your leak by probing with the helium hose without contaminating your system.

Why helium? First, normal atmospheric air only contains 0.00052% He by volume naturally, so your sensor is looking for an element not normally found in your vacuum system. Second, helium is a very light, inert molecule that can make its way through the smallest leak paths. Third, good purity, dry helium is readily available in pressurized bottles at nominal cost.

The sensor for selectively detecting He is also of straightforward and rugged design. Here is how it works. Some of the gasses being pumped are passed over a hot filament and ionized. (See Figure 9). The positive ion is accelerated by some potential, V, into an area with magnetic field, **B**. A moving charge (the ion) in a magnetic field will be deflected by an amount that depends on its charge, q, mass, M and velocity, V, according to the equation shown below right. So, the mass of He is known and we fix **B**, V and q. We put a hole at the calculated distance x for He and look at what comes out with an ion collector. If the mass of the ion is larger than He, it will be deflected to a longer distance x_1 and will not make it out of our hole. The only atom lighter than He, hydrogen, will travel a shorter distance than x, and again, not make it out of the hole. So, with this selector, we only get a signal that depends on how many He ions are seen. Thus…. the He leak detector!

$$x = \sqrt{\frac{8VM}{B^2 q}}$$

There are eight common units used for leak rate measurement, but the most widely accepted are: torr-l/sec, mbar-l/sec and the S.I. standard, Pa-m^3/sec. Section 3 details specific leak rates tolerable for each vacuum range.

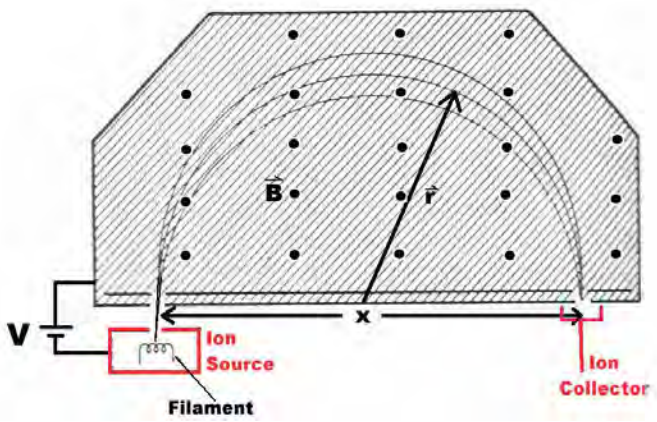


Figure 9 - Principles of a helium leak detector sensor.

Sources of Residual Gasses in Vacuum

When you evacuate a chamber, the first thing that you do is pump out the volume of gas contained in your vacuum chamber, vessel or beam pipe. There are lots of gas molecules (Table 2) to evacuate and the out-rush of gas propels other molecules encountered along the way in classical, fluid dynamic, viscous flow. Pumping in this pressure region is relatively easy, fast and inexpensive to do. Many of the non-reactive gasses that would be difficult to pump at low pressures are caught up in the gas flow stream and efficiently evacuated at these near atmospheric pressures. You can quickly obtain vacuums in the 1 torr (100 Pa) range with simple pumps.

Table 2: Air Composition
(at 50% RH and S.T.P.)

Gas	Partial Pressure (torr)	% by Volume
N_2	594	76.858
O_2	159	20.618
H_2O	11.9	1.57
Ar	7.0	0.919
CO_2	0.25	0.033
Ne	0.014	0.0018
He	0.004	0.00052
CH_4	0.0015	0.00020
Kr	0.00084	0.00011
H_2	0.00038	0.00005
N_2O	0.00038	0.00005
Xe	0.000066	0.000009
Ozone	0.000053	0.000007

Getting to lower pressures however, becomes more and more difficult for the following reason: "Nature abhors a vacuum." As you evacuate your vessel, to rough and medium vacuums, gas molecules become more rarified and the vastly reduced out-rush of air no longer helps with the self-pumping process. At these pressures, about 1 torr to 1 millitorr (10^2 to 10^{-1} Pa), a transition flow regime called Knudsen flow dominates. Additionally, atmosphere wants to re-enter: via leaks in the vessel's joints and welds, by permeating seals and valves and even by permeating defects in the material of the vacuum vessel itself. These leak sources must be overcome with good tight chamber design and construction.

Now you are below the 10^{-3} torr (10^{-1} Pa) vacuum range and into the molecular flow region. This means that, in order for further pumping to occur, the individual gas molecules must wander over to the pump mechanism, via the normal Brownian motion collision process, in order to be evacuated from the system. There is no longer a stream of gas to direct air molecules toward the pump. It is a random collision process that brings any individual particle into contact with the pump mechanism and allows it to be removed from the system.

At these vacuum levels, other processes begin to dominate nature's drive to fill your vacuum. Here is a *key concept* and a starting analogy for high vacuum levels and beyond. If you could put an open galvanized (i.e. zinc-coated) steel trash-can full of water into the vacuum of space, the water would quickly boil away even at room temperature and lower. This is because the vapor pressure of water (Figure 10) is much higher than the pressure surrounding the can. So the water vaporizes into the vacuum, filling it uniformly with its water molecules and effectively raising the pressure in the surrounding space. When all of the liquid is gone, there is still a thick layer of adherent water and gas molecules adsorbed on the surface of the can, held there by van der Waals forces. This layer desorbs into the vacuum at a slower rate than the liquid, depending on vacuum level and temperature. If the can has surface cracks or is slightly porous, air and water molecules are even more tightly absorbed in these micro-pockets in the bulk of the metal but will eventually migrate to the surface and into the vacuum. (Note: the proper use of the word absorbed would be that water molecules are held in the atomic structure of the bulk metal. There is an important distinction between the words: *adsorbed*, surface bound, and *absorbed*, bulk material bound.) At an even slower rate, the zinc metal coating on the surface of the galvanized can vaporizes and flies off into space, filling it uniformly with Zn atoms. Finally, at an extremely slow rate, the iron atoms of the base metal of the trash-can have their own vapor pressure and slowly fly off into the vacuum. This exact same process happens with *all materials*, liquids, polymers, metals, ceramics, glasses, minerals, etc. to greater or lesser extents, depending on the material's atomic composition, electronic configuration, phase, surface area, physical configuration and the vacuum level to which it is subjected.

So, as you try to pump into the high vacuum (HV), ultra-high vacuum (UHV) and extreme high vacuum (XHV) regimes, these new sources of chamber gasses — surface outgassing, bulk outgassing and bulk material vaporization — start to dominate the composition of the remaining gasses in your system. In addition, gasses coming from virtual leaks (the surface cracks and pores of our trash-can analogy) and from insidious contamination (fingerprints, grease, oil, machining lubricants, old banana peels, etc.) take on great importance. In Table 3, there is a listed grouping of elements and compounds by vapor pressure at room temperature[ix]. As you go to better and better vacuum you must start further and further down on the list and use only the materials there and lower on the table. Otherwise, just as in the galvanized trash-can example, the bulk material will vaporize and contaminate your vacuum.

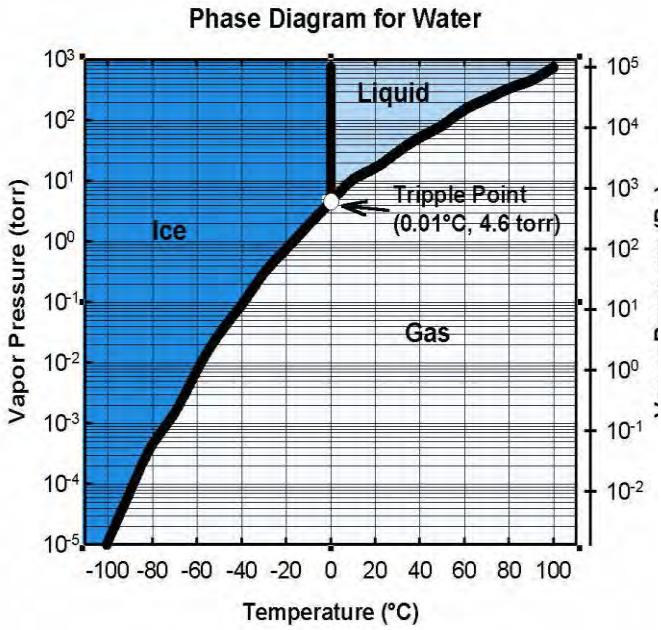


Figure 10 – Vapor Pressure (Phase Diagram) for Water.

Now that you have a relative feel for vapor pressures of different materials, you must understand that the *vapor pressure of each material increases rapidly with increasing temperature*. Just as in a tea kettle, where the hotter the water gets the more steam evolves; the hotter a solid gets, the higher its vapor pressure. As you go to higher and higher temperature, you must start lower and lower on Table 3 to maintain the desired quality of vacuum. For example, if you want a crucible to vaporize material in your high vacuum system, the parts that will be getting extremely hot will have to be made of the materials in the XHV portion of the table, the parts holding those parts and getting hot will need to be made of materials in the UHV or XHV portion of the table, and so on.

Some exceptions to the rules illustrate that not only is the choice of material important but the surface configuration of that material, i.e., the part that directly contacts the vacuum, can have a drastic effect.

Example 1:

A typical mistake made is putting brass metal parts into vacuum. Brass is an alloy of ~65% Cu and ~35% Zn. So you look at Table 3 and decide Cu is great for UHV and Zn is O.K. in HV so brass should be acceptable. As measured however, brass selectively outgasses Zn at about twice the rate of a piece of bulk Zn metal! (Do not put bare brass into HV or better.) The author has had occasions where the easy machinability has made brass the prime material for certain complex components so here is a trick. Brass can be chrome plated completely pinhole-free and Cr is an excellent UHV metal that completely encapsulates and protects the brass from contact with vacuum. Cr-plated brass parts have been known to function wonderfully in 10-6 torr (10-4 Pa) at room temperature without vacuum contamination for years.

Example 2:

For extreme vacuums, it would look like stainless steel (mostly iron) is a superior material to aluminum. Again, a surface treatment can make a great deal of difference. Stainless steel, polished and baked, has an extremely low outgassing rate, that is the amount of gas released into a volume at a given vacuum per unit surface area. Many aluminum alloys, similarly polished and baked, form a natural barrier oxides and have outgassing rates 100X lower than the best vacuum prepared stainless steel materials. A good source of further information on vacuum materials can be found in an article by B.S. Halliday "An Introduction to Materials for Use in Vacuum",

Table 3: Vapor Pressures for Different Materials

(Combined Vapor Pressure Groupings at 30 °C)

Acceptable Vacuum:	Element, Compound or Material
Never in Vacuum	HF, CS_2, Br_2, H_2O, I_2, No corrosive gasses
	Any significant quantities of liquids, hydrocarbon solvents or detergents
Rough Vacuum	Hg, At, Fr, S, Cs, ,
	Fomblin Pump Oils, paints, PVC, tapes
Medium Vacuum	Rb, K, brass, 303 stainless steel
	High vacuum pump oils, plastics,
High Vacuum	P_4, Cd, Se, Na, As, Zn, Pb, Te, Mg, Li, Sr, Yb, Ca, Sb, Tl, Ra, Eu, Ba, Bi, Pb, Sm, In, Mn, Tm
	Ultra-high vacuum pump oils, silicate ceramics, kapton, cured polyimide coatings, common solders
UHV	Ag, Ga, Am, Dy, Ho, Er, Al, Cu, Sn, Be, Sc, Nd, Gd, Pr, Pu, Au, Pd, Cr, Ge, Pu, Tb, Lu
	silicon oils, viton elastomers, pyrex glass, macor ceramic, alumina ceramics, 304 & 316L SS
XHV	Fe, Co, Y, Ni, Si, Ti, Lu, Ce, Ac, U, V, B, Pt, Th, Rh, La, V, Ir, C, Os, Re, Ru, Mo, Nb, Ta, W, Zr, Hf, Tc

Original reference:
RCA Vapor Pressure Tables by R.E. Honing, et. al. (1957 to 1969).
Reproductions can be found at: http://www.mcallister.com/vacuum.html [ix]

Residual Gas Detection

Besides the leak detector, the other most useful instrument for diagnosing HV, UHV and XHV problems is the mass spectrometer. This instrument, inserted into your vacuum vessel, not only detect leaks, but also gives you an instantaneous readout of the total system pressure and, most importantly, the partial pressures of all the atomic and molecular species present in the chamber. If you understood the principles of the He leak detector sensor in Section 2.6, then it is a simple extrapolation to understand how this is accomplished. Going back to Figure 9 and its associated equation (rearranged at right), if you change the strength of the magnetic field, **B**, and everything else about the geometry of the sensor remains the same, you can make a different atomic mass pass through the hole located at x, correlated with the magnetic field strength $\mathbf{B}^2$.

$$\frac{M}{q} = \frac{B^2}{8V}x^2$$

So if you sweep **B**, and plot the ion collector output you have a graph of all of the partial pressures of all the elements. (Actually a plot of partials vs. mass/charge ratio.) A key concept that must be presented here is that of a cracking pattern. Remember that the gas molecules are ionized by a hot filament? Well, when a multi-atomic molecule is ionized, it can be stripped of one electron, broken into smaller pieces and even stripped of multiple electrons. Luckily, the probability of each of these occurrences for each type of molecule is well understood. For example, a water molecule, H_2O when ionized by a white hot filament can be 'cracked' into H_2O^+, HO^+, O^+ and H^+ ions with mass-to-charge (M/*q*) ratios of 18:17:16:2 respectively with relative abundances of 100:23:1:0.1. Each cracking pattern is like a fingerprint for the molecule that created it and this signature is invaluable when trying to unravel vacuum problems involving molecules with overlapping mass-to-charge partials. Tables of these cracking patterns can be found in mass spectrometer books and in O'Hanlon's text listed in the reference section.

Today, the cleverest way to produce an ion selecting field is not with a magnet, but with an electric field generated by a four-pole antenna. The sweeping electric field created by a radio-frequency generator causes ions with selected mass-to-charge ratios to spiral into a detector while other M/*q* ions divergently spiral away from the detector. The system is compact, requires no bulky magnets or electromagnets and is currently the state-of-the-art technology. These devices are known as residual gas analyzers (RGA's). They produce spectra like that shown in Figure 11, can be highly automated and accurate and are indispensable for vacuum diagnostics.

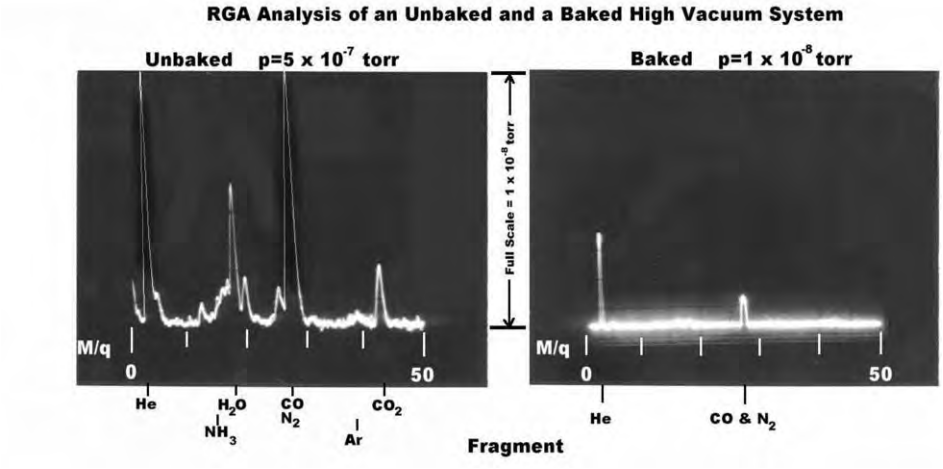


Figure 11 - RGA Spectra for an unbaked and baked HV system.

Keeping it clean

When working in vacuum, the cleaner the better. As you attempt to move your vessel, beampipe or chamber from rough vacuum through HV into XHV, even 18th century theologians knew:

"Cleanliness is indeed next to godliness"
– John Wesley (1703-1791)

For each increase in vacuum range, you have to increase your effort to maintain a clean, smooth, well-designed vacuum surface. "Clean" means simple surface wiping for rough vacuum, progressing to multiple-step ultrasonic cleaning and vacuum baking for UHV and XHV. "Smooth" means rust-, flux- and scale-free for rough vacuum, progressing to electro-polished, chemically smoothed and barrier oxide-treated for UHV and XHV. "Well-designed" means pinhole-free for rough vacuum, progressing to virtual leak-free, differentially pumped seals and hydrogen impermeable for XHV.

The best source for detailed information on vacuum cleaning is contained in an article in the Journal of Vacuum Science and Technology, "A survey of vacuum material cleaning procedures".[x] In it are detailed prescriptions for cleaning, baking and handling; stainless steel, aluminum, copper, ceramics, glass, molybdenum from major industries and government laboratories.

Section 3 will present a specific set of guidelines for each vacuum range. Remember, you can always achieve a poorer vacuum level by following cleaning precautions for a better vacuum level but the converse is not true.

Baking

Once you get into the UHV regime, your base pressure, that is, the lowest attainable pressure in your chamber with all chamber activity at a stand-still, is determined by the outgassing from the adsorbed water and atmospheric gasses on the vacuum surfaces of your equipment. You can eliminate this thick adsorbed water layer by heating the chamber above 100°C (120, 150 or 170°C are typical temperatures depending on the lab) while continuing to vacuum pump. We sometimes call this a low temperature bake-out or water bake-out. Typically, heating tapes, pads or blocks are attached to the stainless steel portions of the chamber and the entire assembly is blanket-insulated as in Figure 12. (This author particularly likes commercially available, internal, infrared bake out heat lamps for this task.) The temperature is raised slowly, taking into account the poor thermal conductivity of stainless steel, and held at 120°C until the measured internal pressure stabilizes for at least eight hours. Upon cooling, the chamber bottoms out to a new and lower base pressure now that the water and adsorbed atmospheric gasses have been removed.

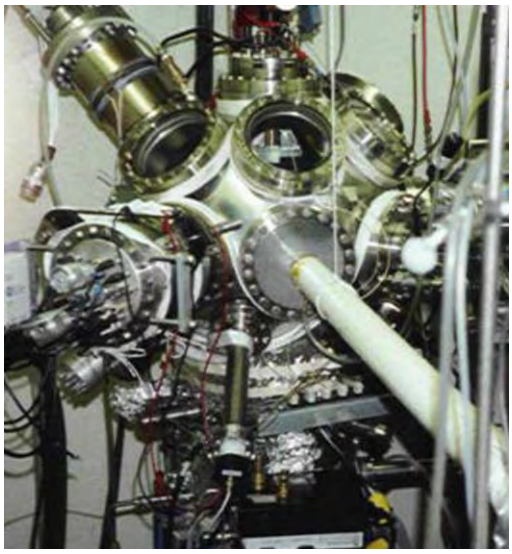


Figure 12 - The author's double-walled XHV chamber with white bake-out heater tapes exposed. Internal IR heating lamps do the bulk of the work of removing internal adsorbates.

As expected, the higher the bake-out temperature, the faster the degassing. For this reason, some labs conduct a high temperature bake-out in the range of 250°C to 750°C to quickly desorb surface water as well as more tightly bound water, adsorbed hydrocarbon contamination and bulk absorbed gasses,

especially hydrogen in stainless steel. Although very effective, care must be taken since glass viewports, welds and ion pump magnets on your chamber can be damaged at high temperatures.

There is no single correct method for achieving the ultimate vacuum, but you can gather the basics from the cleaning, fabrication and baking sections above. Each lab, facility and vacuum guru has their own particular bag-of-tricks learned from experience. In fact, along with each lab's idiosyncrasies, the author feels some measure of luck or even divine intervention is needed to get to low base pressures.[xi]

Venting

With all the talk about adsorbed water and atmospheric gasses being the primary hindrance to high and ultra-high vacuum, you should look for any method possible to keep your clean vacuum surfaces from contact with water, including humid atmospheric air.

One of the tips for keeping adsorbed water away from the inside of your system is to vent your chamber with dry nitrogen when it must be opened for maintenance or installation of a new component. If you open your chamber's vent valve directly to the air, the ambient humidity immediately forms a nice cloud as the air hits the low pressure environment inside. Of course, for rough or medium vacuum, this is not a high priority concern and venting to air, filtered air or even air pulled through a solid dryer material (like Drierite) is acceptable. But venting low HV, UHV and XHV chambers to atmospheric air is unacceptable. Most laboratories have bottles of dry nitrogen available to back-fill vacuum chambers. A danger is that you can unintentionally over-pressurize your chamber if it is not equipped with a fail-safe blow-off valve on the backfilling system. One of my favorite tricks is to vent with readily available *liquid nitrogen*. To do this, you drop one end of a vacuum hose into an open Dewar of liquid nitrogen. After the immediate boil-off purges air from the line, connect the other end to the chamber's vent valve and slowly open the valve. As liquid nitrogen is pulled up the hose it immediately vaporizes. You can't get a dryer gas than the boil-off from a liquid nitrogen source and, as opposed to using a pressurized bottled gas, there is no danger of over pressurizing your chamber from an open Dewar of liquid.

One final tip; if you must make a quick emergency repair inside your vacuum system, vent the chamber, then keep a slight positive pressure of dry nitrogen or some other inert gas flowing as a continuous purge as you open your system's load lock or windows. You have prevented humid air contact with clean chamber walls and can quickly recover your former vacuum with only a short low-temperature bake. If storing a vacuum vessel, it is best to store it under vacuum. Alternately, store it completely buttoned up, filled with a slight overpressure of dry nitrogen or other inert gas. Again, this keeps away build-up of the thick layer of water and atmospheric gasses that will form.

Good Practices for different vacuum levels

Rough vacuum levels

In rough vacuum environments, physical pinholes, cracks and gross contamination by dirt and grease are the main concerns. Clean the inside or vacuum side of all your parts to remove soldering fluxes, greases, oils, scales, rust, etc. Leak check parts by pressurizing them, swabbing joints with a soap solution and looking for bubbles.

Key Parameters for Rough (Roughing or Fore) Vacuum Conditions	
Pressure range:	Atmosphere (~10^2) to 10^0 torr
Major residual gasses:	All components of Air
Gas density:	~10^{19} to 10^{16} molecules/cm^2
Mean free path:	~10^{-5} to 10^{-2} (cm)
Time to form 1 monolayer:	~10 ns to 10µs
Particle flow conditions:	viscous flow
Pumps:	Mechanical, sorption, steam ejector or better pumps
Pressure gauges:	diaphragm gauge, spiral tube gauge, liquid U-tube manometer or better

Medium vacuum levels

Medium vacuum requires care in fabrication and cleaning beyond the rough vacuum requirements above. All vacuum parts should have a soap cleaning and clean running water rinse. Avoid any of the 'rough vacuum' materials in Table 3. Use clean gloves when handling or assembling components. Leak check as above.

Key Parameters for Medium Vacuum Conditions	
Pressure range:	~10^{-1} to 10^{-3} torr
Major residual gasses:	All components of Air, H_2O, CO_2, pump oils, hydrocarbons, H_2
Gas density:	~10^{16} to 10^{13} molecules/cm^2
Mean free path:	~10^{-2} to 10^{1} (cm)
Time to form 1 monolayer:	~10 µs to 10 ms
Particle flow conditions:	Knudsen flow
Pumps:	mechanical, ejector, diaphragm, scroll or better pumps
Pressure gauges:	thermocouple gauges or better

High vacuum levels (HV)

You now have to worry about good vacuum design and vent all blind holes in your vacuum parts. Parts should be leak-tight at least at a level of 10^{-7} torr-l/s leak rate or better. Shot, bead or grit blast all vacuum surfaces to remove surface contaminants. Scrub parts clean with detergents appropriate for the material. Alternately, vapor degrease all vacuum components. Standard industry practice for material selection and cleaning are suggested by the American Vacuum Society Recommended Practices Committee[x]. Wear gloves to avoid fingerprints on the parts (clean cotton gloves are O.K.). High vacuum requires that you work in a clean area, avoid dust and dirt contamination. Avoid materials higher on Table 3 than the high vacuum level.

Key Parameters for High Vacuum (HV) Conditions	
Pressure range:	~10^{-3} to 10^{-7} torr
Major residual gasses:	pump oils, hydrocarbons, H_2, contaminants, system outgassing
Gas density:	~10^{13} to 10^{9} molecules/cm^2
Mean free path:	~10^{1} to 10^{5} (cm)
Time to form 1 monolayer:	~10 ms to 10 s
Particle flow conditions:	molecular flow
Pumps:	oil or Hg diffusion pumps, rotary roots-type, molecular drag, turbo or better pumps
Pressure gauges:	thermocouple gauges, cold cathode gauges or better

Ultra-high vacuum levels (UHV)

For UHV, every part must be of excellent vacuum design; blind tapped holes, blind crevices, poor internal welds and large areas of stacked materials are subtle sources of gas problems. Leak testing shall indicate vacuum tightness at a sensitivity of 10^{-11} torr-l/sec. Parts must be mechanically or chemically polished to minimize surface area. All parts must be ultrasonically cleaned or vapor degreased, deionized water rinsed, alcohol rinsed to displace water and then dry nitrogen dried. Consult standard texts on vacuum technology such as O'Hanlon, Roth and Rosebury and the AVS Recommended Practices[x]. Work clean, in a clean area separated from high traffic areas, using vinyl or powder free latex gloves. Assemble components on lint-less cloths or clean commercial food grade aluminum foil and wrapped until installed in your chamber. Use only cleaned tools to avoid transfer contamination. Avoid any materials not designated UHV or XHV in Table 3. You will have to 'bake' the chamber and contents under vacuum to de-gas it.

Key Parameters for Ultra High Vacuum (UHV) Conditions	
Pressure range:	~10^{-7} to 10^{-11} torr
Major residual gasses:	H_2, H_e, H_2O, CO, CO_2, CH_4 (+ other cracked hydrocarbon contaminants)
Gas density:	~10^{9} to 10^{5} molecules/cm^2
Mean free path:	~10^{5} to 10^{9} (cm)
Time to form 1 monolayer:	~10 sec. to 30 hr.
Particle flow conditions:	molecular
Pumps:	turbo pumps, getters, ion pumps or better
Pressure gauges:	ionization gauges or better

Extreme-high vacuum levels (XHV)

For the XHV ranges, every part must be of outstanding vacuum design. Take measures in addition to those recommended for UHV levels. Chambers and components must be electropolished or chemically polished smooth. Since hydrogen permeation becomes an increasing problem below 10^{-11} torr (10^{-9} Pa), increasing use of barrier-oxide treated aluminum and refractory metal parts instead of stainless steels can help. All components should be UHV baked prior to insertion into XHV systems. Use special contamination free, residue free aluminum foil products[xii] and lint-less cloths for component assembly. Use cleaned tools specifically reserved for XHV assembly only, to avoid transfer contamination. Lint, dandruff and other bodily contaminants must be avoided with proper gloving, hair covering and body coverings and/or by working in HEPA filtered down-flow clean hoods and rooms. Double glove to avoid fingerprint or sweat contamination. You will have to 'hard bake' the chamber and contents under vacuum to de-gas everything. Avoid any materials not on the XHV list.

Key parameters for Extreme High Vacuum (XHV) Conditions	
Pressure range:	$<10^{-11}$ torr
Major residual gasses:	H_2, He, cracked hydrocarbon contaminants, system outgassing
Gas density:	$<10^5$ molecules/cm^2
Mean free path:	$>10^9$ (cm)
Time to form 1 monolayer:	>30 hr.
Particle flow conditions:	molecular
Pumps:	getter (Ti or NEG) pumps, ion pumps
Pressure gauges:	special ionization gauges, capacitance gauges

Space vacuum levels

To give you a sense of scale (and for a little fun) see Figure 13, pressures at different distances above the earth. The supersonic transport, Concorde, traveling at 11 to 18 km above the earth is at pressures too low to sustain human respiration but is only in the poorest of rough vacuums according to our scale definitions. At the level of the Space Shuttle orbit, ~240 miles (380 km) vacuum levels plummet to 10^{-9} torr (10^{-7} Pa), UHV levels. However, the Shuttle outgases so much that UHV surface science experiments would soon be contaminated if conducted outside of the cargo bay. At 10,000 miles (~15,000 kilometers) or one earth diameter above our planet, pressures drop to 10^{-13} torr (10^{-11} Pa). Finally, at 0.65 parsecs (~2 x 10^{13} kilometers) away or the distance half way between our sun and the nearest star, pressures are estimated to drop to 10^{-15} torr (10^{-13} Pa).

Key Parameters for Extreme High Vacuum Space Conditions	
Pressure range:	$<10^{-13}$ torr
Major residual gasses:	H_2, He
Gas density:	$<10^2$ molecules/cm^2
Mean free path:	$>10^{11}$ (cm)
Time to form 1 monolayer:	>100 days
Particle flow conditions:	molecular
Pumps:	ion, getter or infinite expanses of interstellar space
Pressure gauges:	Ion extractor gauges

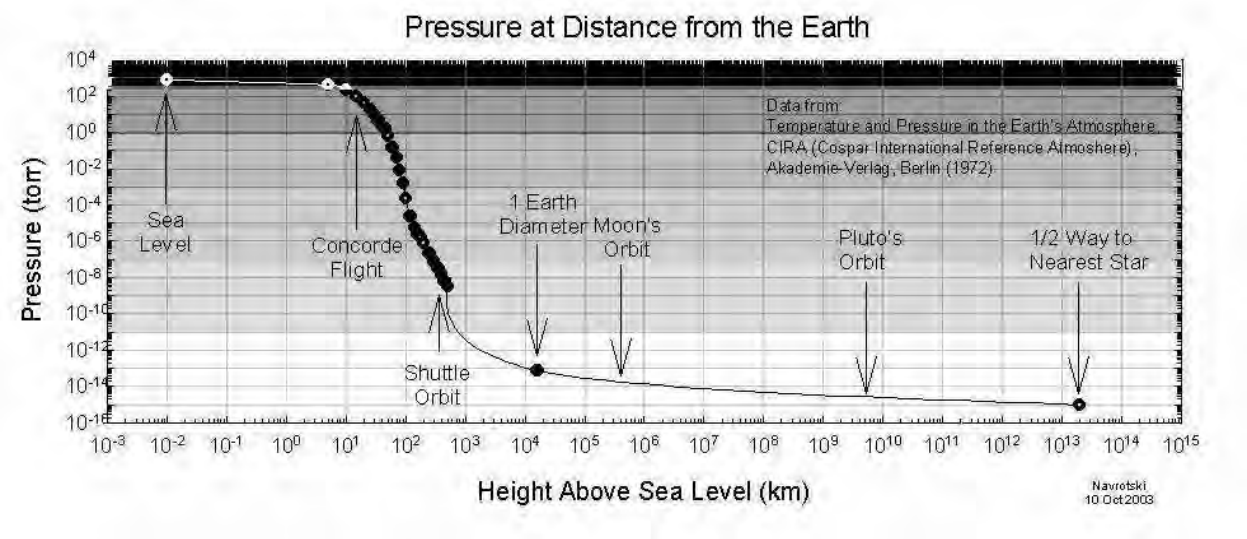
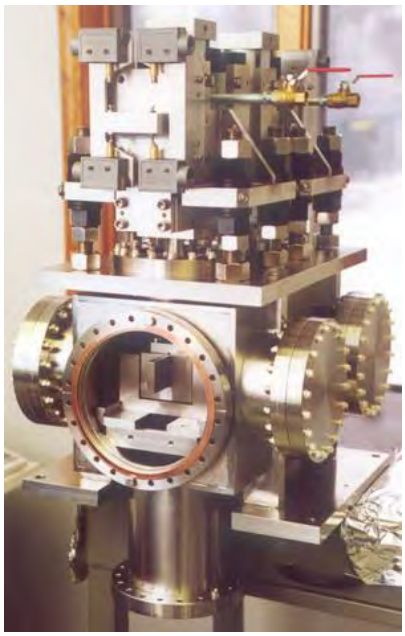


Figure 13 - Vacuum levels at distances above the earth's surface.

Summary

Hopefully, by reading this primer you have a basic understanding of what vacuum is, how you create it, how you measure it and most importantly, how you avoid contaminating it once you have it. As stated repeatedly, this is not an easy task. The higher the vacuum, the more time, effort, care and money it will take to produce and maintain your vacuum and vacuum equipment.

For further information, your next step should be to review the University of Alberta's coursework on vacuum basics at http://www.ee.ualberta.ca/~schmaus/vacf/basics.html. After that, borrow a copy of J.F. O'Hanlon, A User's Guide to Vacuum Technology and scan it cover to cover. With just a little more study, you will be well on your way to developing a comprehensive understanding of the wonderful world of vacuum. Finally, you can always contact the design and engineering staff at ADC, Inc. for advice on vacuum component construction.



Resources

Books

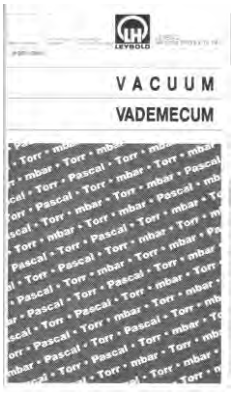


Figure 14 - Leybold's pocket reference.

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Web

Web Vacuum Basics for Amateurs: http://www.ee.ualberta.ca/~schmaus/vacf/basics.html
World-wide Vacuum Journals: http://www.sansalone.de/engl/lk_vacuum_journals.htm
American Vacuum Journals: http://www.avs.org/JVST/journals.html

References

[i] IEEE/ASTM SI 10-1997 "Standard for Use of the International System of Units (SI): The Modern Metric System." (Revision and re-designation of ASTM E380-93 and ANSI/ASTM Std 268-1992)

[ii] Software units conversion tool *Qvert*, http://www.bykeyword.com/pages/detail9/download-9810.html.

[iii] Software units conversion tool *Convert*, http://www.joshmadison.com/software

[iv] A good Web source for vacuum history: http://www.mcallister.com/vacuum.html

[v] Technical details for vacuum pumps: order a Leybold catalog from http://www.leybold.com . The technical information section is quite comprehensive although reference material is not on-line.

[vi] The American Vacuum Society: http://www.avs.org/

[vii] Worldwide vacuum association listings: http://www.sansalone.de/engl/LK_vacuum_associations.htm .

[viii] Vacuum System Calculations: http://www.lesker.com then follow leads to *Tech Info* then *Vacuum Technology*.

[ix] RCA Corporation vapor pressure charts. Original references: R.E. Honig, RCA Review 18 (1957), R.E. Hoing and H.O. Hook, RCA Review 21 (1960), R.E. Hoing RCA Review 23 (1962), R.E. Hoing and D.A. Kramer, RCA Review 30 (1969). Scanned copies available on-line at: http://www.mcallister.com/vacuum.html

[x] Y. Tito Sasaki, A survey of vacuum material cleaning procedures: A subcommittee report of the American Vacuum Society Recommended Practices Committee, *JVST A* 9 (3), 1991 pp.2025-2035.

[xi] Graduate students at Cornell have been known to conduct secret ceremonies and to sacrifice virgin copper gaskets to the vacuum god "Uhv" to insure a successful pump-down.

[xii] UHV grade aluminum foil: http://www.allfoils.com/uhvfoil.htm