# An Introduction to Vacuum Technology for the Amateur Scientist



# the Bell Jar

Vacuum Technique and Related Topics for the Amateur Investigator

# An Introduction to Vacuum Technology for the Amateur Scientist

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The content of this booklet is derived from articles which have appeared in *the* **Bell Jar** (ISSN 1071-4219), the quarterly journal of vacuum technique and related topics in physics for the amateur experimenter.

This booket was prepared for the Citizen Scientists League, and may be freely distributed, in its complete and unaltered form, throughout the community of amateur scientists for educational purposes. Commercial use is prohibited.

## the Bell Jar

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# A Note on Safety

**Notice of Disclaimer:** Many of the topics, projects, and materials that are associated with vacuum are inherently hazardous to life, health, and property. Please do not undertake the utilization or implementation of any of the information presented herein unless you have an appropriate level of experience. While care has been taken to assure the accuracy of the material presented, the author may not be held liable for any damages and/or injuries resulting from the use or misuse of information.

*Glassware*: Treat all glassware under vacuum with respect. **Safety glasses should be worn at all times to protect your eyes from flying glass should the glassware break and implode.** Large glass vessels should be screened with a suitable protective screen. A piece of polycarbonate plastic (e.g. Lexan) is satisfactory. Before each use, check all vacuum glassware for scratches, cracks, chips or other mechanical defects that could lead to failure. Consult a qualified technical glassblower concerning repairs of damaged glassware.

*High Voltage and X-rays*: High voltage experimenters are naturally drawn to vacuum because of the many interesting phenomena that may be studied in vacuum. High voltage safety is discussed in a number of amateur oriented publications such as the ARRL's "Radio Amateur's Handbook." An additional concern is the generation of x-rays by high voltage discharges in evacuated vessels. While not a great concern in rough vacuum conditions and voltages of 10 kV or so, higher voltages at milliTorr level pressures can produce harmful levels of x-rays under certain conditions. A simple radiation monitor should be available to check your experiments for radiation. When working with devices *intended* to produce radiation, a regular dosimetry program should be maintained by the experimenter in order to monitor dosage over time.

## Forward

My thanks are extende to Sheldon Greaves of the Citizen Scientists League for inviting me to contribute some material on vacuum technology to the group.

This document is intended to provide a brief overview of some of the concepts and hardware associated with vacuum, with an emphasis on the use of simple and reusable materials.

Access to medium and high vacuum is much more straightforward today than was the case 20 or more years ago. Some of the changes include:

- The wide use of standardized fittings ("KF" and "CF" fittings)
- An active surplus market (eBay, etc.) where high quality components may be purchased for (sometimes) very low prices.
- Refrigeration service vacuum pumps are excellent for many vacuum projects and the prices have come down substantially over the past decade.

In spite of the improvements in hardware availability, the availability of information that is relevant to the needs of the amateur has generally been inadequate or woefully out of date. The purpose of my journal *the* **Bell Jar**, established in 1992, was to fill that gap. The journal started as a print quarterly and was published for 10 years. During the late 1990s the material began a transition to the web and since 2000 a considerable amount of new material has been added.

All of the content from Volumes 1-5 have been consolidated into a compilation *The First Five Years*. A compilation for Volumes 6-10 is nearing completion. Both of these may be purchased in Acrobat format.

The information that follows has been drawn from *The First Five Years* as well as a variety of open articles that are available on my website. Some of the material is also new, prepared specifically for the Citizen Scientists League.

If you find that vacuum could be a useful adjunct to your science activities the next step beyond this publication would be to explore the pages at www.belljar.net. In addition to the content on the site there are many useful links. Specific questions may be addressed to the author.

Steve Hansen December 2011

The chart on the next page depicts how vacuum is produced (types of pumps), how it is measured (types of gauges) and how vacuum can be used in practical applications.



## 1. Basics of Vacuum

## Background

"Modern atomic physics is the child of the vacuum pump."

Karl K. Darrow, a researcher at Bell Laboratories, made this statement in his 1932 book "Electrical Phenomena in Gases." Indeed, the development of vacuum pumps capable of reaching very low pressures has been intertwined with most of the advances in physics since the mid-nineteenth century. The simple low pressure electrical discharge tubes developed by Geissler and others quickly progressed from curiosities to devices with significant implications. The discovery of x-rays by Roentgen in 1895 represented a watershed. The identification of the electron and the invention of the cathode ray oscilloscope tube happened at about the same time. Other developments quickly followed: the vacuum tube made the radio industry possible and vacuum coating processes led to new types of optical elements as well as to integrated circuits. The scanning electron microscope, mass spectrometer, laser, computer, microwave oven, compact disk and even plasma treated tire cords would all be fiction without vacuum and vacuum processes.

Unfortunately, even though it pervades our technology and our lives, vacuum is a field that has not been very accessible to the amateur and the non-specialist, mainly due to a severe lack of information specifically targeted toward that audience. Amateur vacuum experimentation did have a period of activity in the late 1950s and 1960s. For those who remember, two good examples were C.L. Stong's *Amateur Scientist* column in Scientific American and the amateur oriented pumps, kits and plans once offered by the firm of Morris & Lee of Buffalo, NY. Between the two, an impressive array of apparatus emerged from the efforts of ambitious basement experimenters. Reported were a variety of gas lasers, x-ray tubes, potential drop accelerators, mass spectrometers, simple & compound electron microscopes and high altitude chambers, to name a few. All of these were cobbled together with converted refrigeration compressors, single stage diffusion pumps, copper & glass tubing, sealing wax and a lot of ingenuity. The staying power of these endeavors is evidenced by the continued recycling of plans, often in the form of poor imitations, for a number of the vacuum related projects in Stong's columns.

In the intervening years there has been an almost complete lapse in the availability of up-to-date information on vacuum technique and apparatus specifically targeted toward the amateur, educator, or professional who likes to tinker. *the* Bell Jar was created at the start of 1992 to bring together those experimenters who have an established interest in vacuum as well as to promote vacuum technique as an interesting and challenging hobby.

Eight years later, the readership numbers in the hundreds and contributors range from true amateurs to professionals with established credentials in the field. This diversity has made for a lively publication and has resulted in favorable comments from the professional community. It is hoped that this compilation, containing material from the first five years of *the* Bell Jar, will help to inspire a new generation of amateurs to undertake experimentation in the fascinating field of vacuum technology.

## **Some Vacuum Fundamentals**

"One man's vacuum is another man's sewer."

-N. Milleron, 1970

Vacuum technology covers a very wide range of pressures and conditions. Vacuum to a person doing fiberglass laminating is very different from the vacuum used by a neon sign worker. A thousand times better than this is the level of vacuum used in electron devices such as x-ray and TV picture tubes. And, a thousand to a million times better than this is the degree of vacuum used in research on the surfaces of materials.

A vacuum system typically consists of one or more pumps which are connected to a chamber. The former produces the vacuum, the latter contains whatever apparatus requires the use of the vacuum. In between the two may be various combinations of tubing, fittings and valves. These are required for the system to operate but each introduces other





complications such as leaks, additional surface area that evolves water and other contaminants, and added resistance to the flow of gas from the chamber to the pumps. Additionally, one or more vacuum gauges are usually connected to the system to monitor pressure.

#### Measurement and Characteristics of Vacuum

Pressure, of course, is defined as the force per unit area exerted by the molecules in a system. At one standard atmosphere this pressure is 1.03 kg/sq. cm (about 14.7 pounds per sq. inch). Traditionally, the pressure in a vacuum system is stated in terms of the height of a column of mercury that may be supported by the pressure in the system (refer to Figure 1.1). This developed from the use of the barometer and related mercury column manometers as the primary way of measuring sub-atmospheric pressures. One standard atmosphere of pressure will support a mercury column 760 millimeters (29.92 inches) high. One millimeter of mercury (mm Hg) is the equivalent of 1 Torr, a unit named in honor of Evangelista Torricelli (1608-1647), the inventor of the barometer. A thousandth of 1 mm Hg is referred to as 1 micron Hg or, in more current terminology, 1 milliTorr (mTorr). In the

professional scientific literature, the *SI* (*Système International*) units are normally used. Here, pressure is stated properly in terms of newtons/sq. meter or Pascal (Pa). If you encounter a pressure in Pa, divide by 133.3 to get Torr.

Pressure in a vacuum system is measured with a gauge. The type of gauge that people are most familiar with is the common Bourdon dial gauge. The Bourdon mechanism is a curved or coiled tube that deforms when there is a pressure differential between what is inside the tube (the pressure being measured) and the air on the outside of the tube (usually the atmosphere). This *gage* pressure only tells you how far away the measured pressure is from the prevailing ambient.

In technical applications we are usually interested in the density of molecules within a chamber. This requires knowing the *absolute* pressure (that is, the pressure referenced to perfect vacuum) as opposed to the atmosphere-referenced indication that the Bourdon gauge provides. This is what the barometer and most other vacuum gauges provide.

Looking again at the barometer illustration, you will note that the realm of technical vacuum occupies a rather small portion of the column. Additionally, that final millimeter is divisible into several levels of vacuum. From 1 Torr down to a thousandth of that is termed the *medium vacuum* range. *High vacuum* begins there and goes nearly a million times lower (to about  $10^{-8}$  Torr). *Ultra high vacuum*, the place where physicists are studying the atomic structures of clean surfaces, goes downward from there. The best level of vacuum that has been obtained in the laboratory is in the range of  $10^{-13}$  Torr, about the pressure found in interstellar space.

The dividing lines between these regions of vacuum were not capriciously arrived at. For example, medium vacuum can be attained with mechanical displacement pumps while high vacuum requires other methods of pumping. The molecular density of gas at medium vacuum also provides a physical environment that is appropriate for a wide range of interesting phenomena and industrial processes. Neon signs and many deposition processes operate at medium vacuum. At high vacuum, molecule to molecule interactions are infrequent and the gas in a system becomes a good electrical and thermal insulator. Thermos bottles and TV picture tubes operate in this region. Ultra high vacuum (UHV) requires very clean, very tight system with bakable (to thoroughly rid the system of water) components. A defining factor of UHV is the time it takes for a molecular layer of gas to adhere to a clean surface. This is a mere instant in a 10<sup>-6</sup> Torr vacuum. At 10<sup>-10</sup> Torr, a layer of molecules takes hours to adhere to a surface. For atomic level surface studies and for many high purity semiconductor manufacturing processes, this fairly long *monolayer formation time* is required.

Obviously, even with a magnifying lens, the simple barometer won't be of much use for pressure measurements in these ranges. In our type of vacuum system a variety of gauges are used. For the most part these gauges work by what would seem to be very indirect methods. In the medium vacuum range it is common to judge pressure using the thermal conductivity of the air in the system. In the high vacuum region, gauges have been developed that measure the electrical properties of the gas when ionized. There are a number of gauges that directly measure pressure. The capacitance

diaphragm gauge is one of the more common. In this gauge a thin metal diaphragm acts as one plate in a capacitor. The other plate is adjacent to the diaphragm and is fixed. When the diaphragm flexes, ever so slightly, the change in capacitance will alter the frequency of an otherwise stable oscillator. This frequency shift is translated into pressure. Very carefully built, these gauges are accurate from many thousands of Torr down into the high vacuum realm. Capacitance gauges are generally used for controlling processes and other precision applications.

All of these gauges produce relative measurements. In order to be accurate they must be calibrated against a *primary* gauge where the pressure can be derived using fundamental units. A common gauge of this sort is the mercury filled manometer. A variety of liquid column and piston deadweight gauges have been developed for use as standards.

Besides the general calibration problem, all indirect vacuum gauges will produce different readings depending upon what gas is in the system. The common thermal conductivity gauges will produce widely differing readings when, for example, argon, helium or neon are present. Calibration curves for common gases are often provided with indirect gauges. If high accuracy is required, mechanical displacement gauges such as the capacitance diaphragm gauge are used. Because these measure true pressure, they are insensitive to the type of gas used.

## Means of Producing Vacuum

#### Mechanical Displacement Pumps

At the heart of the typical vacuum system is a mechanical pump. While most common air pumps use reciprocating pistons to create pressure or vacuum, this type of pump is not suitable for high quality vacuum of the sort needed in scientific applications. The reciprocating motion, leakage past the piston and the dead space that exists above the piston all conspire to limit the level of attainable vacuum. The types of pump that do work fall into the category of the rotary, oil sealed pump. As shown in Figure 1.2, this type of pump has a rotating off-center cylindrical rotor that "sweeps" air through the housing in which the rotor is located. Air is kept from passing between the vacuum and pressure sides by means of a set of two vanes that are arranged across the diameter of the rotor. The entire mechanism is lubricated and sealed by immersion in an oil bath.

The mechanical pump may either be used by itself in applications where only a moderate degree of vacuum is needed or in conjunction with other types of pump (for example the so called diffusion pumps) where higher degrees of vacuum are required. In this latter case, the mechanical pump is referred to as a *fore* or *backing* pump. Here the purpose of the mechanical pump is to bring the pressure in the system down to a level which will permit the operation of the high vacuum pump.

One rotary pumping stage will achieve a vacuum of about 1 Torr in normal use. In order to get better vacuum it is standard practice to place two pumping stages in series, coupled by a common shaft. In the case of most industrial duty pumps, the specifications will usually state an ultimate vacuum of 0.1 milliTorr. However, this level of vacuum is usually only attainable under ideal circumstances. A more realistic value is 5-10 milliTorr.

There is a large market for industrial grade vacuum pumps for such applications as semiconductor fabrication, applying protective and decorative coatings, and lightbulb manufacturing. As a result there is a also a thriving market in used and rebuilt pumps. New, the industrial grade pumps can cost well over \$1000. In the smaller sizes, rebuilt



Figure 1.2 - Mechanical Pumps. Rotary vane (left) & rotary piston (right).



pumped high vacuum stage (left).

pumps may be obtained for \$500 or so. Flea markets and metal scrap yards are also a good, if not reliable, source. I have obtained good pumps for less than \$35 through such sources. With belt driven pumps, the worst of the duds may be avoided by ensuring that the pump can be turned by hand and that such action results in a good "suck" when the hand is placed over the inlet.

For many school or hobbyist needs, there are some other low cost alternatives to these industrial pumps. These include the single stage rotary piston compressors that are used in some air-conditioners (good to about 1 Torr) and the 2-stage vacuum pumps used by service technicians for the recharging of refrigeration systems (good to about 20 milliTorr).

## High Vacuum Pumps

Mechanical pumps lose their efficiency at very low pressures. At a certain point, in the region of 1 mTorr, air doesn't respond very well to being squeezed and pushed around by pistons and rotors. Here the gas molecules don't really flow. They more or less wander into the pump. The most common type of pump for use in the high vacuum realm (and the one that is still best suited to general amateur applications) is the oil diffusion pump. This pump, invented by Irving Langmuir in 1916, utilizes a jet of vapor (generated by the boiling of hydrocarbon or synthetic oil) that forces, by momentum transfer, the incoming molecules toward the outlet side of the pump. These pumps only work at low pressures and the outlet of a diffusion pump must be coupled to a mechanical backing pump. Diffusion pumps are uncomplicated, quiet and only require simple (but sometimes tedious) maintenance. The major disadvantages are the migration (*backstreaming*) of oil toward the vacuum chamber (which may be minimized with baffles and/or refrigerated traps) and the catastrophic results from accidentally opening the system to atmospheric pressure: the oil breaks down and goes everywhere. Oil diffusion pumps generally operate at outlet pressures in the range of 100 mTorr or less. Ultimate pressures of 0.01 to 0.001 mTorr are readily achievable with small apparatus and simple baffles. Most of today's pumps have 3 stages with inlet sizes ranging from 2 inches on up.

Pumping speed is related to the inlet area of the pump. A typical 2 inch pump will have a speed of about 100 liters/sec. For most amateur and small scale laboratory applications, pumps with inlets of 2 to 4 inches are the most convenient and economical to use.

A variety of other styles of high vacuum pump have been developed. However, these are usually difficult to use in the home or school laboratory environment we are discussing here. They are also more expensive to maintain and servicing is usually beyond the capabilities of the typical amateur. Examples of such pumps include the turbomolecular (or turbo) pump, which is built roughly like a turbine, and the gas capture pumps (ion, cryoabsorption, and sublimation) that either entrap gas within a material or bury the gas under a constantly deposited film of metal. Most of these pumps are used in applications where extreme cleanliness is required or where very high vacuums need to be attained. Wide range turbo-drag pumps that have very modest roughing requirements are also well established.

Figure 1.3 shows the elements of a rough vacuum system along with how a diffusion pump would be inserted in order to make a simple high vacuum set-up.

#### Vacuum Terminology

The language of vacuum is extensive and what follows only covers the bare minimum. However, these are the terms and concepts that will be found to be the most valuable to the beginning vacuum experimenter.

*Mean Free Path.* Reduction in pressure results in a lower density of gas molecules. Given a certain average velocity for each constituent molecule of air at a given temperature (at room temperature this is about 1673 km/hr) an average molecule will travel a certain distance before it interacts with another at any given pressure. This average distance between collisions is the mean free path (l). At 1 Torr this distance is 0.005 cm, a value that scales directly with pressure. Thus the mean free path would be 5 cm at 1 mTorr and 50 meters at 0.001 mTorr. The lengthening of mean free path at low pressures is a key enabler for devices such as vacuum tubes and particle accelerators as well as for processes such as vacuum coating where microscopic particles such as electrons, ions or molecules must traverse considerable distances with minimal interference.

*Flow.* Gases at very low pressures behave very differently from gases at normal pressures. As a reduction in pressure occurs in a vacuum system, the gas in the system will pass through several flow regimes. At higher pressures these include *viscous* flow where the molecular mean free path is substantially shorter than the size of the system's lines and chambers. Viscous flow may be either *laminar* where the flow is regular with no eddies, or *turbulent* where the flow is irregular. Moving deeper into the vacuum environment, *molecular* flow occurs when the molecular mean free path exceeds the tubing diameter. Here the molecules behave statistically without regard to what their neighbors are doing. A third flow region, called *Knudsen* or *transition* flow, exists between the viscous and molecular regimes. This flow regime generally coincides with the medium vacuum range.

Which flow regime the gas is in depends upon several factors including tube diameter and pumping speed. As a rule of thumb, when the ratio of the average mean free path in a tube (lave) to the radius of the tube (r) is less than 0.01, the flow is viscous. When the ratio lave/r is greater than 1.00 the flow is molecular. One of the factors which determines pump applicability is the flow regime it needs to operate in. Mechanical pumps are not effective in the molecular region whereas diffusion pumps are. In viscous flow, fast pumping speeds will result in turbulence and lower speeds will produce laminar flow.

**Pumping Speed and Throughput.** The speed of a pump (S) is the volume of gas flow across the cross section of the tubing per unit time. The common units are liters/second. Since the density of a gas changes with pressure (i.e. the mass or number of molecules of gas in a given volume) an important measure is *mass flow* or *throughput* (Q) which is the product of pressure (P) and speed (S) with the units of Torr-liters/second. A useful exercise is to use Avogadro's law to determine how many molecules are flowing at a throughput of 1 Torr-liter per second. Then, for a given gas, compute how many grams per second are flowing at that throughput.

If you think of the vacuum system as an electrical circuit, throughput is like current flow and it is constant everywhere in the circuit. The various elements of the system (lines and pumps) are analogous to resistances except instead of voltage drops there are pressure differentials. In putting together a vacuum system you want minimal pressure differentials in the connecting lines and maximum throughput everywhere.

A simple example will pull this together. Consider a small diffusion pump that has a rated inlet speed of 100 liters/second at 0.0001 Torr (0.1 mTorr). Q would be 100 x 0.0001 or 0.01 Torr-liters/sec. Now, connected to the outlet of the diffusion pump we have a mechanical forepump which is capable of maintaining a pressure of 0.1 Torr. Given the fact that Q at the diffusion pump inlet must equal Q at the outlet and that there is a pressure of 0.1 Torr at that outlet, the minimum speed of the forepump must be 0.1 liters/sec, a speed easily met by even very small mechanical pumps. On the other hand, if the diffusion pump inlet pressure is 0.01 Torr (10 mTorr) - say just after the pump is started or if it is working against a very gassy load - the forepump would have to have a speed of 10 liters/sec to allow the diffusion pump to work at full speed. This would be a large pump.

To summarize all of this, at high diffusion pump inlet pressures, the speed most likely will be constrained by the speed of the forepump. At low inlet pressures there is so little mass flow that a very small forepump can keep pace with even a large high vacuum pump. In fact, in a tight system you can shut off the forepump once a low enough pressure has been reached simply because so little mass remains in the system.

**Conductance of Tubing.** As mentioned above, the tubing in a vacuum system can represent a significant resistance. When one end of a tube is connected to a pump, that end of the tube will have a higher pumping speed than will the other end. For viscous flow, as would be the nominal case for roughing lines (i.e. mechanically pumped), the conductance, C, is dependent upon gas pressure and viscosity and, at room temperature, is (for a tube diameter of D cm, length of l cm and at an average pressure of P Torr):

$$C = 180 \frac{D^4}{l} P_{ave}$$
 liters/sec

An example would be a foreline of 2 cm diameter and 60 cm long. At one end is a small mechanical pump; the other end is connected to the outlet of a diffusion pump. Referring to the manufacturer's literature for the pump we find that the pumping speed of the roughing pump is 0.5 liter/sec at 100 mTorr, the maximum recommended foreline (outlet) pressure of the diffusion pump. Plugging in the numbers, we find that the line conductance is 4.8 liters/sec. Thus, the line is not limiting the capabilities of the forepump.

Because of the statistical nature of molecular flow and the very low absolute pressure gradients, pressure is not a factor in this flow regime where, for example, a diffusion pump would operate. Here we have:

$$C = 12 \frac{D^3}{l}$$
 liters/sec

An example here would be a 2 inch (5 cm) diffusion pump which has a specified inlet pumping speed of 100 liters/sec. The pump is connected to a small experiment chamber through 60 cm of 2.5 cm diameter tubing. Inserting the numbers, we find a line conductance of only 3.1 liters/sec. This may be adequate for the small chamber but it certainly throttles the pump significantly. If a 5 cm line were substituted (same length) the conductance would rise to 25 liters/sec.

In either case, the most important thing to bear in mind is that conductance is strongly influenced by the tube diameter. 1 cm to the third or fourth power is a whole lot less than 3 cm to the same powers. The bottom line is: go for fat tubes, and keep them short, particularly in high vacuum lines.

*Outgassing and Vapor Pressure*. Assuming that a system is tight, as the pressure gets lower most of the load is from gases evolving from the surfaces of the materials in the system. This becomes significant below pressures of around 100 mTorr. Outgassing will be the main limiting factor with regard to the *ultimate pressure* which any particular system may reach, assuming that leaks are absent. Leaks may be either *real* leaks, like holes in the chamber, or *virtual* leaks that are caused by gas escaping from, for example, screw threads within the system or porous surfaces that contain volatile materials. The level of outgassing is reduced by keeping the system clean and dry and with a proper selection of materials. If the construction of a system is appropriate to the practice, adsorbed layers of water vapor and other gases may be evolved by heating the system in an oven or with a hot air gun to a temperature of at least  $150^{\circ}$  C and usually more. For most applications down to about  $10^{-5}$  Torr this level of cleaning is not required. However, the system components should be kept clean (no fingerprints or other grime), dry and, as much as possible, sealed off from room air.

Related to outgassing are the *vapor pressures* of the materials used in the system. All materials evolve vapors of their constituent parts and these vapors will add to the gas load in a system. Water is the worst commonly encountered material and is a good example of what vapor pressure means. At 100° C, the vapor pressure of water is 1 atmosphere (760 Torr). Under those circumstances, when the vapor pressure is equal to the surrounding pressure, we know what happens - the water boils. At room temperature, the vapor pressure of water drops to 17.5 Torr and it will boil at that pressure. Water is not a good material to have in high vacuum systems. Other materials having high vapor pressures include some plastics, particularly those with volatile plasticizers, and metals such as mercury, lead, zinc and cadmium. Low vapor pressure materials include glass, copper, aluminum, stainless steel, silver, some other plastics and some synthetic rubbers. As vapor pressure is a function of temperature, some higher vapor pressure materials, e.g. zinc bearing brass, are quite acceptable in many applications as long as excessive temperatures are not encountered.

**Backstreaming.** It is always hoped that the flow of gas and vapor in a vacuum system is away from the chamber, through the pump and out to the atmosphere. However, this is not the case at low pressures when the system is in the molecular flow regime. Here we are dealing with very small absolute pressure gradients and the gas is so rarefied that the molecules move independently of one other. Fluid-like flow simply doesn't exist. In this regime pump oil molecules,

a common contaminant in vacuum systems, will have a great capacity for wandering against the general flow. This is one reason why diffusion pumps always have some sort of baffle or trap between the pump and chamber. Otherwise fairly large quantities of oil vapor will backstream out of the pump and into the chamber, contaminating what ever it is that you want to keep clean.

## Materials

Materials choices become increasingly restrictive as one progresses from rough vacuum (down to around 10 to 100 mTorr) to high vacuum (better than 1 mTorr down to around 0.001 mTorr) and then to the ultra high vacuum (UHV) range. The following paragraphs list some common materials and their regions of applicability.

*Rubber.* Natural gum rubber is the traditional choice for rough lines. Use heavy wall tubing designed for vacuum use and never expose rubber to elevated temperatures. A short length of rubber tubing with a pinch clamp (a parallel jaw woodworker's clamp for larger lines) makes a handy valve. Today most folks avoid rubber in favor of synthetics.

*Plastics.* While most plastics are limited by a combination of high water uptake, poor high temperature performance and gassy plasticizers, almost anything will work down to about 1 mTorr. Wire reinforced PVC is now commonly used in place of rubber for rough vacuum applications. I prefer it over rubber. Polystyrene is preferable over acrylics and polycarbonate plastic may be used in applications to about 0.1 mTorr. Polyethylenes are very useful materials in high vacuum as they are clean and have almost no tendency to ingest water. However, temperature resistance is poor and you can pretty much forget about trying to glue pieces together or to anything else. Teflon has excellent mechanical and vacuum properties. Nylon and Delrin soak up water but they are usable in high vacuum provided that exposure to the atmosphere is minimized.

*Synthetic Elastomers.* These are commonly used to fabricate O-rings as used in flanges and couplings. Common names include Buna-N and Viton. Components using these materials may be used in the high vacuum range Buna-N will withstand temperatures to about  $100^{\circ}$  C for short periods. Viton may be baked and is UHV compatible. Silicone elastomers are usable to a couple hundred degrees but tend to be permeable to gases.

*Epoxies.* Solvent free epoxies are of great use where any combination of glass and metal are to be stuck together (feedthroughs, accelerator columns, etc.). Several high vacuum formulations are available from vacuum equipment suppliers. For general vacuum applications good old hardware store white or clear epoxy works pretty well.

*Greases and Waxes.* A great variety of this stuff is around. The better materials (like the Apiezon products) can be used into the UHV region. One wonderful product to keep around is Apiezon W wax. It is applied at 100° C, can be used at 80° C, is fully compatible with high vacuum, and can stick together anything made of glass or metal. Unlike epoxies, you may rework or modify by reheating. I try to avoid greases. They have a wonderful tendency to get on everything. Properly installed O-rings don't need grease in most circumstances.

*Glass.* The traditional material for high vacuum. Transparent, clean, insulating, cheap if you know how to work it, etc. For people whose glassworking skills only extend to watching it sag (me), a good variety of prefabricated shapes are available from suppliers of scientific glassware.

*Metals.* Stainless steel is the standard for modern commercial high vacuum apparatus. I've had good success with standard copper water tubing and wrought copper fittings at rough to high vacuum.

**Solders.** My favorite joining material is 2 to 4% silver - tin soft solder. This is often called "hobby" solder and it is readily available at hardware stores in little packages with a tube of water soluble flux. It flows nicely, is compatible with copper, brass and stainless steel and it is strong enough for most work. Silver solders (actually brazing rod) are good where high strength and is needed but you will need a gas-oxygen torch to work anything except the smallest pieces. When using hard solders, select ones that are free of toxic or high vapor materials such as cadmium.

## Leaks: The Good, the Bad and the Ugly What leaks are, how to classify them and how to make useful leaks

### **INTRODUCTION**

When a vacuum chamber is evacuated with a pumping system, the rate of pressure decline will slow and eventually, for all practical purposes, cease. The minimum pressure that the system reaches is called the base pressure.

The job of the vacuum pump is to remove gas molecules from the system. In theory, a high vacuum pump should be able to remove each and every molecule that wanders into its inlet. In practice, the system itself is continuously contributing a seemingly infinite number of molecules and the pump has to contend with this load.

What this means is that a 1 liter chamber has more gas than the 1 liter's worth. There is gas that has adhered (adsorbed) to the walls of the chamber. There is gas that is within some of the materials that make up the system and that will, at reduced pressures, evolve into the chamber. Some materials will turn to gas (vaporize) at low pressures. Finally, some gas will enter the system through holes, cracks and other gaps in the system's walls.

In some cases gases are introduced intentionally into a vacuum system. This is the case with sputtering systems, ion sources and chemical vapor deposition systems, to name but a few examples.

Each of these gas sources is a leak. The bad ones are termed real leaks. In these the offending gas is transmitted into the system through an actual channel from the outside world. The ugly ones are a result of gas sources within the system. These may result from poor materials choices or contamination of the vacuum surfaces by, for example, finger prints. These internal sources of gas are termed virtual leaks. The intentional leaks, the good ones, are the ones that are used to introduce process gases into the system.

Good, bad or ugly, the common denominator is that leaks represent the ingress of gas molecules, at some given rate, into the vacuum system.

In this article we'll discuss how to differentiate between real and virtual leaks, how leaks are sized, and how to make and size intentional, predictable leaks.

### **IDENTIFYING LEAKS**

The performance of a vacuum system is dependent upon a whole host of parameters: the type and size of the pump, the size of the chamber, the length and directness of the lines between the pump and the chamber, and so forth.

If you have a system that you run on a regular basis, you will get an understanding of how quickly it pumps down and what the base pressure is. If you make a change to the system (perhaps it's just a matter of having opened the system or



Figure 1.4 - Normal & Abnormal Pumpdowns

perhaps you put some new fixtures in or appendages on the system) you might notice that it doesn't behave as well as it did. What you might observe is a slower pumpdown and a poor base pressure. The differences are illustrated in Figure 1.4.

A lesson to be learned is that it is useful and frequently important to keep notes of your system's performance. If you don't have a baseline, you won't know when something is amiss.

At this point we know something is wrong with the system. However, the pumpdown profile won't reveal what the problem is. It could be contaminated pump oil or it could be a partially closed valve. Or, it could be a leak.

Here is where an additional system feature is valuable. This feature is an isolation valve that is located at or near the pump inlet. If the system is pumped to its base pressure and the isolation valve is closed, the pressure in the system above the valve will rise at some rate. (Obviously this



Figure 1.5 - Differentiating Leaks

requires that the gauge be located above the valve where it should be anyway.) The rate and form of this pressure rise profile is indicative of the size and type of leak.

This is another place where a baseline is important: every system will exhibit some pressure rise. The baseline will let you know when it is abnormally high. If the pressure rate-of-rise is not abnormal, then you will have to look elsewhere for the problem. This could be a pump problem or a leak below the isolation valve.

If the pressure is rising abnormally, use your watch and record the pressure at a succession of equal time intervals. Try to let the pressure rise a couple of orders of magnitude and then plot the results on a linear scale.

Referring to Figure 1.5, a real leak will yield a linear pressure rate-of rise curve. The slope of the curve is a function of the leak rate and the volume of the system: the larger the leak, the steeper the slope; the larger the volume, the shallower the slope. We will quantify this later in the article.

The leak can be through an actual hole or channel, say a crack in a weld, a badly seated gasket or some other aperture. Or the leak can be a result of the gas from the outside room permeating through some component of the system. Elastomers, as are used in O-ring seals, are permeable to gases. There's no actual hole but gas molecules can work their way through the bulk of the material. Different elastomers have varying permeabilities: silicone is really bad with regard to this while Viton is relatively non-permeable. Permeation is selective as well: helium will work its way through a gasket much more easily than nitrogen.

Minimizing permeation leaks is a matter of selecting the correct elastomers (or other organics) and then minimizing the exposed areas of those permeable materials.

Permeation is not restricted to organics. At very low pressures, getting into the ultrahigh vacuum realm, permeation through glass and metals can start to become a problem.

Getting back to the rate-of-rise test, if the slope gets shallower as time goes on, you probably have a virtual leak. For example, gas trapped in a threaded hole under a bolt will leak out slowly causing the pressure to rise. Eventually the gas will all leak out (since this is a finite source) and the leak will appear to go away. This sort of situation can be verified by repumping the system and then looking again at the pressure rate-of-rise. Since the gas has already been dissipated, the virtual leak will no longer be present. It is for this reason that you can buy special bolts and screws for vacuum use that have holes drilled through them.

Material	10-6	10-2	1	10
Water	-110	-60	-15	15
Mercury	-40	45	120	190
Octoil	35	125	200	235
DC-704	50	145	210	250
Cadmium	125	270	380	
Zinc	175	350		
Lead	425	710		
Silver	680	1050		
Tin	820	1200		
Copper	850	1250		
Carbon	1900	2400		
Tungsten	2400	3250		

Table 1.1 - Vaporization Temperatures for Selected Materials at Various Pressures. Numbers across the top are pressures in Torr. Values in the columns below the pressures are the vaporization temperatures in °C. These values have been derived from vapor pressure curves from several sources and are rounded off.

Moisture and surface contamination (finger prints, etc.) will have a similar effect. Eventually these will cease to be gas sources if the system is pumped for a long enough period of time. (Depending upon the level of vacuum required, it may be a *very* long time.)

You have to be cautious of the vapor pressures of the various materials that make up the system components as inappropriate choices can result in virtual leaks. Organics within the chamber will be sources of virtual leaks. If you are trying to get vacuum levels below  $10^{-6}$  Torr, you have to be concerned about metals and metal alloys that have high vapor pressure constituents.

Table 1.1 on the previous page lists a number of representative materials that might be found in vacuum systems along with their vaporization temperatures at several selected pressures. It can be seen that water is a problem at almost any pressure: at  $10^{-2}$  Torr, water will vaporize at any temperature over -60 °C.

Brass, an alloy of copper and zinc, is usually an acceptable vacuum material for medium vacuum work. However, zinc has a fairly high vapor pressure. Thus, if a brass component is used at  $10^{-6}$  Torr in an application where the brass will be heated to a temperature of over 165 °C, the zinc will freely vaporize. A similar situation will be seen with cadmium plated hardware.

Copper is a pretty safe material given its low vapor pressure. Aluminum, stainless steel and other "high vacuum" materials similarly have very low vapor pressures.

If your virtual leak source is due to a basic materials incompatibility, then no amount of pumping is going to rid the system of that problem.

To conclude this section, what you'll probably find in your rate-of-rise test is a mixture of low-level real and virtual leaks that are harmless to your application (and which would probably take forever to fix anyway), and occasional nasty leaks that have to be fixed. These nasty things will also be combinations of real and virtual leaks. Now we'll look at how to size a leak.

## SIZING LEAKS

As stated before, leaks represent molecules entering the system. Therefore, the proper way to specify a leak is in language that relates to how many molecules per unit time are being admitted. However, talking about x molecules per second is a bit inconvenient as no other common vacuum measurement uses molecules as a unit. To get around this we have to go to the gas laws and Avogadro's number. The relationship that everyone learns in high school chemistry goes as follows:

A container of 22.4 liters volume at 0 °C and one standard atmosphere pressure (760 Torr) will contain Avogadro's number of molecules, i.e. about  $6.02 \times 10^{23}$  molecules. This number of molecules is termed a mole and will have a mass in grams equivalent to the atomic mass of the particular gas as measured in atomic mass units (AMU).

For example, a 22.4 liter vessel of nitrogen (atomic mass 28) at standard conditions (0 °C and 760 Torr) will contain Avogadro's number of molecules and the gas will have a mass of 28 grams. The same container of helium will contain 4 grams of that gas.

If the temperature and pressure in the vessel are not standard, then the ideal gas law is invoked to adjust for the deviation from standard conditions. If the gas is at a higher pressure (but at standard temperature), there will be a proportionally larger number of molecules. If the gas is at a higher temperature (but at standard pressure), then there will be fewer molecules. (One caveat: make sure that you make temperature adjustments with Kelvin (absolute) units.)

The bottom line here is that if volume, temperature and pressure are specified, it is then possible to determine how many molecules are in the volume. This gets us to terms that we can use in vacuum practice as we always talk in terms of volumes, pressures and temperatures.

For vacuum purposes, the above relationship is normalized to more convenient volume units, i.e. liters or cubic centimeters. What we end with are *standard condition* volumes.

Using liters, the standard condition volume term is the Torr-liter. This represents the molecules contained in a one liter volume at a pressure of 1 Torr. Not stated but understood is a standard temperature of 0 °C. Adjusting from the relationship of 22.4 liters of gas at 760 Torr and 0 °C equals  $6.02 \times 10^{23}$  molecules, one Torr-liter would then contain about  $3.5 \times 10^{19}$  molecules.

Using cubic centimeters, the standard condition volume term is the std cc (scc). This represents the number of molecules contained in a 1 cc volume at a pressure of 760 Torr. A standard temperature of 1 °C is also understood. Going back to the pressure/temperature/volume relationship for a mole of gas, a std. cc contains about  $2.7 \times 10^{19}$  molecules.

In vacuum practice there is a term called *throughput* which is usually abbreviated as Q. This was briefly discussed in the very first issue of *t*BJ. Throughput is simply the number of standard condition volume units that flow in a given

period of time. Thus we have the usual terms of Torr-liters/second (T-l/sec) and standard cubic centimeters per second (Std. cc/sec) or minute (sccm).

The convention for which set of units you pick is usually based on the application. Q within the vacuum system is usually specified in terms of T-*l*/sec since pressures are usually measured in Torr and volume in liters. Leaks are usually quantified in terms of Std. cc/sec. Q from intentional leaks (i.e. flow control devices) is usually specified as Std. cc/minute (sccm) or, for high flows, Std. liters/minute (slm). Some handy conversions are:

1 Torr-liter = 1.32 Std. cubic centimeter (scc)

1 Torr-liter/sec = 79 Std. cubic centimeters/min (sccm)

Another term you will run into for leak rates is Atmosphere cc/sec (or Atm. cc/sec). This is about equivalent to sccm except that the pressure reference is the prevailing ambient pressure (usually near 760 Torr unless you live in Denver or Albuquerque).

Since all of these terms relate to molecular quantity, thence to the actual mass of the gas, standard condition volumes are also called *mass quantities* and standard condition volumetric flow is also called *mass flow*.

#### **RELATIONSHIP BETWEEN Q, PRESSURE and SPEED**

Again going back to Volume 1, Number 1, there is a simple way of tying some basic vacuum units together in a useful way. In the term T-l/sec we have pressure (Torr) times speed (liters/sec). Thus, mass flow is equal to speed (e.g. pumping speed or line conductance) times pressure:

$$Q = P \times S$$

Please refer back to that issue for a few examples of how this is applied within vacuum systems. Suffice it to say that this is like Ohm's law:

$$I = \frac{E}{R}$$

where I (current), which represents electrons per unit time, is analogous to Q (molecules per unit time), E (voltage) is analogous to pressure, and 1/R (reciprocal resistance or conductance) is analogous to speed.

#### DETERMINING THE SIZE OF A LEAK

With that bit of grounding behind us, it is fairly straightforward to quantify the size of a leak in units such as sccm by using the pressure rate-of-rise measurement discussed above. As noted:

- A pressure rise over time indicates a leak.
- A linear rate-of-rise indicates a constant leak rate, typical of real leaks.
- The slope of the curve is a function of both the size of the leak and the volume of the system.

Knowing that the Q of the leak is related to pressure, volume and time, we can determine the size of the leak from the rate-of-rise curve (pressure change with time) assuming we know the volume of the system. Of course, since we are measuring the mass flow, Q, as standard condition volumetric flow, we also have to adjust for these standard conditions. The relationship that does this is:

$$Q(sccm) = 79 \left[ \frac{273}{273 + T} \right] \left[ V \frac{\Delta P}{t} \right]$$

The guts of this equation is in the right hand set of brackets where  $\Delta P$  is the change in pressure in Torr, t is the time, in seconds, over which the change in pressure occurred, and V is the volume of the chamber in liters. The units here are

Torr  $\times$  liters  $\div$  seconds. The left hand brackets give us the temperature adjustment factor where 273 is 0 °C in Kelvin units and *T* is the temperature of the gas in the chamber, also in K. Finally, the 79 is the conversion from T-*l*/sec to sccm.

The biggest uncertainty here is the volume of the system. With a rule and a bit of time, a reasonable volume determination can be made.

## **Orifices and Orifice Leaks**

Orifices represent a very simple and inexpensive way to introduce gases to a vacuum system. The gas can be used to assist a process and/or it can be used to control the pressure in the chamber.

## THE SPEED OF SOUND

The accepted value for the speed of sound ( $C_s$ ) in air under normal conditions is about 320 meters/second. Many years ago it was noted that the speed of sound was greater in the summer than in the winter. Researchers empirically determined that this temperature dependence amounted to an upward change of about 60 cm per second for every degree C increase in temperature. Expressed more precisely, with every property given its proper due, we have

$$C_s = \left[\frac{\gamma P}{\rho}\right]^{\frac{1}{2}}$$

where  $\gamma$  is the ratio of the molar specific heat of the gas at constant pressure to the molar specific heat of the gas at constant volume ( $c_p/c_v$ ),  $\rho$  is the gas density and *P* is the gas pressure.

#### NOZZLES AND SONIC FLOW

Consider the configuration of Figure 1.6. Here there is a piece of tubing with an orifice mounted in the tube.  $P_1$  and  $P_2$  refer to the pressures to the left and right of the orifice respectively.

Obviously, if  $P_1 = P_2$  there will be no flow of gas through the tube. If  $P_1$  is increased slowly (with  $P_2$  remaining constant) gas will flow through the orifice. The amount of gas flow (the mass flow) will now be a complex function of  $\gamma$ , orifice geometry, temperature,  $P_1$  and  $P_2$ .

The orifice, since it has a cross-sectional area less than that of the tubing, serves to increase the velocity of the gas that is flowing through the tube. Bernoulli found that as velocity increases, pressure falls:

$$\frac{P}{\rho} + \frac{V^2}{2} = \text{Constant}$$

This velocity/pressure relationship can be shown with the familiar experiment where one blows air between two parallel sheets of paper and the sheets move toward each other.

Bernoulli's law is only valid for constant density flow and for gases. If the flow is not too great the gas is, for all practical purposes, incompressible.

Going back to the orifice, if the pressure  $P_1$  continues to increase relative to  $P_2$ , a point will be found where the velocity of the gas through the orifice becomes sonic. That is, the gas velocity reaches the speed of sound. This condition occurs when  $P_1$  is about twice  $P_2$ .

It turns out that the gas velocity cannot exceed the speed of sound. So, further increases in  $P_1$  only increase the density of the gas going through the orifice. Since the density is directly proportional to the pressure, the mass flow becomes directly proportional to  $P_1$ :

P1 P2	<i>P</i> 1	<b>P</b> 2

$$Q$$
 (mass flow) = k $P_1$  when  $P_1 \ge 2P_2$ 

Because the density is now increasing, the gas has become compressible.

Figure 1.6 - Orifice

This sonic flow condition, also called choked flow, can be used as the basis for some types of mass flow meters and controllers. The value of k can be calculated or it can be experimentally derived.

A simple controlled leak may be fabricated from a laser drilled orifice of the appropriate size for the application and pressure range and a source of regulated pressure gas. Lenox Laser has a wide variety of flow orifices at reasonable prices. They also have a nice on line calculator for computing standard condition flow rates. Check out their web site at http://www.lenoxlaser.com/.

## 2. Hardware

## **Refrigeration Service Vacuum Pumps**

# Vacuum pumps that are sold for the servicing of refrigeration systems offer good performance at reasonable cost.

## **INTRODUCTION**

While commercially available mechanical pumps provide an optimal solution in terms of speed, reliability, and ultimate performance, they are also relatively expensive even in used or rebuilt condition. For the amateur with modest requirements, or for someone who is just beginning to experiment with vacuum apparatus, there is a good alternative: the type of vacuum pump that is used in the refrigeration service trade for recharging refrigeration systems. (They should not be confused with the vacuum pumps that are incorporated within refrigeration systems.) With some slight modifications, they are well suited to the purposes of the vacuum experimenter and educator. Such pumps may be obtained at relatively low cost, have good vacuum capabilities, are fairly rugged, offer many features of industrial vacuum pumps and can reliably achieve pressures to 20 mTorr. They are also suitable for backing small diffusion pumps.

Two pumps of domestic manufacture have been evaluated over the years. One is a two-stage 4 cfm pump manufactured by Robinair. The other is a two-stage 3 cfm pump manufactured by J/B Industries. These pumps represent two of the more popular models and they are commonly available at local distributors who cater to the HVAC and appliance repair trade. Prices generally run in the \$350 range but substantially lower prices may occasionally be had when the dealer has made a volume purchase agreement with the manufacturer.

More recently, a number of pumps of Chinese origin have appeared on the market. These are generally under \$200 and the author's experience with them has been very favorable.

Common features for these pumps include direct drive and gas ballast valves. Shut-off valves are provided with the USA pumps while the Chinese pumps forgo this nicety. Oil drains are conveniently located and the exhausts are either directed through the lifting handle or through a muffler on the pump's housing.

As supplied, these pumps are designed to be used with small diameter refrigeration charging hoses and the inlet fittings are dual flare coupings. Such hoses (typically with an inside diameter of about 3/16") have a very low conductance and the only real modification needed is to make an adapter that can couple the pump to a hose of more reasonable diameter.

The pumps usually come with 1/2" or 1/4" flare fittings. Hose adapters and reinforced vacuum tubing may be found at <u>http://www.belljar.net/chambers.htm.</u> The 1/2" adapter mates with 1/2" id hose and the 1/4" adapter mates with 3/8" id hose.

#### **COPING WITH WATER VAPOR**

Contamination of the oil in any pump by water (or any other high vapor pressure liquid) will undo any attempt to achieve a good vacuum. The pump oil should be changed on a regular basis or after performing any experiments involving water or volatile solvents.

Many pumps (these included) have a provision called a gas ballast which is very useful when pumping condensable vapors. The gas ballast is a valved arrangement by which atmospheric air may be admitted to the compressed gas in the exhaust stage of the pump just before the exhaust cycle. Diluting the moisture-laden air in this part of the pump prevents the vapors from condensing. Lowest pressures may not be attained while the gas ballast valve is open. The usual procedure is to start the pumping cycle with the gas ballast operating, then slowly

close the valve as the vapors are removed and the pressure plateaus. The pump should then continue pumping to a lower pressure.

## **HVAC Vacuum Pump Manifold and Chambers**

## Vacuum pumps that are used in the HVAC trade offer an economical path for medium vacuum experiments. This article details a simple manifold and chamber options for a simple vacuum system that is suitable for the hobbyist and educator.

## **INTRODUCTION**

This article describes a simple and relatively inexpensive bell jar type system that is suitable for hobbyists as well as middle and high school classroom use. It is based on a two-stage refrigeration service vacuum pump and a readily available plastic belljar/baseplate assembly. With a modest complement of hardware store plus some specialty items, the pump and bell jar can be integrated into a flexible system which also includes a rudimentary provision for pressure control. I have built a number of these systems for the American Vacuum Society's education outreach program and the results have been good. An example of one of these is shown in the photograph below. The pump is a JB Industries DV-85N rated at 3 cfm.



Figure 2.1 - Pump with Chamber

## PUMP AND MANIFOLD

I have used any number of the pumps that are offered to the refrigeration service trade. In years past two major manufacturers were JB Industries and Robinair. These manufacturers still have a strong presence but there is now a plethora of Chinese import pumps. The fit and finish of the Chinese pumps is not quite as good as the USA pumps but they seem to work very well. That said. I'd recommend the USA built pumps, especially Robinair's 2 stage 6 cfm pump model 15600. The remainder of this article will use the Robinair as the basis for the platform. (My pump is an older model in red, the newer Robinair "CoolTech" pumps are blue.) The pump has a rated base pressure of 20 milliTorr and with a tight and clean system this is readily achieved.

The pump comes with a tee inlet fitting equipped with 1/2" and 1/4 inch male SAE flare fittings (MFL). The 1/2 inch fitting is vertical and provides a good conductance path to a vertically oriented manifold. The first task is to adapt the flare to 1/4-inch pipe thread and this is done using a 1/2 female flare adapter to 1/4" male NPT. The flare connection can be sealed either with a copper flare gasket or with an o-ring that is inserted into the female flare fitting.

The manifold, as shown below, has three primary components that are connected with a 1/4" brass cross: a KF16 flange for the attachment of a gauge, a 1/8" needle valve with 1/4" hose barb for gas inlet control and a KF40 flange for the chamber connection. The 1/4 MFL fitting on the pump's inlet manifold is used with the provided o-ring sealed cap as the vent valve. All threads are sealed with "5 minute" epoxy. A picture of the completed manifold is shown in Figure 2.2 below.



Figure 2.2 - Manifold on Pump

## ADAPTING A PLASTIC BELL JAR

Nalge Company manufactures a low cost vacuum bell jar and base plate under their Nalgene brand. These are available from a variety of science supply houses for about \$100. Edmund Scientific Co. has this item as catalog number 3071338. A drawback of this simple chamber is the little (low conductance) side port that is used for evacuation. I have developed a modification of the baseplate as shown in Figure 2.3.

Start by drilling a 9/16" hole in the exact center of the baseplate (this is easy from the underside). I use an auger bit in a hand brace - the plastic is soft and cuts easily. Tap the hole (3/8 NPT) from the bottom and only run the tap deep enough such that the nipple is just a bit loose when the end of the nipple is flush with the top of the plate. Coat the threads of the nipple with clear epoxy and screw the nipple into the hole. Then feed more clear epoxy into the area around the nipple. Use toothpick or other thin implement to break bubbles and work the epoxy into the volume. Don't use fast setting epoxy! I also rough up the plastic in this area with a Dremel tool prior to gluing to give the epoxy something to grab to. When the area around the nipple is filled, slide the washer over the nipple, fill the remaining threads with epoxy and install the 3/8 to 1/4 adapter. I strongly recommend doing a dry assembly first if for nothing more than practice. Leave this assembly upside down until the epoxy has cured. As for that tiny side port on the base plate, attach a short length of flexible tubing and pinch it closed with a pinchclamp.



Below are pictures of the completed baseplate and the belljar and baseplate mounted on the manifold and pump.



Figure 2.4 - Completed Baseplate and Chamber Mounted on Pump

The plastic used in the Nalge product is gassy so only modest base pressures will be achievable. This should be in the 100-150 milliTorr range.

## GAUGING

The system shown in Figure 2.1 has a Bourdon gauge for measuring pressure. The Bourdon is a standard piece of equipment for the AVS's high school outreach program but is of limited use in many applications. Elsewhere on my site and in the compilations are details on commercial and homemade thermal conductivity gauges i.e. thermocouple and Pirani. These will operate over the normal operating range of this system (10 milliTorr to a few Torr). The commercial gauge tubes are generally supplied with either 1/8" NPT or KF16 ports. For the pipe thread gauges a KF16 to 1/8 female NPT adapter flange will be required (along with clamp and center ring). KF16 ported gauge tubes will connect to the manifold directly.

## **OPERATING PROCEDURE**

Read the manufacturers' manuals and precautions as supplied with the pump and the chamber. Place the rubber gasket on the bell jar base plate. Ensure that it is flat and centered between the molded ribs. Place the bell jar on the gasket, centering it.

## Pumpdown:

Ensure that the valves are in the following positions:

- \* Pump isolation valve (1/4 turn valve on pump body): Open (handle vertical)
- \* Needle valve: Closed
- \* Main vent (threaded O-ring sealed cap at the pump inlet): Closed
- \* Baseplate vent (pinched tube): Closed

Turn on the pump and monitor the pumpdown via a gauge. At about 1 Torr the sound from the pump will change from a gurgling sound to a clicking sound. If you are pumping moist air, open the gas ballast valve to prevent water from condensing in the pump. Turn off the gas ballast when the moisture has been dealt with in order to get the best base pressure.

## Venting:

Turning the pump off without isolating the pump or quickly (a second or two or less) venting will result in pump oil backing up through the manifold into the chamber. The best method is to:

- \* Close the pump isolation valve while the pump is operating.
- \* Vent the chamber and manifold.
- \* When the chamber is at atmospheric pressure, turn off the pump and open the isolation valve.

Do not stop and restart the pump while under vacuum. This puts a high load on the pump. If the chamber is to be opened and repumped, it is not necessary to turn the pump off. Just control the pumping action by opening and closing the isolation valve.

## Pressure Control

The chamber may be set to any pressure from full vacuum to just below atmosphere by using the needle valve (lower pressures) or the needle valve and isolation valve (higher pressures). At lower pressures, simply open the needle valve to raise the pressure. You will note that even with the needle valve fully open, the pressure remains fairly low. Further increases may be obtained by throttling the pump by partially closing the isolation valve. The needle valve may be used to admit other gases into the system. Under no circumstances should the system be used with flammable, corrosive, toxic or oxidizing gases.

## ADAPTER FOR GLASS CHAMBERS

The standard KF40 flange permits a wide variety of apparatus to be attached to the manifold. I have developed some glass tubes that are available through my website, for example, are examples of the types of apparatus that can be used with this pumping system. To attach glass tubing a compression adapter is required. I have standardized on a 1-3/8 inch coupling as brass couplings in this size will mate directly to a KF40 x 1.625" braze flange. Rather than brazing I use tin-silver solder. This flows easily, is strong and is also easy to rework, should that be desired. A coupling assembly is shown in Figure 2.5 below along with a complete assembly with glass discharge tube.



Figure 2.5 - KF40 to 1-3/8" Tube Adapter

## A Primer on Using Ace Threds <sup>®</sup>

### **Adding Feedthroughs to Glassware**

## **I. INTRODUCTION**

I have frequently mentioned Ace Glass Corporation's [http://www.aceglass.com] line of proprietary Ace Thred<sup>®</sup> connectors. Ace patented the configuration in 1972 [1] as a gas tight, low stress connector. Ace makes the claim that this is "the most important glass connection since the spherical joint." That may or may not be so, but I have found these connectors to be wonderful for vacuum chambers that are designed for service in the ranges down to high vacuum.

The principle of the Ace Thred is shown in Figure 2.6 below. The thred itself is a precision molded connector of borosilicate glass. The glassblower attaches this connector to the apparatus at the desired locations. The other parts of the thred consist of a nylon bushing and a fluorocarbon o-ring. When assembled around an appropriately sized rod (usually aluminum if the rod is serving as an electrical feedthrough), the tightening of the bushing compresses the o-ring between the glass connector and the rod. The bushing is not exposed to vacuum thus making the seal compatible with medium and high vacuum systems.

## **II. SPECIFICATIONS**

The table below shows Ace Glass' published data for the various sizes of Ace Thred bushings. My favorite sizes are numbers 7, 11 and 25. These take rods or tubes in diameters of 1/4, 3/8 and 1 inch respectively.

In my experience the vacuum ratings are on the conservative side. I have used sizes to #25 in applications well below  $10^{-4}$  Torr.

Thred Size	Rod OD (mm)	Vacuum (Torr)
#7	6-7	<10-5
#11	9-10.5	<10 <sup>-5</sup>
#15	12.5-14	<10-5
#18	16-17	<10-4
#25	24-25	<10-4
#36	34-35	Not Rated
#50	47-48	Not Rated
#80	80	Not Rated

Materials selections for the bushings are nylon and teflon. Since the bushing itself is not exposed to vacuum, nylon may be used for all applications except where better temperature performance is required. Plugs





Figure 2.6 - #11 Ace Thred with 3/8 inch Diameter Aluminum Feedthrough

(discussed later) are different in that the nose of the plug is exposed to vacuum.

#### III. SAFETY

In the case of straight rod sections that are inserted into Ace Threds it is essential that some kind of stop be added so that there is no chance of the rod being drawn though the fitting by the differential pressure created by the vacuum in the apparatus. This can be done by machining a step into the rod or, as shown in Figure 2.6, affixing a shaft collar or other device. Shaft collars are convenient since they can be easily repositioned.

I have seen a 3/8" rod get propelled axially through a piece of glassware when the Ace Thred bushing was loosened just a little bit. The result, fortunately was minor damage but the velocity of the rod was high enough to puncture the screened center rings at the pump out port. Needless to say, if the rod were aimed at an adjacent glass wall, it would shatter the apparatus.

As a final note, as with all glass vacuum (or pressure) apparatus, use protective eyewear at all times.

## **IV. AN EXAMPLE**

When I was a teenager I constructed (well, partially constructed - I ran into problems with the van de Graaff generator) a simple potential drop particle accelerator following Frank Lee's design that was described in a 1959 Scientific American *Amateur Scientist* column [2]. A later column [3] described a design for a similar apparatus by Larry Cress. Figure 2.7 shows a rendering of the upper part of Cress' accelerator tube.

Cress' tube consisted of several pieces of 1-1/4 inch diameter pyrex tubes, each about 3 inches in length. The simple ion source, driven by a spark coil, was mounted on a disk of lucite. The acceleration electrodes were disks of brass with 3⁄4 inch copper tubes soldered to holes in the disks. All of the components were joined with vacuum sealing wax (Apiezon W).

My take on an implementation with Ace Threds is shown in the right hand side of Figure 2.7. Please note that I have never built this specific tube but I have done equivalent structures many times.

The ion source is at the top of a tube that incorporates a ring seal and is topped with an 11mm Ace Thred. A sleeve with a threaded hole in the side slips over the inner 5/8" outside diameter tube. A 7mm Ace Thred is to the side and the 1/4" diameter rod has a threaded piece that screws into the sleeve.

The acceleration electrodes are rings that are fitted to the glass tubing. Within the rings (or machined as part of the rings) are tubes, much like the copper tubes in Cress' design. As with the ion source electrode sleeve, a 7mm Ace Thred is associated with each electrode.

The target is held in a side arm that is terminated in a 25mm Ace Thred. This permits easy insertion of the targets.

Finally, the bottom of the tube is a straight section that mates with a compression adapter.

### V. A MULTI-PURPOSE DISCHARGE TUBE

A "real" design is shown in Figure 2.8. This tube may be used as an x-ray source, electron accelerator or simple ion accelerator. A Langmuir probe may also be inserted into the tube.

At the top of the tube is a #11 Ace Thred. The top section of glass is sized at 1 inch inside diameter so that 1 inch rod and tube stock can be used for electrodes. A #7 feedthrough is provided just below this section. This may be used for a number of purposes such as for a bias or extraction electrode. This is also where a Langmuir probe would be inserted.

The bottom side feedthrough is a #25 Ace Thred. This can be used for targets, to hold a phosphor screen,



Figure 2.7 - Accelerator Columns

or as a sample holder if items are inserted for ion or electron irradiation.

At the lower end is a straight section of 1-3/8 inch diameter, sized to fit a standard compression fitting.

Figure 2.8 shows the tube in a simple cold cathode x-ray tube configuration. The cathode is an aluminum "dish" 1 inch in diameter. The target holder is made from 1 inch rod stock with one end milled half way through. Two 8-32 holes are drilled and tapped in the flat section, about 1 inch apart. The target is made from sheet tungsten (salvaged from an evaporation boat) that is fastened by clamping the sheet under the heads of the two screws. Figure 2.9 is a photo of the same tube in the cold cathode x-ray configuration.



Figure 2.8 - Multi-Purpose Discharge Tube

## VI. PLUGS & ELECTRICAL FEEDTHROUGHS

Another useful piece of Ace Thred hardware is the face seal plug. As shown in Figure 2.10 these are simply solid bushings that have an o-ring groove near the tip. (There are also plugs that seal just under the head of the plug but these expose the entire length of thread to vacuum and, in my experience, are not as reliable under vacuum.)



Figure 2.9 - Tube Configured for X-Ray Production

Plugs can be used to make electrical feedthroughs and are especially valuable when several connections are to be made and where the plug might serve as the support for a subassembly that will be in the vacuum chamber.

Materials selection is important in the case of plugs as the plug material is exposed to vacuum. I've used nylon plugs in the #36 size down to 1 micron and teflon #50 well below that.



Figure 2.10 - Ace Thred Face Seal Plug

My method for making a feedthrough is shown in Figure 2.11. This is a 2 electrode feedthrough that I made for a miniature evaporation chamber that's being used at a technical college. In this case I used a 36mm plug made of nylon.

I used 5/16 inch diameter aluminum rod with 8-32 hardware. I drilled the plug part way through the same diameter as the rod and then continued each hole to clear a length of 8-32 brass threaded rod. The seal is made by placing a small o-ring on the threaded rod



Figure 2.11 - Feedthrough

where it will be compressed at the bottom of the aluminum rod when the hardware is tightened. A suitable o-ring for this application would be 5/32" inside diameter x 9/32" outside diameter (1/16" cross section). Needless to say, the bottom of the aluminum should be machined to a smooth finish.

The smallest rod that I have used is 1/4" diameter with #6-32 hardware. A suitable o-ring would be 1/8" inside diameter x 1/4" outside diameter (1/16 cross section).

Figure 2.12 shows an RF feedthrough built for a small RF sputtering chamber. The connector is an HN bulkhead mount type . The wire that is shown is the ground connection that goes from the connector housing to the substrate holder.

## **CITED REFERENCES**

 Charles M. DeWoody, *Flexible Pressure-Type Joint* for Rigid Tubing, US Patent 3.695,642, October 3, 1972.
C. L. Stong, *How to Make an Electrostatic Machine* to Accelerate Both Electrons and Protons, Scientific American, June, 1959.

[3] C.L. Stong, *How to Build a Machine to Produce Low Energy Protons and Deuterons*, Scientific American, August, 1971.



Figure 2.12 - RF Feedthrough

## **Common Vacuum Flanges and Seals**

#### **INTRODUCTION**

Connections between components in vacuum systems may either be permanent connections made typically by welding or brazing or demountable connections using a variety of flanges and fittings.

Most vacuum systems tend to be modular, using standard components wherever possible. The manufacturers of vacuum components such as valves, gauges, flow controllers, etc. generally offer their products with a selection of standard fittings so that the user can match those items to their specific hardware and application. There is also a wide variety of standard vacuum component hardware including straight tubes, bends, tees, crosses, reducers and so forth. These come in varying diameters and lengths, all equipped with an assortment of standard flanges.

Any kind of connection poses an opportunity for leaks so it is important that the person working with vacuum hardware knows how to properly use and care for the various fittings.

There are too many specific configurations of separable connector to cover each one. This chapter will concentrate on the principles behind the two major classes of demountable fitting: the o-ring sealed flange as exemplified by the ISO-KF system and the metal sealed flange as exemplified by the CF system. Other sealing systems will vary in terms of their physical manifestations but their principles are the same. The reader who is interested in specific dimensional data is invited to visit the various manufacturers' catalogs. A selection may be found in the Appendix.

## ISO KF O-RING SEALED FLANGES

The ISO-KF series is widely used in low and medium vacuum applications and may be used selectively in high vacuum equipment. "KF" refers to the original name "klein Flansch" or "small flange." KF flanges may also be referred to as QF ("quick flange"), NW or DIN. The KF system is standardized and is recognized by ISO, DIN and Pneurop.

Each flange is sized according to the largest tube ID that can be welded to it. The outside diameter is a standard North American tube dimension, in inches. The standard KF flanges are KF16 (0.75 inch OD tube ), KF25 (1 inch OD tube), KF40 (1.5 inch OD tube) and KF50 (2 inch OD tube).

Figure 2.13 on the next page shows the design features of the KF series flanges. Each flange set consists of a pair of similar flanges and a metal ring (center ring) and o-ring assembly. The center ring has three functions: it positions the o-ring on the flange seal area, it maintains the ID of the o-ring against the force of atmospheric pressure and it determines the amount of o-ring compression when the flanges are drawn together.

In addition, a clamping assembly holds the flanges together. The clamps usually consist of two hinged aluminum half-rings that encircle the flanges and are closed with a thumbscrew or toggle. When tightened the clamp draws the flanges together against the center ring.

The seal is created by the compression of the o-ring. The amount of compression is important. Over the life of the seal the o-ring has to maintain an adequate amount of force on the flanges. Too little compression and the seal will be poor or unreliable. Too much and the seal may fail over time if the o-ring material takes a "set" and the degree of force declines. The shoulder on the center ring has a width that assures a proper amount of o-ring compression (about 25-30 percent).

KF seals are designed to work with finger tightening of the clamp. The o-rings are used dry - no grease or lubricant. (Greases are traps for contaminants and should never be used with anything but sliding seals.)

There are a wide variety of materials choices for the o-rings. The default is viton but other materials may be used depending upon application (temperature, chemicals, radiation, etc.).

Periodic inspection of o-ring seals is advisable. Issues to look for include excessive set, flow of the o-ring, embrittlement, tackiness and cracking.

In the figure, note the weld. Proper vacuum welds are made from the inside of the tube and are generally in the form of a butt weld. This minimizes the possibility of trapped areas that can create virtual leaks. The standard welding process for flanges and tubing is orbital tungsten-inert gas welding.

For larger sizes of tubing there are ISO extensions to the KF series, for example the LF ("large flange") series. This uses the same general sealing geometry but with bolts or clamps to draw the flanges together. Bore sizes range from 63 to 1000mm.



Figure 2.13 - KF Flange System

## **CF METAL SEALED FLANGES**

O-ring seals such as the KF system are very easy to use. Since they do use elastomers, there are applications limitations at high and ultrahigh vacuum and with some process materials. In these cases an all-metal configuration is required. The most commonly used all-metal flange system is the CF. The CF is derived from Varian Corporation's trademark

ConFlat design. The CF is not formally standardized but the equivalent CF flanges from other reputable manufacturers are interchangeable.

Referring to Figure 2.14, the CF flanges use a knife edge that bites into a soft gasket. The gasket material is usually OFHC copper. The knife edge has a shallow angle on the outside and a slightly off-vertical angle on the inside. The seal is made in the area where the steeper side of the knife edge has cut into the gasket.

In assembling a CF flange the bolts are sequentially tightened so that the force on the gasket is uniform. In the tightening process the perimeters of the flanges are slightly deformed. This creates something equivalent to a hairpin spring that extends from the perimeter of one flange, through the knife edges and back to the perimeter of the other flange. This effect produces a very strong and enduring force on the seal area.

When a CF flange set has been properly tightened there will be a uniform gap between the flanges (purists will use a feeler gauge to set the proper gap). Tight bolts and a uniform gap are signs that the flanges have been coupled correctly. No gap usually means that the assembler has forgotten to insert a gasket.

Never reuse a metal gasket. The resulting seal may leak immediately or it may fail at some random time. Given the number of bolts that have to be tightened the cost of the gasket is small compared to the time and effort required to redo the job.

The CF flange system is very robust and represents essential hardware for high and ultrahigh vacuum. Mated flanges will survive the large temperature excursions that are required for bakeout and they are free of the outgassing and permeation issues that render o-ring systems inappropriate.

CF flanges are commonly available in sizes from 1-1/3" to 10" OD. Single piece as well as two piece rotatable flanges are manufactured.



Figure 2.14 - CF Flange System

## OTHER METAL SEALED CONNECTORS

For smaller lines the VCR system developed by Swagelok Corporation is widely used. These will be found on some vacuum gauges, MFCs and instruments that deliver gases to vacuum systems. These connectors use screw male-and female fitting pairs and a small metal gasket. Made for tubing sizes from <sup>1</sup>/<sub>4</sub> to <sup>1</sup>/<sub>2</sub> inch these are essentially miniature versions of the CF.

## DYNAMIC SEALS

The seals described above are static seals. In many cases it is necessary to transmit rotary motion into a vacuum system for moving a fixture of rotating a valve "flapper." This is generally done with an o-ring that is placed in a groove on the rotating shaft as shown in Figure 2.15

The o-ring is placed in a groove that has been machined into the shaft. The dimensions of the groove are such that the o-ring will receive the correct amount of compression and will stay within the confines of the groove when the shaft is inserted into the housing.

Dynamic seals are one place where the o-ring should be lubricated. The lubrication is not related to the seal itself but is required to ensure that the o-ring can slide in the housing without sticking or tearing. The amount of lubricant required is very small - just enough to give the o-ring a shiny surface appearance.



Figure 2.15 - Dynamic Shaft Seal





## 3. Example Projects Covered in the Bell Jar



Richard Hull's Fusor III. This device uses Inertial Electrostatic Confinement (IEC) to produce neutrons via the  $D_2$ - $D_2$  nuclear fusion reaction. The active fusion region in a demo device is shown below. This is a very active program involving many amateurs. More info at http://www.fusor.net.





Above: Ions from the author's plasma gun shown being magnetically separated by mass and charge by the magnet that is located just above the gun's muzzle.

Below: Thomson e/m apparatus developed by the author for a historical exhibit. The manifold is fabricated primarily out of copper pipe fittings. Electron gun is left, deflection electrodes and magnets at center and phosphor screen at right.





pump will keep going to a hard vacuum, the stopcock must be closed when the desired vacuum is reached. Pressure rises in the tube are compensated for by re-opening the stopcock.

George reports that with a 4" gap spark coil he can view the bones in his hand with a fluoroscope screen 1 foot from the window.



X-ray apparatus is fairly simple to make and can be used in many activities. Like any radiation producing device, x-ray tubes require shielding, montoring and great care in use.

To the left is Jon Rosenstiel's vacuum stand with home made x-ray tube just above center right in the photo.

Below is a picture of the tube, fabricated from surplus glassware and metal components. Nothing fancy.





An x-ray of an integrated circuit.



Another home made x-ray tube. This one is by Tim Raney and is modeled after a Crookes tube with a displaced anode.





Plasma ashers are a valuable tool in materials research and biological analysis. RF activated molecules react with organic materials to convert them to gaseous species (typically carbon dioxide and carbon monoxide) which can be pumped away. High quality microwave ashers can be built using discarded microwave ovens adapted with simple internal glass (generally pyrex or quartz) chambers. Needless to say, caution must be exercised when modifying a microwave oven to prevent radiation leakage. Also, if oxygen is used, the vacuum pump must use an inert pumping fluid to prevent fire or explosion.

The photographs above show a reactor built by Hideaki Page.



Ion sources may be used in particle accelerators, etching systems and deposition apparatus. Ion source design and development is a fertile field for the amateur. The photo above shows a saddle field source that is used to produce fast beams of neutral atoms. The source was built by the author and uses a surplus vacuum tee, a couple of flanges and some bits of stainless steel and ceramic tubing.



Here is a disassembled view of Carl Helber's quadrupole mass spectrometer. Below is a mass scan clearly showing the merged water peaks and the nitrogen peak.

