The First Five Years
1992-1996

the **Bell Jar**

Vacuum Technique and Related Topics for the Amateur Investigator

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1992-1996

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. Further information on this journal will be supplied on request from:

***the* Bell Jar**

http://www.belljar.net

A Note on Safety

Notice of Disclaimer: Many of the topics, projects, and materials discussed in this journal 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, neither the editor nor the authors may 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. Several suppliers of radiation detection instruments are listed in the Appendix.

Contents

Forward

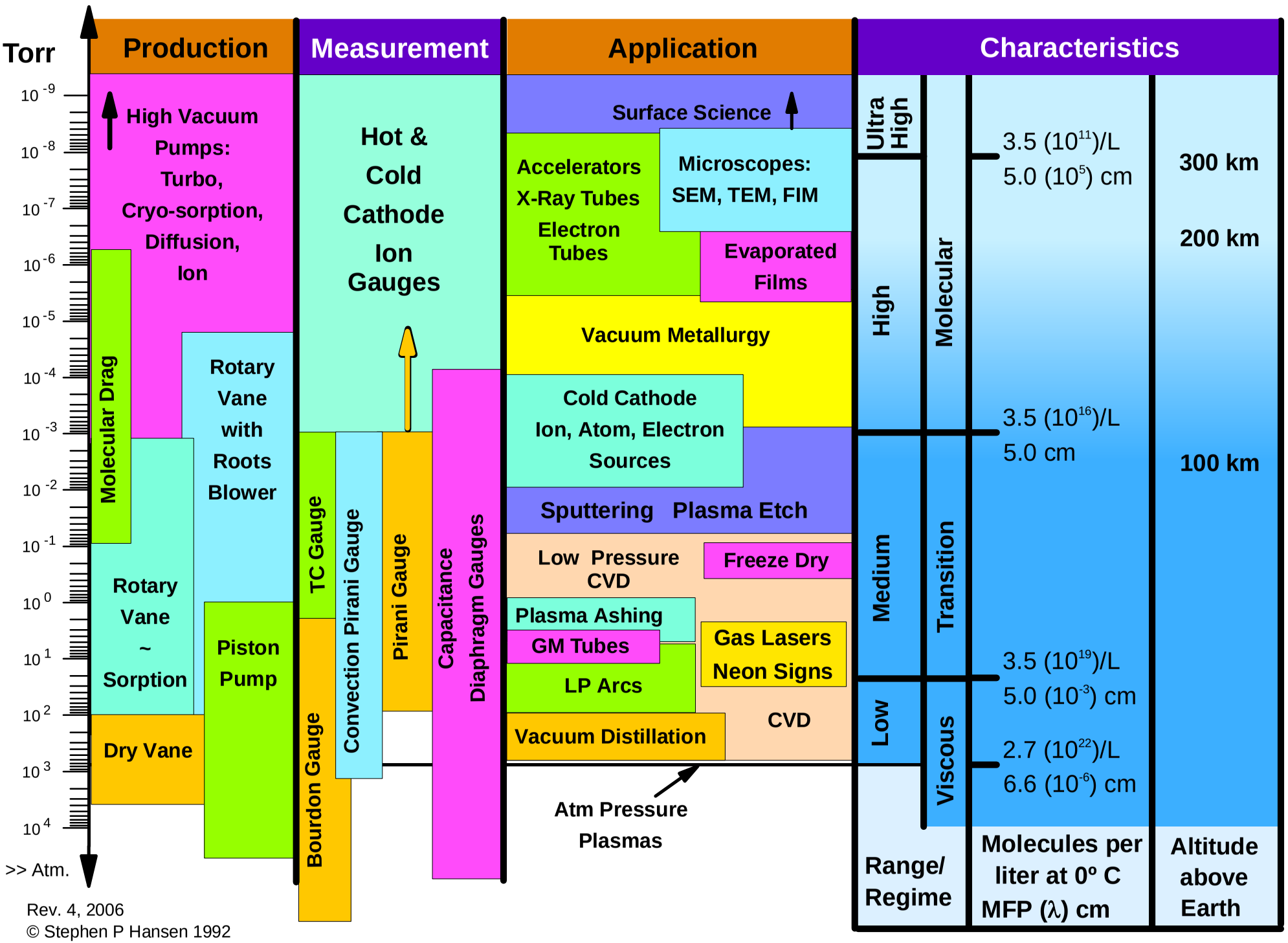
The incentive for producing this compilation came from several factors: I was getting tired of handling the boxes and folders containing the hardcopy issues; My wife, Chris, was getting tired of these same boxes, strewn all around the upstairs bedrooms; Volume 1, originally produced on an obsolete word-processing typewriter, existed only in print form and the originals were getting rather dog-eared; Some of the later issues were in peril because a number of the electronic files had gotten corrupted; A certain percentage of each issue (classifieds and the like) had gotten outdated; The sets of back issues were beginning to cost more than I thought they should.

As a result, I have finally produced the work now at hand. When I announced this project, over one year ago, I thought it would be pretty easy to pull up all of the files and stick them together in some coherent form. However, for some of the reasons noted above, and because I just had to tinker a bit, it turned into a larger project.

In this compilation are all of what I feel are the useful articles from the 20 issues that comprised Volumes 1-5. To keep the length manageable and the content relevant, I did cut out the time-sensitive items and the various bits of commentary. I will keep my eye out for items that should have been retained in this compilation but fell through the cracks. They will be collected and will appear at some future date, either in the next compilation or in a regular issue. I did try to correct a few errors, although I'm sure that there are still a few lurking in these pages. As I find these (or they are brought to my attention) I will publish corrections in later publications and/or on line. Some attempt was made to group articles by topic so referring to a related article shouldn't be difficult.

Steve Hansen
November 1999

The chart on the next page depicts how vacuum is produced (types of pumps), how it is measured (types of gauges) and how it is used. This compilation will touch on most of these items.


Torr
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High Vacuum Pumps: Turbo, Cryo-sorption, Diffusion, Ion
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Rotary Vane ~ Sorption
Piston Pump
Dry Vane
Hot & Cold Cathode Ion Gauges
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Pirani Gauge
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Surface Science
Accelerators X-Ray Tubes Electron Tubes
Microscopes: SEM, TEM, FIM
Evaporated Films
Vacuum Metallurgy
Cold Cathode Ion, Atom, Electron Sources
Sputtering Plasma Etch
Low Pressure CVD
Freeze Dry
Plasma Ashing
GM Tubes
Gas Lasers Neon Signs
LP Arcs
CVD
Vacuum Distillation
Atm Pressure Plasmas
Ultra High
High
Medium
Low
Molecular
Transition
Viscous
3.5 (10^11)/L
5.0 (10^5) cm
3.5 (10^16)/L
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3.5 (10^19)/L
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2.7 (10^22)/L
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Range/ Regime
Molecules per liter at 0° C MFP (λ) cm
Altitude above Earth
Rev. 4, 2006
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Part 1: Fundamentals and Tutorials

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

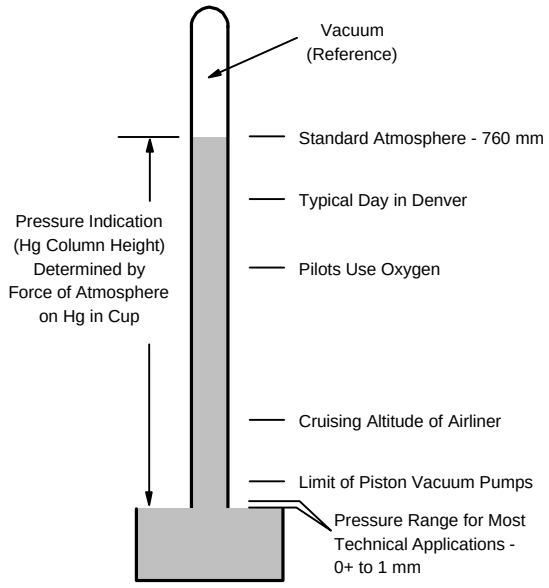

Figure 1.1 - The Mercury Column Barometer

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^{-6} Torr vacuum. At 10^{-10} 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

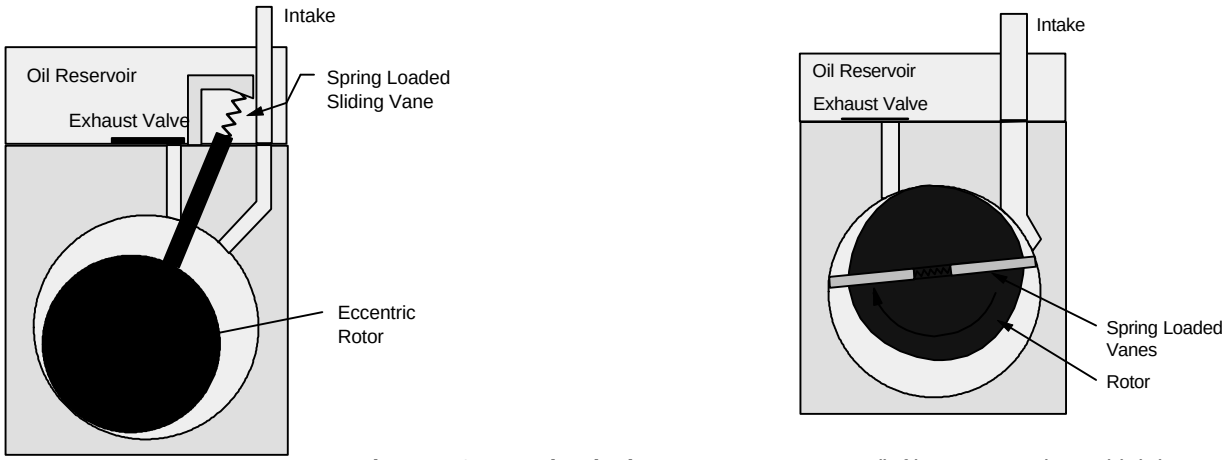

Figure 1.2 - Mechanical Pumps. Rotary vane (left) & rotary piston (right).

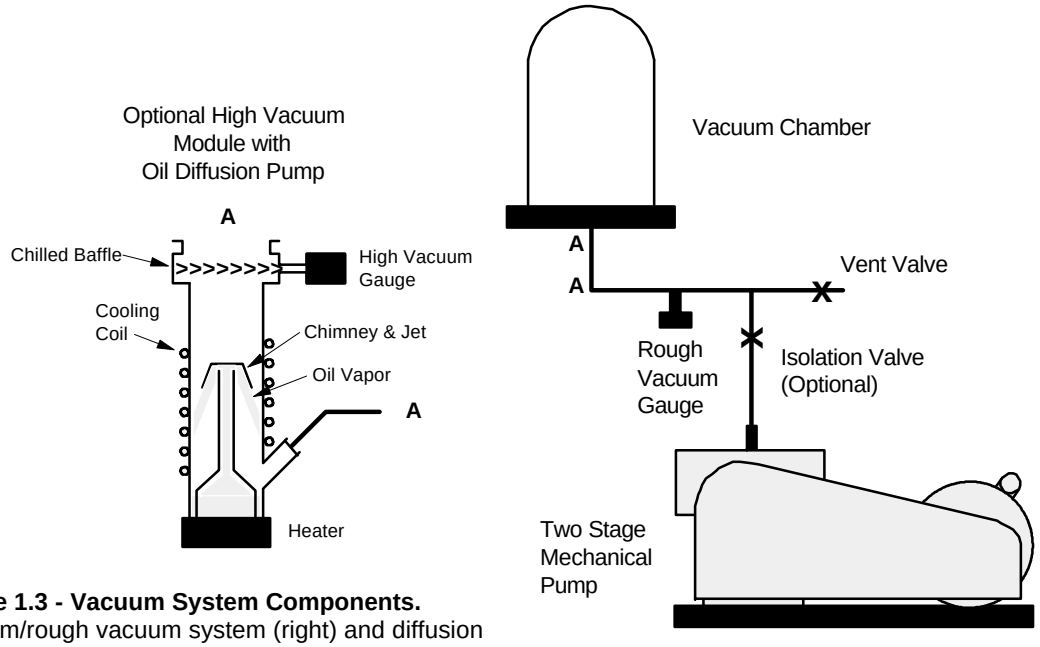

Figure 1.3 - Vacuum System Components.
Medium/rough vacuum system (right) and diffusion pumped high vacuum stage (left).

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 (l_{ave}) to the radius of the tube (r) is less than 0.01, the flow is viscous. When the ratio l_{ave}/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):

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

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:

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

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° 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° 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.

The Writings of Michael McKeown

Mike McKeown notes that he is a European happy to live in the USA despite this country's insistence on measuring everything in Imperial Units. He is the Marketing Manager for Kurt J. Lesker Company. My thanks to Mike for providing the following articles. Other insights by Mike may be found on the Kurt J. Lesker website at http://www.lesker.com.

The Units of Pressure Measurement

This article was originally presented in Volume 1, Number 4.

That wonderful commentator, James Burke, gave an excellent account of the beginnings of vacuum science in his PBS program “Connections.” The following is based on my recollection of Burke’s story and is not, therefore, guaranteed to be accurate. The nationality slurs are my own. Burke is too polished to sink to this level.

All vacuum problems started with the Italians. There was one obstacle to mining in Italy in the early 1600s ... Water. It was everywhere. Before the miners could dig, the water had to be pumped to the surface. It irked them that their ‘suction’ pumps could only ‘suck’ water up to a height of 32 feet above flood level (or rather, the contemporary equivalent of 32 feet). At that point, the pump effluent had to be spilled into vats and another pump used to suck the next 32 feet. Why couldn’t they use one pump for the whole distance? What was magical about this height of 32 feet?

They posed the problem to Galileo but he did little with it until three months before his death when he tossed it to a mathematician named Torricelli who came to study under him. Torricelli had been kicking around ideas about ‘oceans of air’ surrounding us and concluded that he could bring this pump problem to a manageable size by using a fluid denser than water. Mercury seemed a good choice. He had his assistant fill a glass tube (closed at one end) with mercury, placed the open end in a dish with more mercury, and raised the closed end. The mercury reached a level equivalent to 32 feet times the ratio of the densities of water to mercury. What would be above the mercury if the glass tube was long enough? A vacuum, of course!

Ah, there’s the rub, as Shakespeare said about the same time. Galileo believed that vacuums (vacua?) could not exist and he had already been put under house arrest by the Church for saying things like the earth went around the sun. There was no way Torricelli was going to broadcast the results of the ‘vacuum experiment’ himself. But he did write to a friend in Rome who copied the letter as sent it to a Father Mersenne in Paris. Mersenne, a minorite friar, acted as sort of a medieval computer bulletin board. He promptly copied the letter again (where was Xerox when it was needed most?) for his friend Blaise Pascal who lived close by and therefore, at a sufficient distance from Rome to ignore the Church’s word - to a certain extent.

Pascal, being a literal kind of man, set up the experiment in full scale using water and ‘mirabile dictu’ confirmed the existence of a vacuum. It followed from Torricelli’s ideas that if the weight of air pushed mercury so far up the tube, then the mercury level would be reduced if the test were done at higher elevations. Blaise Pascal’s brother-in-law lived in central France in an area surrounded by mountains and was apparently adventurous (and strong) enough to march a complete mercury barometer to the top of the nearest mountain. The rest (to use a very bad pun) was downhill from there.

In my opinion, Torricelli, a mathematician, was a premature software guy. Blaise and his brother-in-law got the job of proving the prediction worked on a grand scale and up a mountain, less because of the Church’s decree and more because Torricelli was reluctant to get his hands dirty. Whatever the reason, Torricelli was honored later by someone naming the pressure measurement unit of 1 millimeter of mercury, the torr.

Nice story, but the Battle of the Units had only just begun. Life with torr would have been lovely, except the Brits had to tinker with it. Liking everything to be in Imperial Units, they converted the 760 mm Hg pressure of the standard day to 29.92 inches Hg. Well, if you like inches, that's ok. But notice how the weather forecasters on American TV long ago forgot that it was the height of mercury they quote every night. Every one of them says, “The barometer is 29 inches and rising.” What's this ... psychokinesis?

The inch thing really got out of hand when manufacturers of rough pumps came on the scene. Rough pumps are arbitrarily defined as those used for: in-house vacuum systems; meat packing; impregnating lumber and transformer coils; making freeze dried coffee, tea or foods (got ya!). That is, any pump that hauls great loads of gas and vapor

day-after-day to a modest vacuum level. These manufacturers noted that if atmospheric pressure was 29.92 inches Hg, they would be shooting for 0 inches. That would look bad in their brochures. So, they calmly inverted the scale. Atmospheric pressure is 0 inches Hg and the best possible vacuum is 29.92 inches Hg, they said. Which left the rest of us struggling with converting inches Hg to torr. (First, subtract the given inch pressure from 29.92 inches, then multiply the answer by 25.4.)

Since the standard meter and kilogram are kept in Paris, I blame the French for the metric system. Not that I really object to it. After all, I accept 760 Torr is really 760 mm Hg without too much argument. But someone, somewhere, noted that 760 mm Hg could not be related to any basic measurement units. The column's height depended on the mercury's density and that wasn't basic in anyone's scheme. "Let's make the pressure unit conform to the cgs (centimeter/gram/second) system" they said with glee, knowing how much they would confuse the rest of us.

And how is this done? First, we must understand that "pressure" is force per unit area. What's the unit of force in the cgs system? Remember way back at school, your science teacher smacked your knuckles for not remembering that the dyne is that force which gives 1 gram an acceleration of 1 centimeter per second per second? You really should have listened because pressure in the cgs system is measured in dynes per square centimeter or, to give its proper name, microbar. Why micro (millionth) bar? Well, try this for an explanation. One million microbars (or 1 bar) is 750.06 torr. That is, the atmospheric pressure of the standard day is 1.0133 bar which is easier to remember than a number which must be multiplied by ten to the "minus oh-dear-I've-forgotten." The cgs advocates rounded off this part of the story by declaring pressures will be measured in millibar (because it's close in value to the torr?). Indeed, to this day, millibar is the unit used for recording both vacuum and weather pressures in Europe.

If I can blame the French for the cgs system, what defence can they offer for the next leap? Some august body set up an international standard for measurements called *Systéme International d'Unités* (or SI). What could be more French than that? They threw out the cgs system's claim to fame and installed the MKS (meter/kilogram/second) system. The unit of force in *SI* units, that is the force to accelerate 1 kilogram at 1 meter per second per second, is called the newton. Don't you get the flavor of collusion between Brits and French here? And what do you think the MKS pressure unit (1 newton per square meter) is called? You've got it - the pascal! I rest my case.

Of course, since the units of length and mass in cgs and MKS are all related by powers of ten, millibar (mbar) and pascal (Pa) have the simple correspondence:

100 Pa = 1 mbar (= 0.75 Torr)

But does that really make you sleep easier at night? If you want nightmares, try this one. In pressure measurement, when does a milli equal a micro? Answer: consider the humble torr. If one Torr equals 1 millimeter of mercury, then 1 milliTorr must be equivalent to 1 micrometer of mercury, right? And what do we call a micrometer? - a micron. All of which says that:

1 milliTorr = 1 micron Hg

But how often have you heard someone quote a pressure of "250 microns of mercury?" We all think of the unit as 'micron' leaving the neophyte vacuum person with the impression that a milli (unit) equals a micro (unit). Just remember, like the weather forecasters we have simply forgotten to add "mercury" to the microunits.

Outgassing

This article was originally presented in Volume 2, Number 3.

This is all you ever wanted to know about *outgassing* and then a whole lot more piled on top. Before you finish, if you ever do, this will seem like the biggest con job, the most outrageous scientific sleight-of-hand ever perpetrated. It is, except for one small point ... it's all true.

What does outgassing mean to you? Well, on a good day, you probably think it's a measurement that tells you the amount of *gas* that's coming *out* of something. If you are pushed really hard you'll probably say 'out' doesn't mean out as much as 'from the surface of.' And does it mean from the surface of any old thing? Yes, except you forgot to mention that it's only important if that 'thing' is under vacuum. Nobody cares if something outgases in the atmosphere, unless it's smelly.

Outgassing, then, is the amount of gas leaving some surface when we are trying to pump out a chamber. What do we mean by 'amount?' Do we mean the pressure? No, that doesn't sound right because if we have two large chambers, one big and one small, at the same pressure, we just know there is a larger amount of gas in the big chamber.

Does that mean volume then? That sounds better. Still, if we have two chambers with the same volume but at different pressures, doesn't the chamber with the higher pressure have a larger amount of gas? 'Fraid so.

Well, can't the amount of gas be defined as *pressure x volume*? Now you're cooking. You give someone this volume of gas at that pressure and they can press it, squeeze it, spindle or fold it, expand it, contract it, make it real long and thin, count the number of molecules, or do whatever they choose and it will always be the same amount of gas. (Strictly, you have to give the temperature as well as the pressure and volume, but why complicate things?)

Does this mean, if we state the pressure and volume of gas leaving a surface, then we've specified the outgassing? Well, no. Think about two surfaces made of the same material, one foot by one foot and the other one foot by two feet. Would you expect the same amount of gas to outgas from both? No. You would expect the larger surface to give twice the amount of gas as the smaller. That suggests we must make outgassing depend on the *area* of the surface. Putting amount and area together, maybe outgassing is ***pressure x volume per area.***

Hey, that's terrific. We must be getting close. But there is still a little hitch here. Suppose you had two identically sized, identical looking surfaces made of different materials, one of which outgassed a great deal, the other outgassed hardly at all. How would you expect to tell the difference between them? If you asked me, I'd dump one sheet in a vacuum system, pump it to some low pressure which I'd note, shut off the pumps, wait for five minutes and read the pressure again. Then I'd remove that sheet, put in the other one and do exactly the same test. The sheet that gave the higher pressure rise after five minutes is the one that outgasses a lot. Outgassing must have time involved in its measurement because it's a rate.

Eureka! So after all of that huffing and puffing, outgassing is simply:

Pressure x Volume per Area per Time

Who said this subject is difficult? It's child's play. It's so easy we can play with different units of pressure, volume, area, and time and specify outgassing rates. How about:

torr x liters per sq. centimeter per second ... ?

- or -

pascal x cubic meter per sq. meter per second ... ?

Both sound great to me. Hey, we getting good at this and if we wanted to be really perverse (and who doesn't?) this should do it:

micron Hg x gallon per hectare per millennium

It too is perfectly fine because it follows the Pressure x Volume per Area per Time formula that we know specifies the outgassing rate.

But before you go off with a rosy glow, listen up 'cos this is where the story really starts. If you have followed every argument so far, you probably suspect that what's written is all true. However, if you have an enquiring mind, about now is the time to rush to your copy of John O'Hanlon's *A User's Guide to Vacuum Technology* 2nd edition, page 445. There you will find outgassing rates quoted in W/m^2. Huh? Say what? Or more correctly, say *Watt*? Yes, that's right. O'Hanlon quotes outgassing in *watts per square meter*.

How can that possibly be? How, in the name of James, did watts get into this act? Where's the 'time' that I claimed was necessary since outgassing is a rate? How did I hoodwink you into thinking I knew what I was talking about?

No doubt such questions are valid but wait a while. This is where the scientific legerdemain comes in. And remember, I did warn you. Let's start back with the units of outgassing that I labeled:

pascal x cubic meter per sq. meter per second **(1)**

Now, remember that Pascal is the *SI* pressure measurement unit and is defined by *pascal = newton per square meter* ('per' being the same as 'divided by') and therefore the outgassing rate units in (1) above look like

$$\frac{\textbf{\textit{newton x cubic meter}}}{\textbf{\textit{square meter}}} \quad \textbf{\textit{per sq. meter per second}} \quad \textbf{(2)}$$

I've separated the expression so we can concentrate on the first bit, where we see cubic meters divided by square meter. Hmm. I should know what to do with that. Doesn't that just equal meter? Sure does. So a rewrite looks like:

newton x meter per second per sq. meter (3)

Notice how I cunningly changed positions of 'per Square Meter' and 'per second' in the right hand bit? More of that later, for now watch what happens to the 'newton x meter' part because we are about to do some wizardry. If you apply a force of 1 newton for 1 meter you are doing 1 joule's worth of work. If you don't believe me, look in the *Handbook of Chemistry and Physics* under the definition of a joule (page F88 in the 67th edition). Using this expression (3) now becomes:

joule per second per sq. meter (4)

If you are still paying attention, you see that I dragged the 'per second' chunk from right to left, leaving the 'per square meter' on the right since I know O'Hanlon's expression has 'per square meter' too. From here, it really is only a hop, step and jump to home base. If you look again at the *Handbook of Chemistry and Physics* under the definition of joule, it also states that:

joule = watt x second

That is, if you apply 1 joule of work to a task, it is the same as applying 1 watt of power for 1 second to the same task. From this, 5th grade arithmetic dealing with dividing both sides by the same thing tells us:

joule per second = watt

Going back to expression (4) we find 'joule per second' there large as life and twice as ugly. Doing what we mathematicians call a substitution (which means sticking one thing in place of another) we see that *Outgassing* is given in *SI* units as:

watts per square meter

Impressed? I sure am. Truth is I'm much more impressed by O'Hanlon giving me the conversion units that lets me get from his, or anybody else's, quoted outgassing values in watts/sq. meter to torr-liters/(cm^2-sec) which is the first outgassing unit I gave you hours and hours ago:

$$\frac{\textbf{watts per sq. meter}}{\textbf{1333.2}} = \textbf{torr-liter/(cm}^2\textbf{-sec)}$$

Wow! Is this O'Hanlon fellow a good guy or what?

So, does all of this 'watts per square meter' nonsense mean anything to us vacuumists? Is it something to do with the work that's needed to get the gas off the surface? Maybe and No are the answers to those questions. The watt is the power or rate of doing work. In this case it's the power available in the gas as it leaves the surface.

Imagine a surface with an outgassing rate of 10^{-3} W/m^2. According to results quoted by O'Hanlon, slightly rusted steel that has just been put into a vacuum outgases at this rate. If you (magically) suspended a plastic sheet of 1 square meter weighing 1 gram, a short distance from the surface and evacuated the far side of the sheet to zero pressure (no atoms or molecules at all!), then after 1 second the sheet would have moved 100 cm away from the surface and be moving at a velocity of 100 cm/sec and accelerating. The gas atoms and molecules leaving the surface would push the sheet (since there is no corresponding gas pressure on the other side). The work done on the sheet in 1 second under those conditions is 1 milliwatt (or 10^{-3} watts as given in the first line of this paragraph).

Take a more common vacuum material, electropolished stainless steel, that has been under vacuum for 10 hours. O'Hanlon quotes its outgassing rate as almost 3×10^{-7} W/m^2. To move the sheet 100 cm away and with a velocity of 100 cm/sec (at that instant) in 1 second means the sheet cannot weigh more than 0.3 milligrams - the merest gossamer.

Oh, come on! Who am I kidding? We'll accept that a set of European burgers, wrapping themselves in *SI*'s cloak of dignity, has dictated that we will use weird units which, by some fluke, equate to watts per square meter. But does this work-on-gossamer stuff really mean anything to we *vacuistic cognoscenti*? Isn't it more in the province of the *vacuous literati*? Truth is, I don't know. I presume it means something to, say, NASA scientists. I expect they have to calculate the effects of the 'solar wind' from the sun's outgassing on their large solar panels, if photon radiation pressure doesn't swamp all other effects. Help me out here, Werner!

Comments from Bill Harrison of Blacksburg, VA: I enjoyed Mike McKeown's somewhat humorous article on outgassing in the last issue. His intuitive development of the *Pressure ▪ Volume per Area per Time* expression was very interesting. Incidentally, the product of pressure and volume produces the dimension of energy. For example, if you fill a 500 gallon tank in the back of your pickup truck with air at a pressure of 100 psi then you will be driving around with

about 1.3 megajoules of energy on hand for whatever use you might want to make of it. If desired, the *Pressure ▪ Volume per Area per Time* outgassing term could be referred to as the *Area-specific Energy Flow Rate*, which perhaps also some intuitive relevance to the outgassing process in general.

Mr. McKeown has quite a few expressions in his short article but only three equations, namely:

$$joule = watt \times second$$

$$joule\ per\ second = watt$$

$$(watts/sq.\ meter) / 1333.2 = torr\text{-}liter / (cm^2 - sec)$$

Now, the first two are certainly correct, absolutely and totally consistent with the *SI* system of units. However, the third is not a valid equality. I recognize the point that writer McKeown is trying to make through the use of the equation, namely that a numerical value taken from O'Hanlon's work and appearing there in units of W/m^2 can be divided by 1333.2 to produce a new number which will have the units of $torr\text{-}liter / (cm^2 - sec)$. I hope your other readers also recognize that this is indeed McKeown's apparent intention in presenting the equation. However, the conversion factor implied is nevertheless incorrect and should instead be written:

$$1\ torr \cdot liter / (cm^2 \cdot s) = 1333.2\ W/m^2$$

This can easily be confirmed by first recalling that, in the *SI* system of units, the equation:

$$1\ W = 1\ N \cdot m/s$$

can be thought of as a definition for the watt (actually 1 J/s), where N is the Newton and s is seconds. Now, the Torr unit of pressure can be expressed in *SI* units as:

$$1\ Torr = 133.322368421\ N/m^2$$

Combining the previous two equations will eventually give:

$$1\ W/m^2 = 7.5 \cdot 10^{-4}\ Torr \cdot liter / (cm^2 - s)$$

An interesting calculation involving W/m^2 can be made using an ordinary household lightbulb, which has a surface area of approximately 1/100 of a square meter. If a 25 watt bulb is used (which is about the lowest wattage to be found in this larger size bulb), then the power per unit area is approximately 2500 W/m^2. This number is about 10 orders of magnitude greater than values commonly encountered in outgassing calculations, and really has no relevance to outgassing problems whatsoever. It serves as an interesting calculational exercise only.

Mike McKeown replies: Bill Harrison points out that as an equation my expression is simply wrong. I should have written out my expression longhand: 'If you are given an outgassing value as qqq x 10^{-q} W/m^2 and you wish to convert it into a value in Torr-liter/cm^2/sec units, then divide the numerical value qqq x 10^{-q} by 1333.2'. I apologize for any confusion I may have caused. I know it is difficult enough remembering whether to divide or multiply by the conversion factor without me throwing an incorrect equation at you. But it is nice to know that at least one person out there reads an article on the conversion of measurement units. Bill, my thanks.

Pump Oils and Other Furry Animals

This article was originally presented in Volume 5, Number 2

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PROLOGUE

Blame Steve for this article. I kept putting him off, hoping he'd forget. "Well, you see Steve, an article of that length ... with my work load. Sheese....." But, nooooo. Like every editor, he's pushy. And in case you don't know it, he's also big. I find that combination very convincing. So, here's the scoop on pump fluids. Unfortunately, until Steve springs for a decent DTP program, you're stuck looking at scruffy, word processing depictions of molecules.

First I'll survey the general properties of vacuum pump fluids needed for mechanical and diffusion pumps. Then I'll delve into fluid chemistry and try to rationalize differences in stability, lubrication etc. Finally, I'll summarize applications and price. I know, I know - for the home experimentalist (or 'HE' as I non-chauvinistically call you) with a vacuum system, the last should be first.

INTRODUCTION

Mechanical Pump Fluids

The primary property we need is *low vapor pressure* (VP). In a mechanical pump, the VP value that matters is measured at the pump's operating temperature (typically 80° C). A fluid with a high vapor pressure at this temperature is not something you want near your vacuum system. So what's high and what's low, you ask? There are three ways to judge: (1) *Technically* - if the literature suggests the oil's vapor pressure is less than 10^{-6} Torr at room temperature and less than 10^{-3} Torr at 100° C, it'll probably be a dandy mechanical pump oil. (2) *Biologically* - if the oil has a strong pong at room temperature, watch out! Anything that smells has to have a higher VP than you want. (3) *Worth-ily* - VP and price are related by something like an inverse cube law. What's it *worth* to you to get a decade reduction?

Second, you need a fluid that is *unreactive* in your application. If it isn't, then it will change into something you didn't bargain for. For example, someone sent us a pump for repair. It had been drained, or so we thought, since nothing came out of the drain plug. However, when we removed the case, the oil sat there in a jellified blob, quivering. The lyrics of an old jazz standard came to mind, "It must be jelly 'cos jam don' shake like that." Needless to say, the pump had seized.

Third, and very important if you want to take advantage of the low vapor pressure and unreactiveness, the fluid must be an excellent *lubricant*, whatever that means. I feel nonplused about lubrication. I know oh-so-well the effects of its absence, but have no idea how lubrication really works. Also, I've heard grown men argue vehemently about the relevance of four ball wear tests to an operating mechanical pump. Do we have any real oil chemists out there?

Fourth, you must choose the right *viscosity*. A two-stage rotary vane pump has, typically, close tolerances between 'rubbing' surfaces and is driven by a low power motor. Put a high viscosity fluid in a vane pump, switch it on, and listen to that puppy whimper before the motor starts smoking. It needs an oil with a viscosity of 40 - 60 centistokes (cs) at 40° C and 7 to 10 cs at 100 °C. A rotary piston has larger clearances and needs a higher viscosity fluid to make the necessary gas seal. Something like 80 - 200 cs at 40° C and 10 - 20 cs at 100° C.

Finally, look for three advantageous but subsidiary characteristics: good *heat transfer* to help cool the pump; a *hydrophobic* nature to shed water vapor; and *non-foaming* so you don't have to always be cleaning an oil slick from your basement floor. In truth, you're usually stuck with what you get for these qualities while you try to satisfy VP, unreactiveness, lubricity and viscosity considerations.

Diffusion Pump Fluids

The first consideration for diffusion pump fluids is also *vapor pressure* but here we need the room temperature value. In this pump, fluid boils, typically around 220 - 240° C and 1 - 2 Torr, and the vapor curtain is directed (downward) from a number of jets towards a water-cooled wall. At the wall, you want the vapor to condense instantly to a fluid with a very low VP. Why? Because evaporation from the wall isn't directed and oil vapor from that source will backstream into the chamber as easily as it will forestream (I just invented that word) to the liquid sump. The lower the VP at wall temperature, the lower the backstreaming into your chamber and the better the pump oil.

As a second property, the fluid should have a *high molecular weight*. Diffusion pumps work by momentum transfer. Gas molecules from the chamber drift (actually, most of them zing around at 900 mph) into the vapor curtain. The oil vapor molecules (also moving supersonically) hit the gas molecules and give them forward/downward momentum. Why must the oil molecule be heavy? Well, you want each collision to barely affect its (the oil molecule's) trajectory to the cold wall, but you want to knock the socks off the gas molecule. Bigger is better, it's the American way. Quantitatively? How about heavier than 300 daltons?

Third, the diffusion pump oil must be *stable* at its boiling point and generally *unreactive*. Specifically, you look for oxidation resistance since the 'active' gas most often seen in HE vacuum systems is the oxygen in air. In contrast to my early pump oil story, one time I drained silicone oil from the diffusion pump of a mass spectrometer into which I had dumped the well-known oxidizing agent UF_6 for many months. To my surprise, despite a black sludge in the bottom of the pump, the oil was in fine shape. Perhaps the diffusion pump had acted as a filter to remove the UF_6 and, thereby, saved the rotary pump's oil from instant destruction.

CHEMISTRY AND PUMP FLUIDS

Mercury

When I first started in the vacuum business (shortly after von Guericke entertained the populace of Magdeburg), mercury-in-glass diffusion pumps were all the rage. That may have something to do with my present-day memory.... now where did I put my computer? Given what we know about heavy metal poisoning nowadays, nobody in his right mind would use a mercury pump in her basement (note to editor - the mixed pronouns are deliberate, reflecting both a-chauvinism and how-life-really-is). It's probably not smart to have mercury-in-glass thermometers in your medicine cabinet in case they break. And as for amalgam in your teeth... well!

Hydrocarbons

As pump fluids, hydrocarbons are ubiquitous. The name is a complete description of their basic chemistry - they can only contain carbon and hydrogen. You can't slip an oxygen into the molecule and continue to call it a hydrocarbon. Surprisingly, some folks, even vacuum fluid suppliers, just don't get that fact.

If you've had any organic chemistry you know that carbon is the one element that joins to itself in strings. A hydrocarbon is a long single or branched string of carbon atoms, the 'backbone', with lots of hydrogen atoms surrounding it. Each carbon has four valencies (`hands to hold onto other atoms' hands), while each hydrogen has one. So, any carbon atom, not at the molecule's end, is joined necessarily to its two neighboring carbon atoms by two of its valencies. This leaves two valencies that must be satisfied with hydrogens. One way of writing this is:

$$CH_2$$

Or, putting a small chain together:

$$-CH_2-CH_2-CH_2-CH_2-$$

What happens at the ends? No big deal, just add an extra hydrogen to make:

$$CH_3-CH_2-CH_2-CH_2-CH_2-CH_3$$

This is called a straight chain hydrocarbon (or *n-alkane* officially).

So, what's a branched chain? If a molecule divides somewhere along its string (so one string becomes two), it means a backbone carbon is joined to three other carbons rather than two. One way to depict this is:

$$CH_3-CH_2-CH(CH_2-CH_2-CH_3)-CH_2-CH_3$$

The bit in brackets ($CH_2-CH_2-CH_3$) is joined to the -CH< forming a branched chain. Of course, you could have >C< where one carbon is joined to four others or have double and triple branching etc. from the backbone. A good example of real live branching is the stuff used as the standard for determining the octane number of the gasoline in your car. It's *iso-octane* with the formula:

$$CH_3-C(CH_3)_2-C(CH_3)_2-CH_3$$

The octa comes from 8 carbons. Go ahead, count 'em.

That example illustrates another property. If you do the arithmetic for a straight chain and a branched chain with the same number of carbon atoms, you find the hydrogen count is the same for both. Two molecules with exactly the same formulæ? (Notice that use of the Latin plural? Oh, the joy of a classical education.) Yup... they're known as *isomers*. What's the difference? In a hand-waving way, branched chain molecules boil at slightly lower temperatures than straight chain isomers. The more branching, the lower the boiling point. Branching adds a little extra lubricity and, as I recall, the reactivity at those 'special' carbons is a little less. But I have to check that in a real chemistry book.

Meanwhile, back at the chain gang, two neighboring carbons can also join with two valencies to form a double bond >C=C<. The resulting *alkene* brings a whole new meaning to the word reactivity. The double bond is susceptible to 'addition' reactions. Say you're determined to find some new process for brightening the vacuum wash and you've put muriatic acid into Clorox (I not II). You'd have a chamber filled with chlorine. If your pump oil is unsaturated, the Cl_2 adds to the double bond to give >CCl-CCl<. Is that a good thing? Usually not. After all, you chose a hydrocarbon fluid for some reason and now it's something else.

OK, we all agree that the alkenes and their damnable double bonds are not what we want. So how can we tell if this fluid is more unsaturated than that fluid without expensive testing? Turns out, the hemi-demi-semi- quantitative answer is - look at it! The more golden brown the color, the more unsaturated it's likely to be. A 'white' oil is a good pump fluid because it's pretty much saturated. If you want a fully saturated oil with minimum reactivity for a hydrocarbon, try a synthetic made by polymerizing ethylene (CH_2=CH_2).

To differentiate between these chain hydrocarbons from what comes next, we lump alkanes, alkenes (and alkynes - no, don't ask!) together and call them *aliphatic* compounds.

Before you sink deep into your couch with a rosy glow, thinking you understand reactivity and unsaturation, let's look at one structure that's simultaneously unsaturated and stable. It has the formula C_6H_6. Draw a hexagon, write `C' at every corner and put a 'radial' line from every C to just one H. Count the valencies that are left over and you find it only works if you draw a double bond between alternate pairs of C atoms.

The structure was dreamed up by some German geezer named Kekule on the top of a London double-decker bus. It's called the benzene ring and molecules containing this ring are called, in general, *aromatic* compounds (in contrast to aliphatic). Specific compounds with these rings are named, for reasons known only to organic chemistry professors, *phenyl* this or *phenyl* that.

Although benzene and phenyl compounds are nothing like as reactive as the alkenes, they are removed from hydrocarbon fluids. I think the reason is, the sulfur in crude oil is associated with the aromatics and since the smell of organic sulfur compounds will stop a charging grizzly bear at 20 paces, they must be removed. In doing so the phenyl compounds go right along with the cleanup.

So, the sequence of making a hydrocarbon vacuum fluid is: crack crude oil; remove sulfur and aromatic compounds; remove organic acids; hydrogenate to remove some unsaturation; and vacuum distill two or three times to reduce 'light ends' and the heavy sludge. After all this, a hydrocarbon pump fluid is still a merry mixture of umpteen different long and short chain length, straight and branched aliphatic compounds and isomers covering a broad range of molecular weights from, say, 200 daltons to 500 daltons (about 15 carbon atoms to about 35). In general, the narrower the molecular weight range (that is, the more distillation steps) the better the fluid in vacuum operation.

Esters

An ester is formed by reacting an organic acid with an alcohol. It's tough to show that in word processing but let's try. An *organic acid* has an aliphatic (or aromatic) chunk attached to the group -COOH. If you read that as it looks, -C-O-O-H, the carbon has the wrong valency. Try it like this:

-C(=O)-O-H

with one oxygen bonding to the carbon with both valencies. The other oxygen has one valency to carbon and one hydrogen.

An *alcohol* is an aliphatic (or aromatic) chunk attached to a >C-O-H group. The carbon's two unlabeled valencies can attach to other carbon atoms (secondary alcohol) or one to carbon and one to hydrogen (primary alcohol). Just to ease what follows, let me rewrite that alcohol as:

H-O-C<

Now, if we react acid and alcohol together:

-COOH + HOC<

out pops H_2O and we get -COOC<.

Low molecular weight esters have 'fruity' smells. Indeed, the bouquet of fruit is due, in part, to esters in the fragrance. And, just as you wouldn't put peaches or strawberries in your vacuum system, low molecular weight esters are not for us. But high molecular weight esters are a horse of a different hue. They are non-smelly and make good diffusion pump fluids since they resist oxidation. The usual explanation for this includes pontifications about the oxygens in the structure acting as electron attractors and decreasing the reactivity of the C-H bonds elsewhere in the molecule. But the truth is, they are good because they're cheap - excuse me - low cost.

One ester that's been around for yonks is dinonyl phthalate. And for those of you not yawning yourselves to sleep - remember the benzene ring? Well, take two adjacent carbons in the ring, toss out the hydrogens and replace them with -COOH groups. That's phthalic acid (pronounced phthalic acid). Now add nonyl alcohol (*non*- in this case meaning 9

carbon atoms - 8 plus the >C-OH group), stir lightly, and you get the ester dinonyl phthalate. With a molecular weight of 396 daltons, it's a pretty good diffusion pump fluid and, yes, it's cheap because it's used commercially by the megaton.

There's a story about this from way back in the early gas chromatograph-mass spectrometer days. Some guy at the NIH in Washington found an alarming increase in the presence of an single, unknown GC-MS peak in the urine analysis of many patients screened by the institute. He, and others, suspected a new street drug but, after a big brouhaha, found it was - dinonyl phthalate. What in the world...?

So, the guy dug into the history of these folk and found out most worked in the burgeoning electronics/computer industries, where they had access to *and chewed* the insulated covering when they stripped a connecting wire. Pure polyvinyl chloride (PVC) is hard and brittle. Add some dinonyl phthalate as a plasticizer and PVC becomes soft and floppy - just right for flexible wire insulation.

Apparently, ingesting the stuff had few side effects which, if you think about it, are obvious. If it passes unchanged into one's urine, then it is neither broken down by the fiendishly strong hydrochloric acid in one's stomach nor metabolized by all those enzymes. And aren't they just the characteristics you want in a good pump oil?

Another ester in common use as a vacuum fluid is di-ethylhexyl sebecate (pronounced by we cognoscenti as *sebber-sate* - not *sebber-cate*). Sebacic acid is an aliphatic di-acid and is, therefore, chemically quite different from dinonyl whatitsface. It's the ester function that makes its properties similar.

Much earlier I carefully wrote 'organic acids' because, strictly, that's what's needed to make esters. However, inorganic acids react with alcohols giving things that might be mistaken for esters. For example, phosphoric acid (H_3PO_4) reacts with triphenyl butyl alcohol (don't worry, I'm not going to touch that one) to give a phosphate 'ester' with a molecular weight of over 400 daltons. It's a pretty good pump oil.

Ethers

The stuff commonly known as 'ether' is really diethyl ether:

$$CH_3\text{-}CH_2\text{-}O\text{-}CH_2\text{-}CH_3$$

It was used in bygone years as an anesthetic. Trouble is, its vapor is highly explosive, it can have bad medical side effects, it's difficult to administer in controlled doses, and, if you survive all that, you have stinky breath for days after being put to sleep.

Ethers are like esters in that both have the >C-O-C< group somewhere in them. Again we can invoke resistance to oxidation because the electrons are pulled from C-H bonds to the oxygen. If you put the ether grouping between successive benzene rings, you can get a dynamite (excuse that pun!) diffusion pump oil - polyphenyl ether. The unreactiveness of the phenyl bit is enhanced in spades by the ether link. Yep, this is a goody! But the price? I have a friend who once had all the mortar on his three story, brick house re-pointed. He claimed, "I now know that re-pointing means you move the decimal point in your bank balance four places to the left." Buying polyphenyl ether has much the same effect.

Silicones

And now for something completely different. Making vacuum pump fluids is small beer to Dow Chemical. Think of all the other silicone products: caulking, rubber, polishes, car waxes, etc and you get the message. Silicone fluids (not *silicon* fluids, please) for vacuum pumps (and all those other products) are based on the backbone ->Si-O-Si-O-Si<-. Like the carbon in hydrocarbons, the silicon atoms (in these compounds) also have four valencies. The other two (or three at end groups) are attached to hydrocarbon chunks, often benzene rings. The stability of this backbone is probably linked to the stability of SiO_2, otherwise called sand.

Dow's DC 705 is really pentaphenyl trimethyltrisiloxane (Oh, come on. It's not that unpronounceable!). Counting the spare valencies on the backbone >Si-O-Si-O-Si< (including the two on the middle silicon that I can't draw), we have 8 total. To those valencies we attach 5 (penta) phenyl groups and 3 (tri) methyl (CH_3-) groups. You can visualize that the phenyls and methyls could occupy a variety of positions around that backbone. Three phenyls might be at one end and the other 2 on the middle silicon. Do we care about the arrangement? Not much. By definition all isomers have the same high molecular weight, similar chemical stabilities and extremely low VPs.

To define all of Dow's silicone fluids you should know that DC 704 is tetraphenyl tetramethylsiloxane (you work it out) and DC 702 is a mixture of phenylmethyl and dimethyl cyclosiloxane. And I'd love to explain that except what in

the world is cyclosiloxane? Some sort of 6-membered ring of alternating Si and O atoms maybe? Just accept that 702 is less expensive and therefore has a higher VP (about four orders higher) than 705.

To complete the story, you should know that someone, in their infinite chemical wisdom or unconscionable greed, has invented perfluorinated silicone fluids. I ain't never seen any, nor got a set of specs, but it sure sounds like it should resist even the force of Obee One Kanobee.

Halocarbon

If you take an aliphatic hydrocarbon, magically substitute chlorine and fluorine for all the hydrogens, you have a fluid that will withstand considerable chemical abuse. However, it seems to me that if the material undergoes chain shortening reactions in a pump, what ends up in the atmosphere are light CFCs with their attendant ozone depleting properties. I chose, therefore, not to continue the discussion of these products. If someone out there knows otherwise, please tell me.

Perfluoropolyethers (PFPE)

As the name states, PFPE fluids are strictly ethers. So why didn't I write about them above? Mostly because their properties and applications depend as much on the perfluoro bit as the ether linkages. The PFPEs are *inert* and there never was a truer name. The vapor has no flash point because it is not a fuel even when pumping 100% oxygen. Trying to convert the fluid into something else with the worst of 'nasties', like fluorine or Lewis acids, does little damage. The slight reaction that does occur simply breaks the backbone somewhere to give a low molecular weight piece that quickly boils off.

So, what is this wonderful stuff? Here, I must introduce you to chemistry's famous 'n' and 'm'. Both represent numbers, like 6 and 7, or 3 and 12. That is, they are a splendidly scientific way of saying: this number doesn't equal that number, but I don't have the vaguest idea what either number is.

Like ordinary aliphatic ethers, the PFPE backbone contains:

$$>C\text{-}O\text{-}C(C)\text{-}C<$$

but since it's a "polyether", it goes:

$$>C\text{-}(O\text{-}C(C)\text{-}C)\text{-}(O\text{-}C)\text{-}O\text{-}C<$$

As you can guess, all the unmentioned valencies on the carbons are taken up with fluorine atoms and really the molecule looks more like:

$$CF_3\text{-}(O\text{-}CF(CF_3)\text{-}CF_2)_m\text{-}(O\text{-}CF_2)_n\text{-}O\text{-}CF_3$$

Now you see why I keep close company with m and n.

There are two well known inert fluids tradenamed Fomblin (from Ausimont) and Krytox (from DuPont). They are not exactly identical but the variations are beyond this discussion. It is said that the invention of Fomblin was a crucial part of the semiconductor revolution. Without it, all the wildly exciting semiconductor processes such as: plasma etching with CF_4 and O_2, ashing photoresists with O_2 plasma, pumping BF_3, $AlCl_3$, and other Lewis acids, would (like a good WWW site) have included sound, motion and lots of interaction, as pump oils exploded or ceased to lubricate exactly 20 minutes after the pump was put into service.

One of the good things about PFPE fluids is that even the light ends that might be broken off the backbone do not pose an environmental threat. It seems the perfluoro fragments have no potential for destroying the ozone layer.

APPLICATIONS

Hydrocarbons

Hydrocarbons have excellent lubricity for mechanical pumps. Some have high enough molecular weights and low enough VPs to work in diffusion pumps. Normal yellow-brown oils are not very good at resisting oxidation but formulators add antioxidants to improve their resistance. Resistance is really a question of degree - does it last 3 days or 3 months in the application.

Hydrocarbons are the lowest priced fluids available and are pressed into all sorts of services well beyond the 'general' applications where they excel. However, ask them to do 'man's work' pumping a plasma system or a semiconductor process and they will fall apart quicker than a plastic Tonka truck knock-off from Taiwan.

If you really can't afford commercial hydrocarbon pump oil, find yourself a second job or a source of straight-cut motor oil - that is, get the stuff before the rheologists start diddling with thixotropic agents to make 10W-40 constant viscosity motor oil. If you are truly inventive, amaze your friends and the local constabulary by making a crude still. Close the system and stick a vacuum pump on it. Then you can distill yourself some real vacuum-grade pump oil. We do it by the ton every day. In truth, we cheat since we use very swish, rotating molecular stills, which are a fair step up from the typical moonshine equipment.

Hydrocarbon fluids come in a variety of viscosities to suit all types of mechanical pumps. Another anecdote is appropriate here. I was visiting a company that casts turbine blades under vacuum and someone questioned a result they had found. Using our W77 oil, a water-cooled rotary piston pump gave perfectly good 20 micron forepressure all morning but suddenly jumped to 300 microns in the afternoon. They'd investigated and found that varying the water flow would change the pump temperature by 5° F and this caused the (reversible) pressure change. Cool pump equals great vacuum - warm pump equals foreline floppo. Together we concluded that W77's viscosity at the higher temperature was too low to seal the pump piston, allowing the compressed gas to leak backwards. They changed to a higher viscosity oil and, bingo!

Silicones

This stuff may lubricate the wheels of your son's wooden race car at the Soap Box Derby but use it in your one-and-only mechanical pump at your peril. Silicones are, of course, wonderful diffusion pump fluids. They resist the effects of many different oxidizing agents and are frequently used in rough-as-guts systems, such as vacuum smelters, since the vapor's not much of a fuel under explosive conditions.

Problems? Yeah, they're pricy! If you are among the willfully extravagant class of HEs that can afford DC 705, why are you reading this article? If you need the chemical resistance of 702 and can afford it, then use it. However, unless you've got a well-maintained LN_2 trap in the pumping line, expect to clean oil from the chamber walls at regular intervals.

Perfluoropolyethers

Different viscosities of PFPE fluids are used in diffusion and mechanical pumps. They have quite reasonable lubricity and, better yet, mechanical pump fluids are offered with a 'rust preventing' additive that actually protects the pump's metal parts from the ravages of corrosive goodies (to a certain extent). The average HE isn't likely to mess with it, since it costs about $170 a kilo. With a density of almost 2 gm/cc, that's all of 500 cc! However, if you are deep into making plasmas, even plasmas in air, PFPEs may save you some pumping heartaches. And if you ever get it so loaded with gunk that the pump seizes, you can have the fluid reclaimed.

Polyethers

The classic polyether is Monsanto's Santovac 5 with five phenyl rings in a row joined by oxygen atoms to form ether groupings. Its VP at room temperature is 10^{-10} Torr: a truly impressive value. Its molecular weight is 449 daltons which, for a diffusion pump fluid, puts it up there in the sumo wrestler class. But, man, is it ever viscous! Put this in your 1 cfm mechanical pump and you'd need a 50 HP motor to turn it over.

Esters

Do not confuse this with the polyester used in your 1970s lounge suit, you sartorially elegant devil you. The di-esters of sebacic and phthalic acids plus the triphenylbutyl phosphate 'esters' are typically not expensive and will withstand a fair old bashing from oxygen. One large company in the pure gas cylinder business uses the phosphate fluid as a mechanical pump fluid for pumping oxygen. They claim it is unreactive, is good at greasing the wheels and has a high flash point. Another company, into vacuum metal casting, uses the same fluid in their diffusion pumps, recognizing that every once in a while they must de-coke their pump jets since the phosphates tend to gum-up at that point.

Most organic esters are used as diffusion pump fluids. The lubrication characteristics of dinonyl phthalate wouldn't convince me to put it in my Welch belt-driven puffing billy. Some of the more complex esters are, however, good mechanical pump oils. One structure, in which the original alcohol had four -OH groups and at least one carbon joined to nothing but carbons has, when esterified with the right organic acid, *steric hindrance* up to its armpits. That is, it's so 'stiff' at the molecular level that any site that might be susceptible to reaction is hidden, preventing the approach of the reactant molecule as it tries to form the all-dictating intermediate stage. I'll confess I'm being coy in my description since we have a proprietary fluid with this structure that's used as an inert fluid substitute in some very nasty processes.

RANDOM CONCLUSIONS

1. If you are pumping oxygen

Be a real chicken! It's your life and house you're putting on the line if the hydrocarbon fluid in your mechanical pump goes whoomp. Remember, even though your chamber is at 10^{-3} Torr of oxygen, the pressure **must** exceed atmospheric when the gas gets blown out the mechanical pump's exhaust valve. Hot hydrocarbon vapor and atmospheric pressure oxygen makes me dive for my leather underwear and full body armor. At least use the phosphate ester for the mechanical pump. It will cost you about $65.00 per gallon.

At the high vacuum end, try a silicone at $70.00 a pint or dinonyl phthalate at $45.00 a pint. You don't need much fluid for most diffusion pumps.

2. If you are pumping plasmas

First of all, please vent the pump exhaust to the downstream side of the house. For mechanical pump fluid you should use an inert or near-inert fluid. But, faced with $1400 per gallon (inert), $220 per gallon (near-inert), or $14.00 per gallon (TKO18 hydrocarbon), you'd need a lot of convincing. Right?

Will the inert really last 100 times longer? Sure it will. You can't destroy the stuff. But that's not the whole picture. While the oil's doing fine, you may have etched the pump metal to a shadow of its former self. A suggestion: use hydrocarbon but change it with mind-numbing regularity. And change it just after you've shut down the system. Don't let the nasties your plasma has made stay in the pump while you go on vacation.

3. If you are pumping corrosives

What's a corrosive? Well, hydrous acid vapor (HCl, H_2SO_4, HNO_3), organic acid vapors (formic: 'cos you're experimenting with ants, acetic: 'cos your looking at Ranch dressing), Lewis acids (BF_3, $AlCl_3$), halogens (F_2, Cl_2, Br_2, I_2) and many others will chew into bearings, gaskets and rotors with a vengeance and form a wondrous gruel in the pump oil while doing it. In general, follow the advice given for plasmas - don't let the stuff stay in the pump. If you can afford it, consider an inert or near-inert.

We know of one semiconductor processor who decided to run hydrocarbon fluids in all pumps on one particularly virulent process. They changed oil every day in every pump by simply draining the old (with the pump still running), wait for a gurgling sound, and then topping up with new oil. They still suffered an average of two pump seizures a day. Well, that's one way. As an HE, you face other problems when you've pumped corrosives - what to do with the contaminated fluid? How do you avoid asphyxiation while changing it?

4. If you are pumping regular old air

You're my kind of guy or gal. Why use anything other than hydrocarbon in the mechanical pump and what you fancy in the diffusion pump? Of course, I recognize that pumping regular old air from atmospheric pressure to ten to the minus, oh dear, means you're not having 'fun' and doing those adventurous things that HEs are wont to do. But remember, I just advise on the pump oil. I'm not the entertainment director for your basement.

5. Offer

If Steve agrees, and if he doesn't you won't be reading this, I don't mind answering any specific questions in an open forum. Write your question about pump fluids or their applications to Steve. He'll forward them to me and will print both questions and my (handwaving) answers.

Backstreaming from Oil-Sealed Mechanical Pumps

Phil Danielson

At the time this article appeared (Volume 4, Number 3) Phil Danielson was the president of Danielson Associates, Inc. and this article is from Danielson's newsletter, The Vacuum Chronicles, *Volume 1, Number 4 (1989) and has been reprinted with permission. Phil has since sold Danielson Associates and has started a new company, The Vacuum Lab, where he does consulting and new product development. His writing now regularly appears in a new trade magazine, Vacuum & Thinfilm.*

INTRODUCTION

All oil-sealed mechanical pumps backstream oil to some extent. As the oil becomes hot from the operation of the pump and the pressure falls into the molecular flow regime hydrocarbon vapors will rise from the pump. Undistilled oils will contain light fractions which will vaporize quite easily due to their high vapor pressure. A slight fishy smell at the pump intake is a good indicator of the presence of light fractions. Vacuum distilled hydrocarbon oils will provide a much lower backstreaming of hydrocarbon vapors since the lighter fractions have been removed and higher molecular weight oils with lower vapor pressures result. Backstreaming will still occur, albeit at a much lower rate than is found with undistilled or partially distilled oils.

Oil need not transfer to the chamber only through vaporization. Most oil-sealed mechanical pumps will throw small droplets of liquid oil that have been mechanically generated out through the pump's intake. These droplets will quickly wet the surfaces of the pumping line. Once the oil has wet the surfaces, it becomes highly mobile and will spread evenly over any surface it can reach.. This phenomenon is usually referred to as *surface creep*.

Surface creep will also occur right out of the pump itself with the oil reservoir within the pump acting as the source. In many cases, this constant surface flow of liquid oil from the mechanical pump into the rest of the pumping system is the greatest single source of hydrocarbons that can enter the vacuum chamber [1].

FORELINE TRAPS

Foreline or roughing traps have been used for a number of years in an attempt to stop or reduce the oil backstreaming from oil-sealed mechanical pumps. Although they have had great commercial success in terms of the number of units used, they have a number of problems that need to be weighed carefully if they are to be considered as a method of stopping the pump oil from entering the chamber. Three types of foreline traps are commercially available:

1. Condensation traps, cryogenic
2. Adsorption traps, absorbent trapping medium
3. Adsorption traps, active surface trapping medium

All have a potential problem that needs constant consideration. They have room temperature walls which will allow liquid oil to coat the inner surfaces and once the oil has passed into the system side of the trap, it becomes a vapor source that can no longer be prevented from entering the system. The amount of time required for the surface oil to work its way through the trap will vary with the application. The best way of determining when oil is passing through is to occasionally remove the trap from the pumping line and then placing a drop of water inside the tubulation on the system side to see whether it beads or spreads. Once oil is detected on the system side, it's time to solvent clean the trap or to bake out the entire trap, walls and all. The time to fully penetrate through the trap will be extended if the mechanical pump is shut off and the trap (fore or roughing line) is air released when the pump is not being used.

Condensation traps (see Figure 1.4) operate by the simple technique of condensing the oil vapors on a cold surface. This is usually done with a bucket or coil filled with cryogen mounted inside a housing. Consider that a trap is a dual flow device in that gas from the chamber or system passes in one direction and backstreaming oil is attempting to pass in the reverse direction. The gas being pumped from the chamber is going to provide water vapor [2] to the condensing surface while oil vapors are being condensed as well. In time, the trapping surface will load up with condensed gases and will require that it be regenerated by warming up. Then again, the trap might warm up accidentally (catastrophically?) by running out of cryogen. In either case, the trap will need to be removed from the pumping line. If the condensed oil is allowed to re-vaporize, it will be easily possible for the oil vapor to enter the chamber.

ABSORBENT TRAPS

Absorbent traps (see Figure 1.5) depend upon an internally mounted array of absorbent material to trap oil vapor, and they do that efficiently. One of the most common materials is 13-X molecular sieve. The porosity of the sieve will absorb any oil vapor it contacts until it is saturated. As water vapor enters the trap, it will be preferentially absorbed over oil molecules. This means that as water enters, oil will leave. Since the trap is under vacuum, oil molecules will enter the chamber as easily as they will re-enter the pump from which they originated.

Although most of these traps are supplied with heaters to drive out the trapped oil, they cannot be baked in place under vacuum or the released oil will enter the chamber. They must be removed from the pumping line and baked out separately to avoid this problem. (Isolating the trap with a bakable valve upstream of the trap and baking the entire assembly will also work - *Ed.*) There is the additional problem that the molecular sieve will slowly break down into dust due to differential expansion/contraction with the thermal bakeout cycles required, and the dust will surely enter either the system or the pump. It is perhaps better to replace the sieve material upon becoming saturated with either water or oil.

ADSORBENT TRAPS

Adsorbent traps (see Figure 1.6) are usually crammed full of fine tendrils of copper wool. The copper surface is oleophilic and will adsorb large quantities of oil vapor. It will also quickly saturate with oil when exposed to liquid oil, requiring frequent cleaning with solvents. Remember that it must be totally dried of solvents before being re-used as an oil trap.

REGENERATION

Aside from removing the trap from the pumping line, there is little choice in achieving regeneration of its trapping ability without contaminating the system it was intended to protect. Traps can also be regenerated by processing under a condition of dry gas (usually nitrogen) flush from the chamber side while regeneration techniques are applied, the contamination of either water vapor or oil will be entrained in the gas flow and carried into the mechanical pump

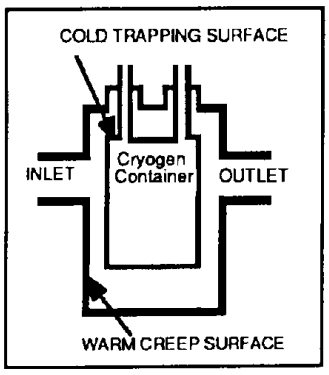

Figure 1.4 - Condensation Trap

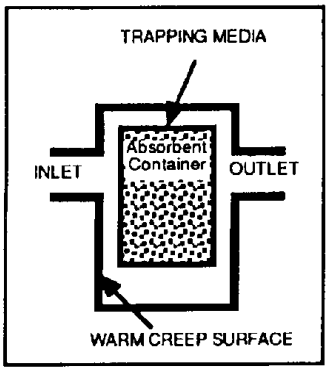

Figure 1.5 - Absorbent Trap

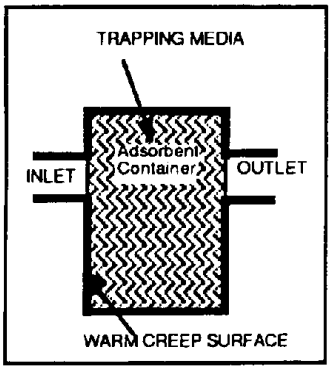

Figure 1.6 - Adsorbent Trap

instead of being allowed to enter the system. Pressures of the purge gas will vary according to the system, but 200 milliTorr to 1 Torr will provide entrainment level pressures in the viscous flow regime.

PUMPING SPEED LOSSES

Since the pumping speed of oil-sealed mechanical pumps drops dramatically with pressure reduction, it becomes necessary to consider the possibility of pumping speed losses through a trap at low pressures. The constriction to gas flow in any of the traps described will become more and more critical as the gas flow undergoes transition from viscous to molecular flow into the low micron pressure range. Thus, the constriction to flow is at its greatest as the pumping speed of the pump is at its lowest and where the ultimate pressure attainable by the pump is the most important. Additionally, the trap causes the greatest loss in pumping speed at pressures and conditions where backstreaming is at its greatest level.

CONCLUSION

Traps can stop or reduce backstreaming by careful use and even more careful regeneration techniques if the penalties imposed by the extreme care required, the possible results of a mistake, and the loss of pumping speed are considered worthwhile. However, using oil- free pumps bypasses these penalties.

REFERENCES

[1] *Roughing it for Better Processing*, R&D, Vol. 30, No. 6, June 1988, p. 87.
[2] *Water Vapor Need Not Drive You Mad*, R&D, Vol. 29, #11, November 1987, p. 93.

Complete Dummies Guide to the Operation of a Typical Diffusion Pumped High Vacuum System

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This material, reprinted with permission, is from Roy's web site at http://www.ee.ualberta.ca/~schmaus/vacf/index.html. Roy has a lot of interesting information on vacuum, as well as topics both related and unrelated.

This article is just for all those people who have never had anything to do with a vacuum system of any kind but need to use one. Diffusion pumped systems are still quite common, and used for all sorts of applications e.g. vacuum evaporators for making metal coatings. The diagram in Figure 1.7 is of a small bell jar system that we use for a variety of things here in the lab.

GETTING STARTED

The system shown here is typically used for vacuum evaporation, etc. This guide assumes that everything is off initially and that the bell jar is at atmospheric pressure to start with.

1. Preliminary

Be sure you have liquid nitrogen available before starting this type of system, and that you have some familiarity with proper LN_2 handling techniques. You should wear eye protection, a long-sleeved lab coat, and protective gloves. Frostbite is painful! Switch power on to the gauge controllers but don't try starting the ion gauge yet as you might destroy the tube if it has a tungsten filament.

2. Water Cooling

Turn on the water cooling lines to the diffusion pump and the water cooled baffle. Verify water flow. Before going too much further, I would like to add a little sermon on water cooling lines. There have been several floods in our lab due to plastic water lines coming off of equipment left running overnight. Soaking someone's precious collection of books or having water get into electrical conduit tends to create a lot more waves than the relatively small expense of taking some precautions. A water flow switch controlling a water inlet solenoid will prevent problems and insure your getting invited to the next bush party or whatever. All of our lab systems are plumbed in with soft copper tubing and are provided with flow interlocks. Since putting these in place several years ago, we haven't had any flooding problems.

3. Valves

All of the vacuum valves in the drawing should be closed to start with. The pump vent valve may be as shown, or in some cases it is part of the rotary vacuum pump in which case it will close automatically when the pump is running.

4. The Rotary Pump

Switch the power on to the rotary pump. The pump will be quite noisy and spew oil vapour out its exhaust when starting from atmospheric pressure. Don't be alarmed, that's normal operation and the noise and vapour should subside as the vacuum improves.

If the racket and oil vapour spewing persist more than a few minutes (or longer for larger systems) it's quite likely that you have a gross leak or major contamination and you will have to shut the pump down and investigate.

5. The Diffusion Pump

Open the foreline to the diffusion pump and wait until the pressure on thermocouple gauge 1 is below 100 millibars. Switch the diffusion pump heater on and wait for a minimum 1/2 hour. Larger pumps often take longer. In fact, just about all diffusion pumps take about an hour to really get going but may be usable in less time with less performance.

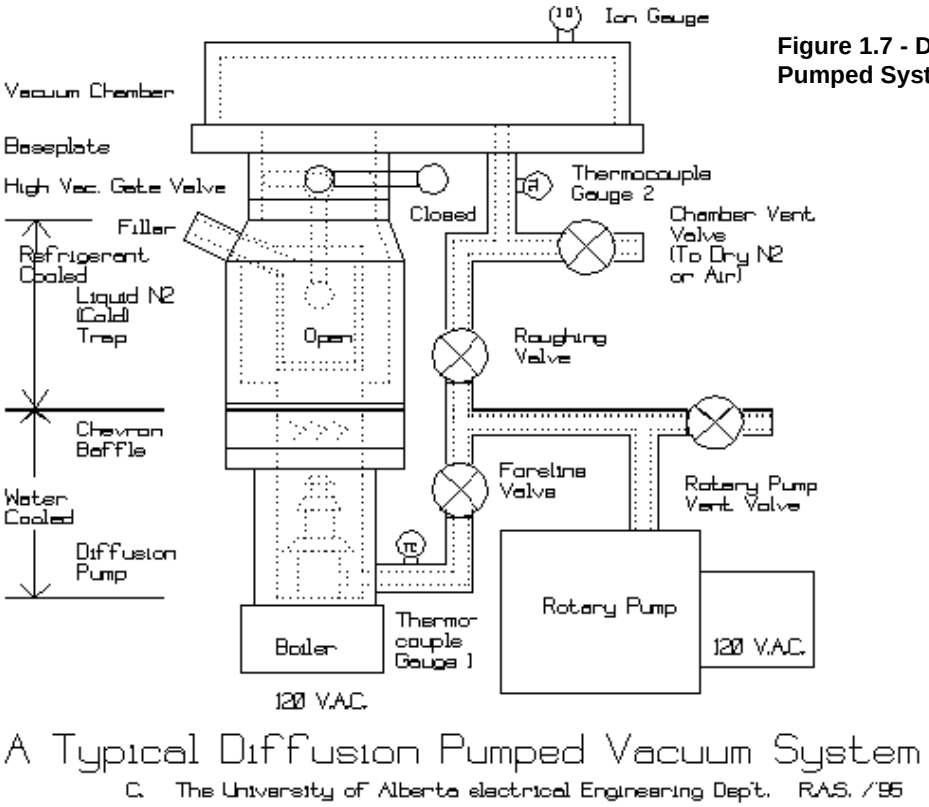


Figure 1.7 - Diffusion Pumped System

Fill the cold trap *slowly* with liquid nitrogen. The LN_2 will boil like mad initially as it hits the warm surfaces inside the cold trap. Be patient and just partially fill the trap initially to allow it to cool slowly. **The important thing to remember at this point is to never let a hot diffusion pump see high or atmospheric pressure, a situation that will crack many diffusion pump oils and create a horrible mess. If you break it you fix it and it may not be pretty!**

6. Rough Pumping the Vacuum Chamber

Close the foreline valve to the diffusion pump. Make sure the vent valve to the vacuum chamber is closed, then open the vacuum chamber roughing line.

The rotary pump will get noisy again, but the noise should subside in a few minutes and the pressure on thermocouple gauge 2 should begin to drop. Keep an eye also on the foreline pressure - if it rises above about 200 millibar you will have to pause by closing the chamber roughing valve, opening the foreline valve, and re-pumping the foreline. Close the foreline valve and re-open the chamber roughing valve when everything looks OK. *Skip the next paragraph unless there is a problem.*

If you can't get the chamber down to 200 mbar or less in about fifteen minutes there is most likely a leak or gross outgassing in the vacuum chamber. Close the roughing valve, re-open the foreline valve, and arrange to leak test or clean the vacuum chamber. DO NOT PASS GO, STOP HERE.

If everything has gone well, rough pump the vacuum chamber down to about 200 millibar, then close the roughing valve.

7. Pumping to High Vacuum

Open the foreline valve, then open the high vacuum (a.k.a. *baffle* or *gate*) valve. Wait for a few minutes, then start the ion gauge. If ion gauge pressure is above 10^{-5} millibar, it is good practice to switch the gauge off and just make spot checks until below 10^{-5} mbar to prevent oxidation of the hot gauge elements. Gauge outgassing should always be done at or below this pressure, with pauses to let the evolved gases be pumped away.

Congratulations, you're now in business! Now you just need patience - reaching ultimate pressure may take several hours. All that remains is to top up the liquid nitrogen trap, check it occasionally, and proceed with whatever it was that you wanted to do in vacuum.

SYSTEM OPERATION

1. To remove or replace items in the bell jar:

1. Switch the ion gauge off.
2. Close the high-vacuum valve.
3. Open the chamber vent valve.
4. Wait for the vacuum chamber to rise to atmospheric pressure.
5. Raise the bell jar. (Caution, don't try to force it open prematurely.
6. Remove/replace items in vacuum chamber.
7. Check the bell jar sealing surface and the baseplate for dirt, etc. and wipe if necessary.
8. Lower the belljar.
9. Evacuate the vacuum chamber, repeating the process.

2. Standby Operation

Running systems continuously to prevent the ingestion of water vapour and to prevent corrosion of oil-sealed rotary pumps is good vacuum practice and should be done if possible. Some means of keeping cold traps filled should be used on an untended system.

Backstreaming from an untrapped diffusion pump will contaminate your vacuum chamber in many cases, so it's best to close the high-vacuum valve, shut the pump heater off, and just keep the rough pump on the diffusion pump foreline. Alternatively, a good water cooled chevron baffle and a diffusion pump fluid selected for low backstreaming may allow continuous operation with no liquid nitrogen. We've had good success with polyphenyl ether but it is very expensive.

SHUTTING THE SYSTEM DOWN

1. Emergency Shut-down due to Power Failure

Close all the valves on the vacuum system and shut off heater power to the diffusion pump.

2. Complete Shut Down

1. Remove items from the vacuum chamber and re-evacuate the chamber to high vacuum.
2. Switch the ion gauge off.
3. Close the high-vacuum valve. Leave the bell jar under vacuum.
4. Switch the diffusion pump heater off.
5. Wait for the diffusion pump to cool down. If the diffusion pump is clean and uses a fluid that will withstand a short time at poor vacuum, you may get away with just closing the foreline valve at this point and letting the pump cool in a “sealed off" mode.
6. Close the foreline valve, leaving the diffusion pump under vacuum.
7. Switch the rotary pump off if you’re into energy conservation, and open the rotary pump vent valve if it’s not automatic.

CONCLUSION

Techniques shown here are generally used for the operation of diffusion pumped systems at this site. Your mileage may vary. Any questions or comments, please feel fee to send a note by post to the address at the beginning of the article or email to *schmaus@ee.ualberta.ca*

Manometers

Steve Hansen

This article was originally presented in Volume 5, Number 3.

INTRODUCTION

Pressure measurement devices are an important, if not critical, component of vacuum systems. Elsewhere we will look at a number of vacuum gauges including the simple discharge tube, the thermocouple gauge, the Pirani gauge and the thermistor gauge.

All of the above mentioned gauges measure pressure by indirect means. In the case of the discharge tube, the degree of vacuum is roughly indicated by the appearance of the glow discharge contained within the tube. The other gauges give an indication based upon the rate at which heat is removed from a hot wire. Since, in the range of a few Torr down to a milliTorr, the thermal conductivity of a gas varies predictably with pressure, it is possible to relate heat transmission to pressure.

The other common type of indirect gauge is the high vacuum ion gauge which indicates pressure based on the rate at which ions are produced by incident electrons.

It is important to note that none of these indirect techniques measures true pressure. Pressure is defined as the force that the gas exerts on a surface and the units are force per unit area. Depending upon which system of units you are dealing with, this can be pounds per square inch, dynes per square centimeter, newtons per square meter, and so forth.

In order to translate the indication of an indirect gauge to real pressure it is necessary to compare that gauge to a device that gives a true pressure indication that is ultimately based on fundamental (directly measurable) units such as length, mass, etc. These devices are called primary standards and the National Institute of Standards and Technology (NIST) develops and maintains standards and procedures for doing this.

Another characteristic of indirect gauges is that their readings are gas dependent. In the case of thermal conductivity gauges, different gases have differing thermal conductivities. Argon, for example, is a much worse thermal conductor than is air. Helium is a much better conductor. Therefore, if such a gauge is to be used with a gas other than

the one that it was calibrated for (by comparison through a chain that leads to a primary standard), a new calibration must be performed for that gas. Mixtures of gases make the situation even more difficult, if not impossible.

In this article we will look at gauges that measure pressure by direct means, i.e. by measuring the force that the gas applies to a surface. We will start by looking at the oldest device, the barometer, and then proceed to other forms of liquid column manometer and will examine some applications of liquid manometers. The article will conclude with a look at the capacitance diaphragm gauge where the displacement of metal diaphragm is measured by electrical means.

THE BAROMETER

The oldest liquid column gauge is the barometer. This device was perfected by Torricelli in the mid 17th century. The device consists of a glass tube, sealed at one end, about a meter long. The tube is first filled with mercury. When the tube is inverted with the open end placed in a container of mercury, the column falls to some level, roughly 30 inches above the surface of the mercury in the container.

The fall of the mercury creates a vacuum (called a Torricellian vacuum) in the space between the top of the liquid column and the upper end of the tube. This acts as a reasonable representation of a zero pressure reference. (Not quite zero because there is some water and mercury vapor in the volume, plus a bit of left over air.) At the other end of the tube, the air in the atmosphere is pushing with some force on the surface of the mercury in the cup. This is about 14.7 psi at sea level on a typical day. The net result is that the column height indicates the pressure differential between the prevailing atmospheric pressure and the zero pressure reference. On a typical day at sea level, this is a height of 760 mm. (Descartes established this as the standard atmospheric pressure, a value which has pretty well stuck.)

The column height is not true pressure. However, if one knows the density of mercury, the "real" pressure in force per unit area units can be determined. Of course, other liquids can be used. Well before Torricelli it was noted that water in a tube could not be "sucked" to a height exceeding about 33 feet. Once the principle was understood, one could allow the whole town to see the barometer reading by erecting a water barometer on the side of a tall building.

The use of mercury made for a compact instrument and the mm Hg unit has held for vacuum measurements: it's still more common to express vacuum in terms of mm Hg or microns Hg or the equivalent units of Torr or milliTorr than the "real" pressure units of bar (dynes per sq. cm) or Pascal (Newtons per sq. meter).

If one is interested in measuring pressures within some sort of vacuum chamber, then an arrangement as shown in Figure 1.8 can be employed. Indeed such devices were employed in the early days of vacuum experimentation. This sufficed quite well for the primitive pumps of the 17th and 18th centuries. These pumps were only good for pressures in the few Torr range. However, when Sprengel and Toeppler introduced their high vacuum mercury pumps, the simple barometer fell short. (No pun intended.) It simply wasn't possible to resolve pressures (i.e. column heights) of less than about a millimeter.

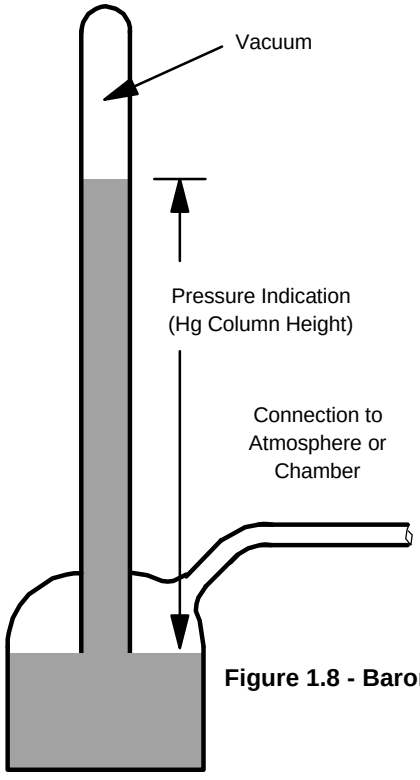


Figure 1.8 - Barometer

THE MCLEOD GAUGE

In 1874, H. McLeod got around this resolution problem with a clever application of Boyle's law. For those who slept through high school physics, Boyle stated that the volume, *V* of a gas at a constant temperature varies inversely as its pressure, *P*. Stated as an equation:

$$PV = \text{Constant}$$

or

$$P_1V_1 = P_2V_2$$

How this works is shown in Figure 1.9. Here we have a 100 cc syringe connected to a dial-type gauge. Let's say that we have very accurately measured the volume of the gauge and its connecting fittings to be 2 cc. Thus the total volume is 102 cc. Within this volume is air at some low pressure - low enough such that the gauge barely registers above zero.

Now suppose that the plunger of the syringe is pushed to its limit. The new volume of the system is now 2 cc. Looking at the gauge, the dial reads 75 Torr. Since we know the initial and final volumes as well as the final pressure, we can solve for the initial pressure. This is 150/102 or a tad over 1.5 Torr.

The measurement is real pressure as the mechanical dial gauge responds to force/area. Therefore, the measurement is not dependent upon the type of gas.

This simplified example also points out a couple of limitations of the McLeod gauge. First, it is an intermittent reading gauge. You cannot get a continuous reading. Second, since the gas is compressed in order to get the amplifying effect (in an actual McLeod gauge, the ratio might be as high as 100,000:1), if the gas is a mixture containing a condensable component (such as water vapor), that condensable will liquefy and will not be part of the measurement. Because of this, a lower pressure will be indicated.

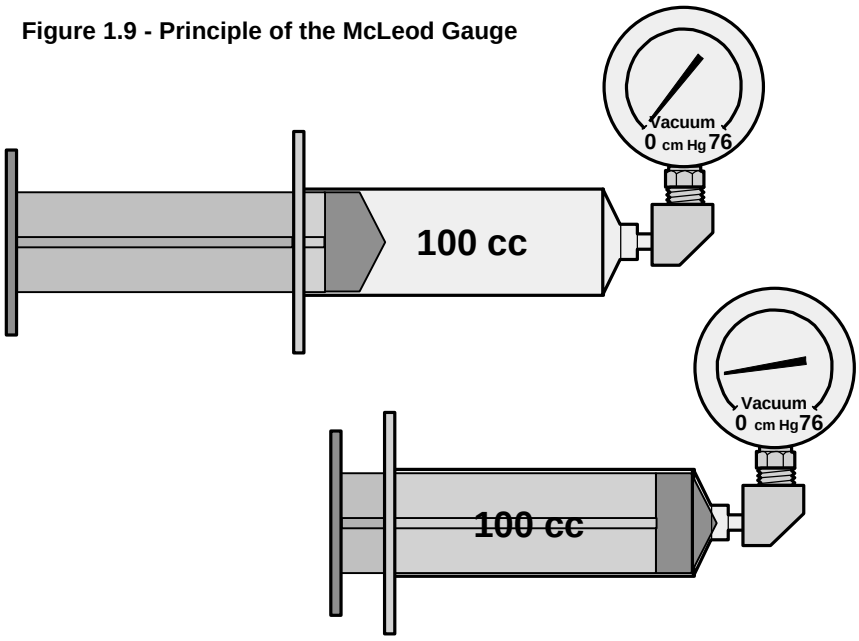
A typical configuration of a real McLeod gauge, taken from John Strong's "Procedures in Experimental Physics," [1] is shown in Figure 1.10. At this point I'll move to Strong's description of this gauge.

"The McLeod gauge is the simplest and most reliable (absolute gauge) for permanent gases, but it has the disadvantage of giving erratic response or no response at all to water vapor, carbon dioxide, ammonia, and pump oil vapors which adsorb on the walls of the gauge or condense to a liquid. This disadvantage is serious, inasmuch as water vapor, carbon dioxide, and so forth are often of importance in the last stages of obtaining a high vacuum.

"The gauge as ordinarily used today (*remember, this dates from 1938*) is essentially the same as it was originally. It is made of glass and is mounted on a vertical board. The difference in the heights of the mercury levels in the gauge and in the reservoir is approximately equal to the barometric pressure *B*. As the reservoir is raised, the mercury level in the gauge comes above the **Y**-branch, thus isolating a definite volume V_1 of the residual gas. This is isolated at the unknown pressure P_1 , the pressure of the residual gas in the apparatus to which the gauge is connected. As the mercury reservoir is further raised, the isolated residual gas is compressed, and when its volume has been reduced to a volume V_2 , the pressure is great enough to produce a sensible difference in the height of the mercury meniscus in the two capillaries, *A* and *B*. At the left in the figure, the mercury levels are shown at the beginning of the measurement, and at the right they are shown in two different positions corresponding to two methods for making readings. In one, if the meniscus in *B* is adjusted to the same height as the top of capillary *A*, the final volume, V_2 is equal to $\Delta h \cdot \sigma$, when σ is the cross-section area of the capillary. The decrease in volume from V_1 to V_2 is ordinarily of the order of one-hundred-thousandfold, with a corresponding increase of pressure in the capillary over that which obtained originally. The construction of the gauge with the comparison capillary *B* of identical bore with *A* eliminates the necessity of making corrections for surface tension. Referring to the earlier equation (Boyle's law) we see that the product P_1V_1 is, in this case, a constant. The original product, P_1V_1, is equal to the final product P_2V_2. From this we get the expression connecting the unknown pressure with the observed manometer difference, Δh:

$$P_1 = \frac{\sigma(\Delta h)^2}{V_1}$$

Figure 1.9 - Principle of the McLeod Gauge



V_1 and σ are constants of the gauge determined when it is constructed. σ is obtained by measuring the length of a known volume or weight of mercury in the capillary. V_1 is determined by filling the gauge with mercury. These original data may be recorded on the board to which the gauge is attached. Here they will not be lost. Values of P_1 determined by the equation are usually laid off on a nonlinear scale, which is mounted behind capillary *A* in order that pressures may be read directly.

"The second procedure of making the observations on V_2 and P_2 is illustrated at the right of the

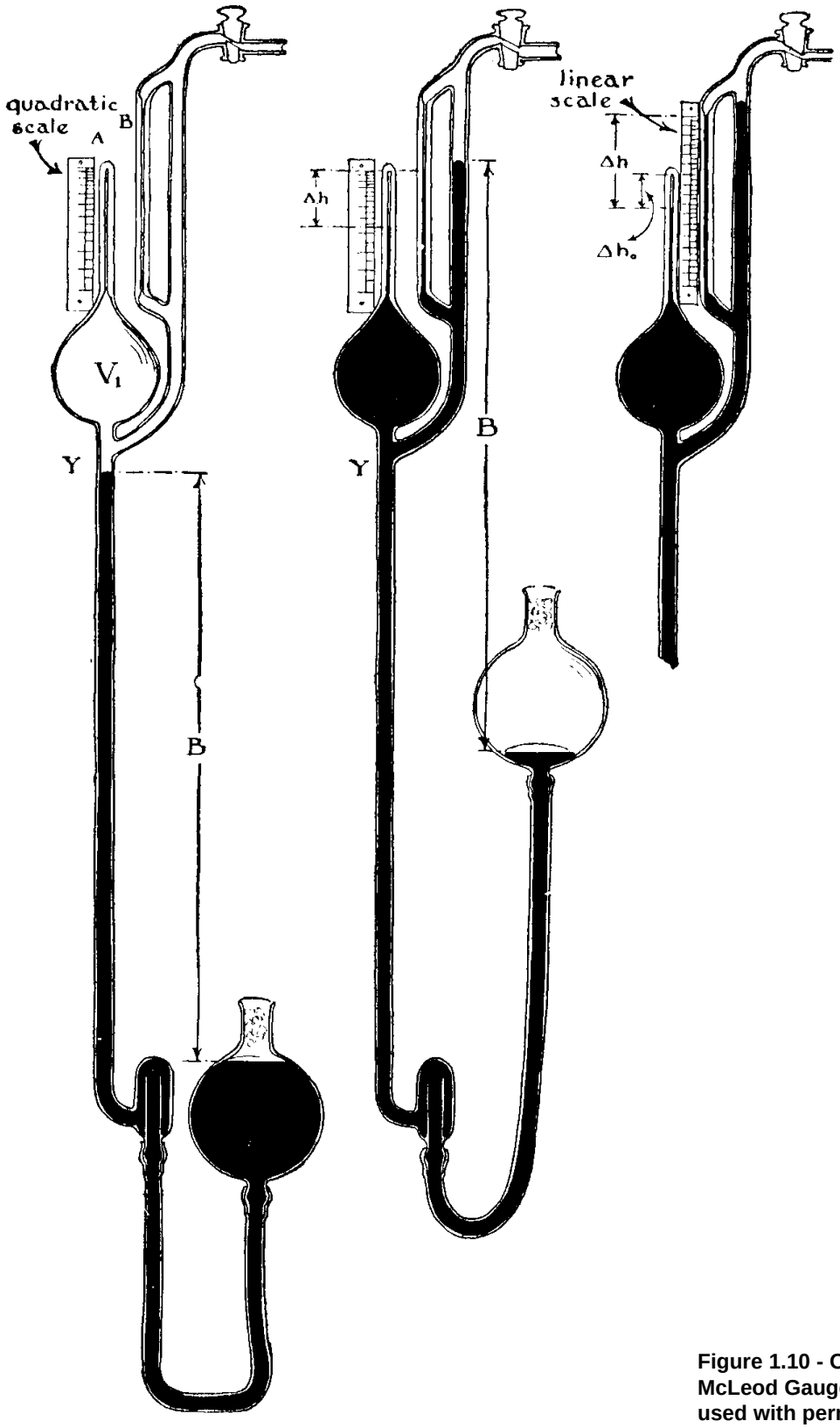


Figure 1.10 - Operation of the McLeod Gauge. From Ref. 1, used with permission.

figure. The gas is compressed to a definite mark on capillary A at a distance Δh_o from the top, so that the final volume, V_2, is the same for every measurement. The final pressure necessary to compress volume V_1 to V_2 is Δh, and the pressure P_1 in the system is determined by these quantities, according to the following equation:

$$P_1 = \frac{\sigma \Delta h_0}{V_1} \Delta h$$

A linear pressure scale computed from this formula is ordinarily mounted behind capillary *B*.

"The McLeod gauge is thoroughly reliable for the permanent gases from 10^{-1} to 10^{-4} mm of mercury. It is less reliable to 10^{-5} mm. Below this the indications are only qualitative, and at 10^{-6} the mercury often sticks in the top of the capillary *A*.

"The gauge is most reliable after it has been outgassed by gently warming it with a soft flame. Three gauges with different values of V_1 are necessary to cover adequately the complete pressure range from 10^{-1} to 10^{-6} mm. Many of the designs of the McLeod gauge are more elaborate that the one shown in the figure. For example, three bulbs may be mounted together with one reservoir, one for low pressures, one for intermediate pressures, and one for high pressures." At this point, Strong goes on to discuss the particulars of using McLeod gauges.

Full size McLeod gauges are not the type of thing that would be terribly useful to the amateur. And, because they have been replaced by better, easier to use devices, they are now rarely found in commercial or laboratory environments.

There are a number of smaller versions of the McLeod gauge. The tilting gauge is one of these and this type appeared from time to time in the older *Amateur Scientist* columns in Scientific American. Finally, even if one has a McLeod in hand, the use of mercury poses a number of hazards ranging from health to system contamination if the gauge is not equipped with a cold trap.

WHAT TYPE OF GAUGE TO USE?

With the McLeod gauge, cumbersome as it is, you do get something that is capable of providing very accurate measurements over a wide range of pressures which, unlike the indirect gauges, doesn't care what type of gas is in the system as long as the gas is permanent (non condensing). But, what if you don't want massive pieces of glassware or the problems associated with dealing with mercury?

It turns out that there are a number of options that are fairly simple and sometimes relatively inexpensive. What you really need to do is assess your gauging requirements based on what you want to do with the system.

For example, one common use of gauges is to give an indication of when it's ok to turn on the diffusion or turbo pump. Probably there's just air or nitrogen in the system and you don't have to be too accurate. One of the simple thermal conductivity gauges described in past issues will do just fine.

If your interest is vacuum evaporation, then it's important to know that you are somewhere below 10^{-4} Torr or thereabouts before you heat the slug or ingot. Again, super accuracy is not needed unless you are doing something really critical. For this an ion gauge (hot or cold cathode) makes sense.

Things get trickier if you are doing something like making a "neon" sign or filling a laser. These are multi-step processes that involve pumping the system to a real low pressure (1 milliTorr and possibly much lower), cleaning the system to get rid of water and other "poisons", and then backfilling to some particular pressure with a gas that is not air. Here we have two gauging requirements: an air measurement at high vacuum with ball-park accuracy to ensure that the proper base pressure requirement has been met, and a high accuracy measurement with some non-air gas to ensure that the device has been precisely filled to the pressure necessary for proper and long-lived operation. This pressure is often in the range of about a Torr to several tens of Torr and the necessary accuracy is on the order of tenths of Torr, give or take depending upon the specific application.

The first part can easily be taken care of with an ion gauge or a Pirani gauge with a good low end. The latter step requires a continuous reading gauge whose reading is gas independent. The pressure range that needs to be covered isn't particularly wide but good accuracy is required within that range.

Going back to the barometer of Figure 1.8 we have almost what we need. Since the pressure range only needs to go to, say, 25 Torr, we can shorten the barometer tube to get a simple, closed-end manometer. Since the glassblowing is a bit intricate, we can simplify the design to the configuration of Figure 1.11 which is nothing more than a U-shaped piece of glass tubing with one end sealed. The next section will discuss this style of manometer in more detail.

THE U-TUBE MANOMETER

The configuration of Figure 1.11 is the basic U-tube manometer. In this implementation what gets physically measured is the difference in the height of the columns. That is, for a 1 mm pressure change, the level of liquid in one arm will change by a half mm in one direction and the level in the other arm will change by a half mm in the other direction. While this gives us a small, cheap gauge, we still have the resolution problem as sub-Torr measurements will be hard to discern.

A way around this is to substitute a material that is less dense than mercury. With a lighter liquid, a 1 mm Hg change in pressure will result in a much larger change in column height. The amount of the change is proportional to the ratio of the specific gravity of mercury to that of the substitute liquid.

Vacuum oils are appropriate fluids to use in U-tube manometers as they have low vapor pressures. A common and inexpensive fluid is di-butyl phthalate. This material has a specific gravity at room temperature of 1.045. Since mercury has a specific gravity of 13.55, a 1 Torr pressure change in the measurement side of the manometer will result in a column height difference change of 13.55/1.045 or 12.97 mm. This added sensitivity makes the oil-type manometer quite useful in measuring with sub-Torr resolution.

There are a host of other fluids with suitable characteristics. All one has to do is look in a vacuum products catalog for low VP fluids. Selection will often be based on cost and the availability of small quantities. Sometimes you will have to do some digging to find the specific gravity. The Kurt J. Lesker catalog does list the significant properties of their fluids including s.g. There are some serious caveats with this type of device. We will look at these and some ways of overcoming them.

First of all, the accuracy of the measurement depends upon there being a perfect vacuum in the space on the reference side. After all, the manometer is just measuring the pressure differential between the volume on the reference side and the volume on the measurement side. Since we can resolve pressures of less than a Torr, any pressure rise on the reference side representing more than a fraction of a Torr will cause an offset in the reading.

What factors would cause the pressure to rise? For one thing, any water that is clinging to the tube will outgas, raising the pressure. Also, oil easily traps air and moisture. This will evolve into the reference volume, raising the pressure significantly (this is less of a problem with mercury). The net result is that the reference side will be a very imperfect vacuum. Of course, gases are also being evolved into the measurement side. However, the pumps, being on this side of the manometer, will be busy dispensing with these gases.

The solution to this is to modify the design such that the reference side can be pumped prior to doing a measurement or series of measurements. Furthermore, since such things as water vapor are slow to evolve at room temperature, the oil can be gently heated to hasten the process.

A more-or-less fully evolved U-tube oil manometer is shown in Figure 1.12. Rather than relying on the fall of the liquid in the reference side to create the reference vacuum, the reference side is pumped during the evacuation of the whole system by opening the stopcock that couples the legs together. Gases are evolved from the oil during the evacuation process by gently heating the oil by means of heater wire that is wrapped around the glass tubing.

Obviously, real leaks on the reference side will also cause an erroneous reading as will continued outgassing of any of the surfaces in this area. This can be quantified by closing the stopcock while the measurement side is still being pumped. If there is a leak, real or virtual, the liquid level on the reference side will fall as the pressure builds in that area.

There will always be some pressure rise. Doing this sort of baseline characterization will tell you how long you can operate the manometer before the reference side has to be evacuated again.

Needless to say, if the U-tube device is made of poor vacuum materials, this outgassing problem will only get worse. Therefore, avoid the temptation to build such a gauge from straight pieces of glass tubing connected together with lengths

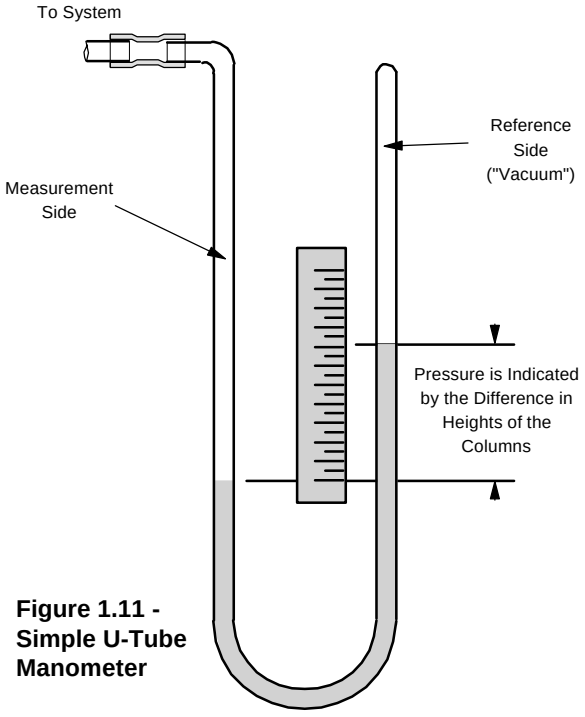


Figure 1.11 - Simple U-Tube Manometer

of rubber hose. If you need to make such a device and want to avoid the glassblowing part, try using clean copper plumbing fittings (tees and elbows) with the glass components sealed to the copper parts with a low vapor pressure sealant like Apiezon W wax.

There are a couple of other details. First, the accuracy depends upon knowing the density of the fluid. Even if you started with clean butyl phthalate or DC704, change it out if it begins to look like coffee. Accuracy also depends upon a good visual reference in terms of the fluid surface and also on a constant volume of fluid. Moore, Davis and Coplan [2] recommend that the tubing be at least 1 cm in diameter to reduce the meniscus effect. They also recommend that the tube be cleaned with dilute HF (use this stuff with caution) before filling to reduce oil adhesion to the walls.

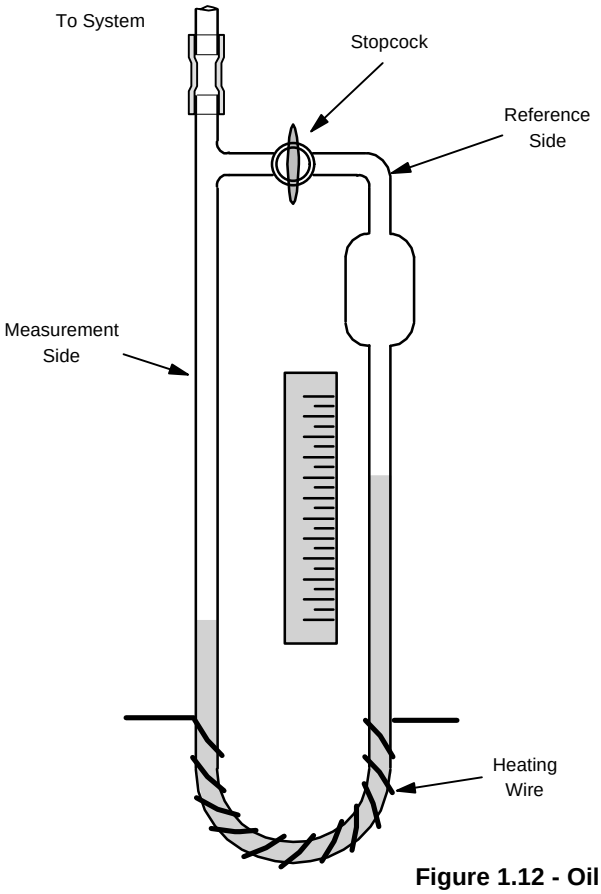


Figure 1.12 - Oil Manometer

SOME APPLICATIONS - VACUUM AND OTHERWISE

A. Backfilling

As was alluded to earlier, a primary application for the U-tube manometer is in backfilling pieces of apparatus with metered amounts of gases at low pressures. Neon signs, gas lasers and spectral lamps are common examples.

Figure 1.13 shows a typical implementation for the filling of neon signs or lasers. Specific examples may also be seen in the various Scientific American *Amateur Scientist* columns that dealt with gas lasers. The classic book, “Neon Signs” [3] also covers this technique.

High purity rare gases are not something that the amateur or small shop would go out and buy in pressurized cylinders. They are usually provided in glass flasks either as single gases or as specific mixes. The basic process of backfilling is simple: evacuate the apparatus, close off the pump, and fill to the desired pressure with the specific gas. In reality this can be a bit tricky.

First you have to precisely admit the gas, taking care not to overshoot. This would require repumping and wasting gas. You also want to be sure that the gas source is never connected directly to the pump. If that happens you will see your expensive He-Ne mix get instantly sucked into the pump and then blown into the room.

An explanation of the manifold in the Figure 1.13 will show how this can be avoided. As a first step and with a fresh, sealed container of gas connected to the system, the entire system is pumped down with all the stopcocks open. Once the device is at base pressure and the apparatus being evacuated has been cleaned up, supply stopcocks 1 and 2 are closed and the seal on the flask is broken. This is usually done by lifting the metal pellet with a magnet and then dropping it on the seal. Now the line from the bottle to stopcock 1 is filled with the gas and the space between the stopcocks is empty.

At this point the pumps and the manometer arms are isolated by closing the appropriate stopcocks. Now a little gas is bled into the volume between stopcocks 1 and 2 by cracking stopcock 1. Stopcock 1 is then closed and the gas is admitted to the apparatus by cracking stopcock 2. The manometer will measure the pressure as gas is admitted. If the pressure does not go high enough, then some more gas is admitted into the volume between stopcocks 1 and 2 and the process is continued.

Should it be necessary to backfill with a mixture of gases, more than one container of gas may be accommodated by adding more arms to the manifold. The mix ratio is determined by the incremental pressures as measured with the manometer.

Properly done, this is a well tested and simple way of backfilling. However, if you were to walk into a modern facility where quantities of signs or lasers are produced, you would probably be hard pressed to find lots of stopcocks and oil manometers. Instead you would see automatically sequenced valves, capacitance diaphragm gauges and automatic gas flow controllers. All of these would be tied together with closed-loop pressure control systems. Nonetheless, the idea is the same.

B. Calibration of Indirect Gauges

The U-Tube manometer is not the easiest device to use over long measurement periods but it does, with proper preparation and handling, give accurate, continuous and true pressure measurements. There are many times when one would prefer to use an electrical gauge, such as a Pirani. The problem is accuracy, particularly if you are dealing with some odd gas or mixture of gases.

This can be got around by comparing the Pirani to a true reading gauge such as the oil manometer and noting the indicated Pirani reading as compared to the real manometer reading. This will work as long as it is done with the gas or mix of gases in question.

If your need is to backfill a tube to 10 Torr with He-Ne, then just see what the Pirani reads when the oil manometer gives a 10 Torr measurement. If you are interested in a series or range of measurements, then do the comparison at several points and develop a calibration curve.

Whenever doing this, make sure that the Pirani and the manometer are closely coupled and that the system is allowed to stabilize at a given pressure. Otherwise you might be inducing pressure drops between the two gauges. Also, don't just do this once and rely on the Pirani gauge to remain stable for months or years. They usually aren't that stable.

C. A Simple Positive Pressure Control System

A couple of years ago while thumbing through some old copies of *Review of Scientific Instruments*, I came across the apparatus that is depicted in Figure 1.14. It really has nothing to do with vacuum of the sort normally covered by this publication but I thought the concept clever enough to merit inclusion at some point. Developed by Henry R. Jacobs [4], at the time a professor of medicine at Northwestern University, a water manometer and a few other parts are combined to make a simple device that can regulate gas pressures in the range of 1 to 50 cm of water, referenced to atmosphere.

Moderately pressurized air is admitted to the device by means of an aquarium-type vibrator compressor. A pinchcock and 1 gallon reservoir smooth any pulsations in the air supply.

The regulator itself consists of a pair of funnels connected by a piece of rubber tubing of about 75 cm length. One funnel, 35 mm diameter in the prototype, has a latex finger cot stretched over its opening. This membrane is placed flush to the large end (36.5 mm diameter) of a #7 rubber stopper. The funnel and stopper are held together with rubber bands.

With water in the funnel/rubber tube assembly, pressure within the range of the device can be regulated precisely and uniformly.

To measure the pressure, Jacobs connected an open end U-tube manometer containing water with a touch of detergent to lower surface tension.

Jacobs concluded his description with this statement: "This is a modest

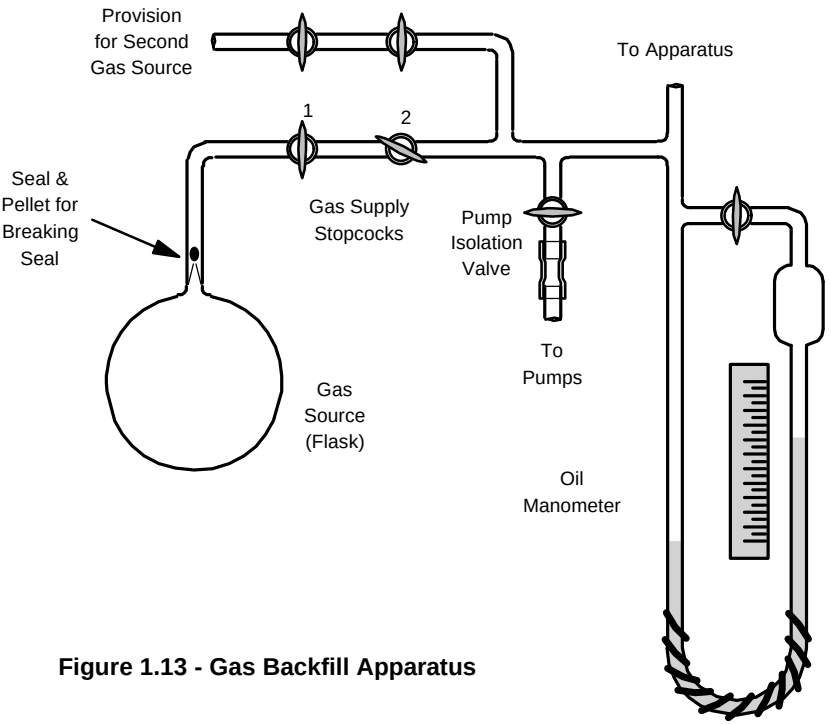


Figure 1.13 - Gas Backfill Apparatus

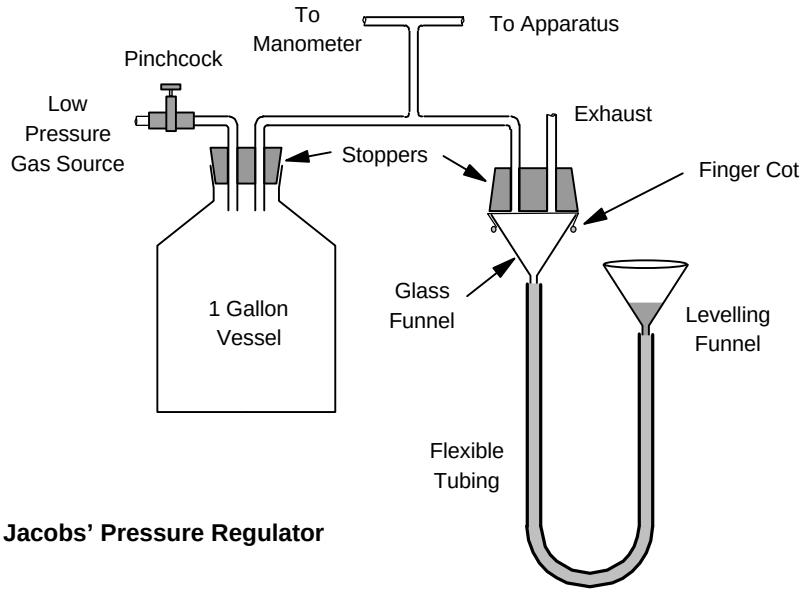


Figure 1.14 - Henry R. Jacobs' Pressure Regulator

but "beautiful" device. The author (who, being age 80, is leaving the field) believes that there is nothing like it available commercially."

THE STATE OF THE ART IN MANOMETERS

In the preceding section I dropped the term "capacitance diaphragm gauge." That's where the art is at today and I'll use this section to offer a brief description of this device.

U-tube manometers are reliable and accurate within limits. For most amateurs, those limits might never be exceeded. However, most industrial and scientific applications require better accuracy and resolution plus the ability to interface the gauge to other instruments. That's pretty hard to do with a column of oil. Finally, in many applications, really nasty gases get handled and these are usually incompatible with liquid manometers.

Thus, the requirements for such a gauge can be summarized as: made of non-corrosive materials, giving accurate pressure readings independent of gas type, a wide pressure range, highly accurate and stable, high resolution, and an electrical (analog or digital) output.

To give measurements independent of the gas type, the gauge has to work on the basis of true pressure, i.e. by measuring the force exerted on a surface of some area. This is how the liquid manometers work as well as the common Bourdon (dial) gauges.

In the CDG, also called a capacitance manometer, the pressure of the gas is applied to a tensioned diaphragm of inconel, an alloy chosen for its mechanical and chemical stability. But instead of measuring the deflection of the diaphragm by mechanical linkages, its changing curvature is sensed electrically by means of a capacitance bridge.

The first capacitance manometers were developed in the late 1920s with more recent development in the 1950s. The concept was made commercially viable in 1961 when MKS Instruments introduced their Baratron® device.

Figure 1.15 shows the basic concept. The sensor consists of a welded capsule which is divided by the diaphragm. Behind the diaphragm is a ceramic substrate upon which there is a metal pattern in a form similar to that of a circular bull's eye surrounded by a ring. As a result of this geometry, between each electrode and the diaphragm there is a capacitance that is defined by the areas of each electrode, the dielectric constant of the intervening gas (usually 1: air or vacuum), and the separation of each electrode from the diaphragm. Each of these variable capacitors is a leg in a bridge circuit. When the diaphragm is flat, the bridge is balanced and there is an output of 0 volts. However, as the diaphragm becomes curved, the bridge is unbalanced and the degree of imbalance is reflected in the output voltage.

The displacements of the diaphragm are small and subtle. Total displacements over the range of the sensor are on the order of a few thousands of angstroms. The circuitry can resolve displacements on the order of the diameter of a hydrogen molecule.

In the case of a differential CDG, both sides of the diaphragm are brought out through tubulations. To make the CDG into an absolute gauge, the reference side is pumped by the manufacturer to high vacuum and then sealed off. In

order to maintain high vacuum over the life of the instrument, a getter is fired during the pinchoff process, just as is done with vacuum tubes. The getter scavenges any gas that might desorb after pinchoff.

When a CDG's measurement port is open to any environment where the pressure is greater than the pressure within the reference volume, the diaphragm will be curved toward the electrodes and the output of the gauge will be some positive voltage. So, how do we translate that output to pressure?

Each CDG is specified according to its full scale (FS) range and it will be able to measure pressure for some number of decades below that full scale. In the case of a 10 Torr device, the output will be 10 volts at 10 Torr and the voltage will vary linearly with pressure. Thus 1 Torr will result in a 1 volt output, 0.1 Torr would give a 0.1 volt output, etc. Needless to say, as the pressure goes to, say, a hundredth of a percent of the full scale, the output voltage will be pretty small - a millivolt. This is about the bottom end of where a useful reading can be obtained and this usually corresponds to its resolution. Thus our 10 Torr FS gauge can measure to about 1 milliTorr although it will be most useful in the first 2 to 3 decades below FS. Want a lower reading? Get a lower range unit. CDGs are available with FS ranges to 20 milliTorr. Need a higher range? They are available to about 3000 psi. But, each one only covers about 4 decades of pressure with more from high accuracy, temperature regulated units.

One fact of life with CDGs is that the zero has to be adjusted upon installation. It should also be checked on a regular basis. There is always an accessible zero adjust pot on the instrument. All you have to do is pump the measurement side to a low enough pressure such that the output *should* be zero volts - usually about 5 decades below the full scale rating. Then you turn the pot until the output *is* zero volts. After doing this, all of the readings to the full scale should be within the manufacturer's specification; 1% of reading for general purpose devices and down to 0.05% for high accuracy instruments.

The zeroing pressure can be a problem with medium vacuum applications. If, for example, you have a 10 Torr gauge which you use for applications in the 1 to 5 Torr range, you will have to pump to below 0.5 microns to zero it. This requires a high vacuum pump. Some people try to get around this by setting the CDG to correspond to the reading

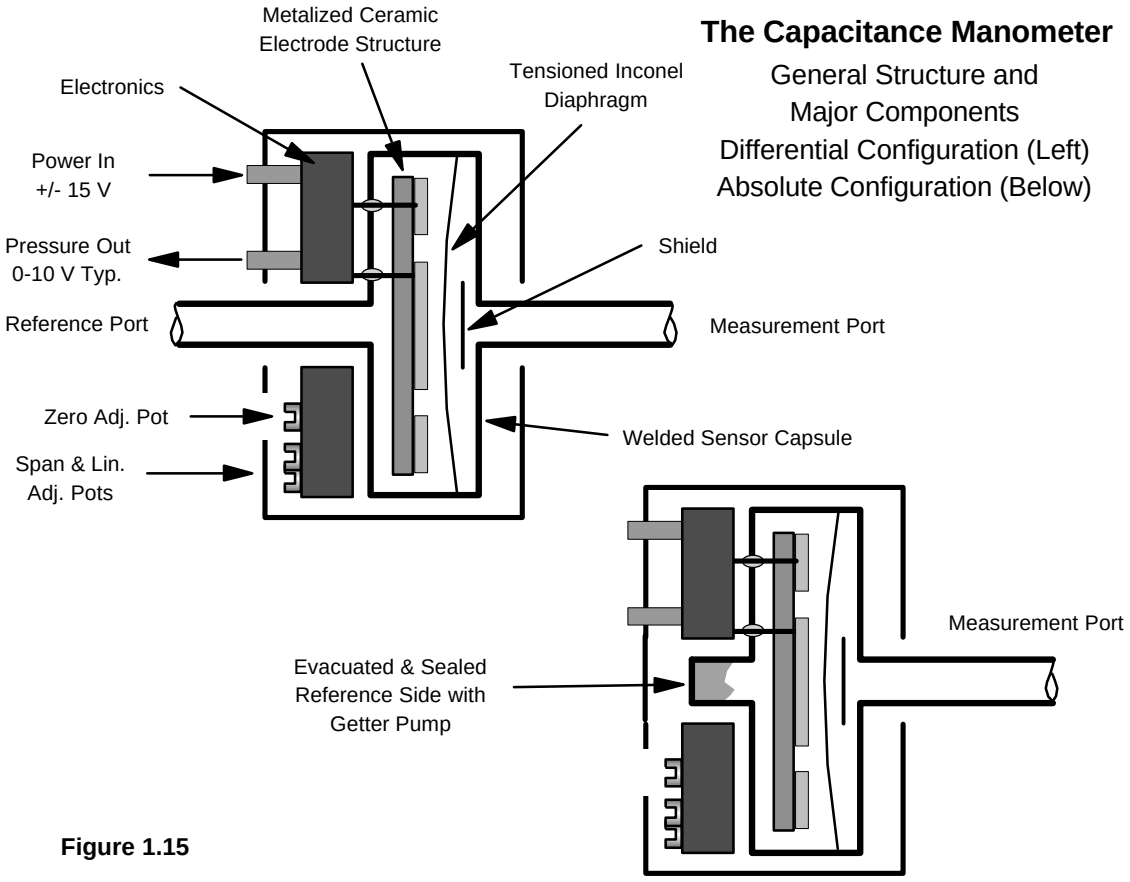


Figure 1.15

of, say, a thermocouple gauge. Wrong. A good TC gauge is lucky to achieve 25% accuracy. So much for the 1% accuracy you paid dearly for.

Zeroing differential gauges is easy - just connect the ports with a piece of tubing to assure the same pressure across the diaphragm and set the zero pot.

Over long periods of time other parameters such as the full scale range (span) and linearity will wander out of spec. Then it's time to send the device back to the manufacturer for recalibration. Although these pots are accessible (usually you have to pry out a plug), they should never be touched unless you know what you are doing and have a full calibration stand.

CDGs are wonderful instruments that have revolutionized vacuum processing. Parting with a substantial fraction of a thousand dollars for one will put off most amateurs.

In a future installment, we'll take a look at some other pressure measurement and pressure regulation devices. If you have come up with or used any neat or simple approaches in these areas, please send along some details.

REFERENCES

[1] John Strong, Procedures in Experimental Physics, (Prentice-Hall, 1938). Now available as a reprint from Lindsay Publications. Mandatory reading.

[2] John H. Moore, Christopher C. Davis and Michael A. Coplan, *Building Scientific Apparatus* (Addison-Wesley, 1983). A second edition is now available. Thanks to David Street for prodding me to mention this book. It's excellent.

[3] Samuel Miller and Donald G. Fink, *Neon Signs* (McGraw-Hill, 1935). Now available as a reprint from Lindsay Publications. Great little book.

[4] Henry R. Jacobs, *Precise regulator for low air pressures*, Review of Scientific Instruments, **54**, 1784 (December, 1983).

Applications for Mechanical Diaphragm Manometers

Pressure measurement using the Dwyer Magnehelic® gauge

Steve Hansen

This article was originally presented in Volume 5, Number 4.

INTRODUCTION

The preceeding article on manometers covered a range of devices including liquid column manometers of various sorts and the capacitance diaphragm gauge. The former can be inconvenient, messy or downright dangerous to use. The latter are a bit pricey for most amateurs. (Unless you got to the surplus store at the right time.)

Another device that can have some usefulness is the mechanical diaphragm gauge. A good example of a fairly sensitive form of this gauge is the Magnehelic®, manufactured by Dwyer Instruments, Inc. (P.O. Box 373, Michigan City, Indiana 46360, (219) 879-8000).

Magnehelics are usually found in HVAC applications: monitoring pressure drops across air filters, determining air flows or static pressures, etc. The gauge mechanism consists of a diaphragm that transmits its position to the indicator needle by means of a magnetic linkage. The diaphragm is mechanically connected to a magnet via a spring lever. The needle pointer is connected to a helix of magnetic material that simply follows the magnet. This mechanism avoids the frictional losses associated with gears, improving sensitivity and lessening the side effects of moderate overpressures (such as bent needles).

Just like the U-tube manometers discussed in the manometer article, these gauges indicate differential pressure. Also, since a number of different materials (metals, plastics, elastomers, etc.) are wetted by the gases whose pressures are being measured, this type of gauge should be used only with non-reactive gases.

New, the Magnehelics are in the $60 price range. Being fairly common, Magnehelic gauges can often be found at surplus outlets.

RANGES AND ACCURACY

Reflecting their usual application to HVAC, all of the Dwyer instruments are calibrated in terms of inches of water. To convert to the more vacuum-friendly unit of Torr (or mm Hg), the "inches H_2O" figure can simply be doubled to get you in the right ball park. To be more precise, multiply by 1.8683.

These gauges are manufactured in a wide variety of ranges. The most sensitive gauge has a full scale range of 0.25 in. H_2O (0.47 Torr). The high range of the standard units is 150 in. H_2O (280 Torr). Stated accuracies are 5% of Full Scale (FS).

The following table lists the ranges, resolution and accuracies of several gauges that would fall in the range of interest for vacuum use. Numbers in parentheses are torr.

FS Range	Minor Div.	Accuracy at 5% FS
0.5" (0.93)	0.01" (0.02)	(±0.05)
1.0" (1.87)	0.02" (0.04)	(±0.09)
2.0" (3.74)	0.05" (0.09)	(±0.18)
6.0" (11.2)	0.2" (0.37)	(±0.56)

Note that, since the accuracy is stated in terms of %FS, any gauge will have its greatest degree of accuracy as a percent of reading at the upper part of its range. Trying to measure to the degree of resolution of a 6 in H_2O gauge (0.2 in H_2O) would be futile as the uncertainty is nearly three times that figure.

ABSOLUTE GAUGE CONFIGURATION

As was done with the liquid column U-tube manometer (see Figure 1.12), all that is necessary to turn a differential gauge into an absolute manometer is to reduce the pressure on the reference side until that pressure is negligible with respect to what is being measured. However, in the case of the Dwyer gauge with its wide mix of materials, it would not be recommended to pump the reference side and then close that side off, hoping that the pressure would not rise. Instead, the reference side should be continuously pumped. When using the gauge for checking another gauge or for backfilling, that problem can be resolved by adding a valve in the pumping manifold.

Used with a decent mechanical pump, it is realistic to assume that the reference side can be pumped to at least 20 milliTorr. Of course, before betting your life or laser on this, you should check the base pressure occasionally by placing an absolute reading gauge on the reference port. This base pressure figure is pretty much down in the mud for a 1 in. H_2O FS Magnehelic gauge.

A CALIBRATION APPARATUS

Figure 1.16 shows an arrangement that can be used for calibrating another gauge of unknown scale or of lesser accuracy. (Remember, the calibration will never be better than the accuracy of the "standard" gauge. Therefore, work in the upper portion of the Magnehelic's range.) For example, this set up could be used to check the reading of a home made Pirani gauge over some portion of its range. Since the diaphragm gauge is gas-type insensitive, the set up could also be used to develop a calibration curve for the Pirani gauge for that particular gas type.

The manifold is constructed using ¼" brass threaded pipe fittings between the pump fittings and the chamber. Connections should be short to minimize conductance drops. Other fittings, such as the gauge ports and the needle valve, have 1/8" fittings so adapters will have to be used at these points. The valve that serves to isolate the reservoir and measurement side from the reference side and pump is a short length of ¼" id rubber tubing with a pinch clamp.

The prototype unit I built uses an aluminum tank as the reservoir. I believe the tank once served as a freon container. The volume of this reservoir is about 4½ liters.

A 6 in. H_2O Magnehelic is shown. This would be a good choice if we were interested in calibrating a Pirani gauge that would be used to measure pressures in the 1 to 10 Torr range. (6 in. H_2O units are currently available from C&H Sales. 2 in. H_2O units are in the Fair Radio Sales catalog. Prices are in the $20 to $25 range.)

The process is started by pumping the entire system down to its base pressure with the needle valve closed and the pinch clamp open. Once the pressure has stabilized (a minimum reading on the test gauge), the pinch clamp can be

closed. Since the pump is now attached to just the gauge, the reference pressure will drop a little more, making things even better.

Next, admit gas through the needle valve. The Magnehelic needle should begin to rise as should the indication from the test gauge. By opening and closing the needle valve, it will be possible to settle at a sufficient number of intermediate pressures to allow a calibration curve to be developed.

OTHER APPLICATIONS

This same apparatus can be used to size devices that serve to admit gases to a vacuum system. These are known as controlled or calibrated leaks, as opposed to plain old leaks that are just a pain. The technique involves flowing gas into a known volume over a certain period of time and measuring the pressure rise during that period of time. A simple calculation gives a leak rate in, for example, torr-liters per second or standard cc/min.

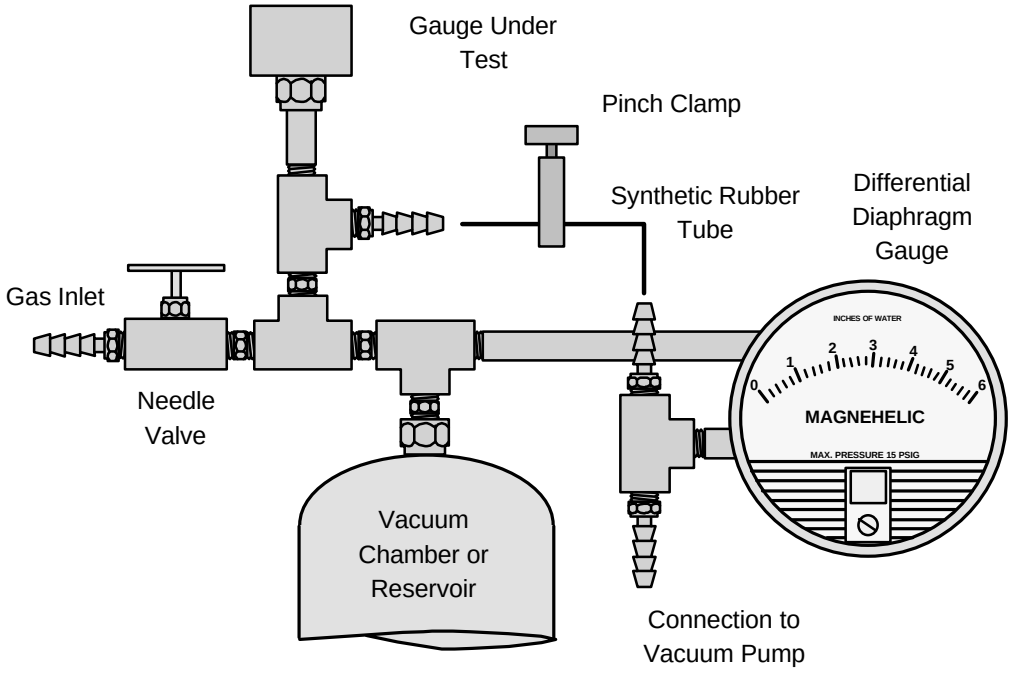


Figure 1.16 - Gauge Calibration Apparatus

Putting Vacuum Systems Together

This following articles cover the construction of vacuum systems of varying complexity. The first article is compiled from a series of articles that appeared in Volumes 1 and 2 and relates to my system, at least as it existed in the 1992-1993 timeframe. (Actually, while I've upgraded a number of things, the basic system remains pretty much the same.) The rest of the material ranges from descriptions of simple systems to Alan Ward and John Moon's ambitious dual chamber evaporator project.

An Amateur's Vacuum System

Steve Hansen

The editor's system will be described. This particular system was designed to be flexible and includes a large chamber (mechanically pumped) plus a diffusion pumped port which may be connected to a variety of apparatus and chambers.

OVERVIEW

Before getting to the specifics of this particular implementation, two figures from John Strong's classic *Procedures in Experimental Physics* [1] delineate very nicely the two broad classes of vacuum system. The first, the static system (see Figure 1.17), is designed for obtaining the highest degree of vacuum possible. These systems incorporate cold traps (when diffusion pumps are used), have no organic materials in the high vacuum side, and are bakeable. While Strong's sketch shows an all glass system, stainless steel or aluminum would be today's norm and the diffusion pump would probably be replaced by a turbomolecular, sputter-ion or other dry high vacuum pump.

The second, the kinetic system (see Figure 1.18), is designed for flexibility and frequent venting. Organics may be used in the high vacuum side and a cold trap (in the case of diffusion pumped systems) is optional. Obtainable vacuum would be in the 0.01 to 0.001 mTorr range with pump speed and patience compensating for the relatively gassy materials which may be used in the system. In the case of today's commercial systems, the distinction may be somewhat blurred (due to the advances in fittings and materials as well as newer pump technologies) but the differentiation is still applicable in the context of amateur built systems. The system described in this article is of the kinetic variety. This allows for a broad range of uses, permits the use of a variety of "alternative" construction methodologies and materials, and most important - is entirely adequate for an amateur's usual requirements (although I'm sure there is someone out there who wants to get into atomic level surface analysis).

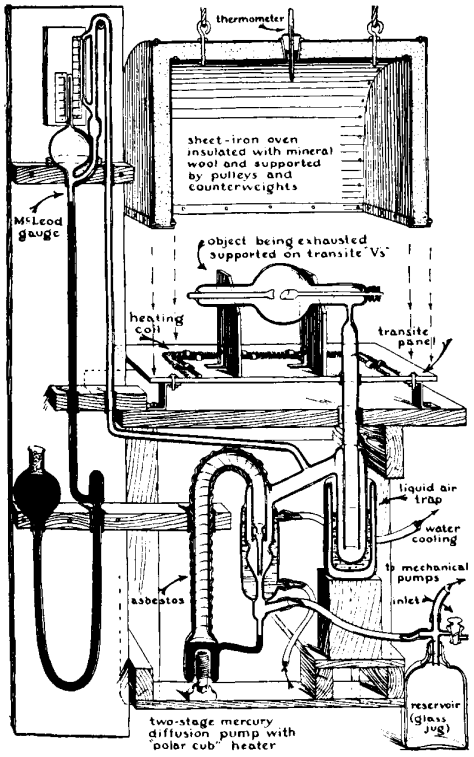


Figure 1.17 - Strong's Depiction of a Static System. From Ref. 1, used with permission.

SYSTEM ORGANIZATION

The two main features of the system are a large glass chamber (9" od x 3/8" wall by 17" tall) which may be evacuated by the mechanical pump and a separate port which is connected to the inlet of a small (2") three-stage oil diffusion pump. The rationale for this division was simple: no experiments or uses were foreseen which would require a vacuum quality better than 10 to 20 mTorr in the large chamber. Also, any of the high vacuum applications would be satisfied by smaller chambers which could be readily affixed to the diffusion pump port. When working on "dirtier" experiments in the jar, the diffusion pump may be isolated very easily by pinching off the rubber foreline tube with a woodworkers clamp. Likewise, when using the diffusion pump, the jar is isolated by blanking off its outlet port. Except for the vent, there are no valves in the system. (The obvious inconvenience is that the diffusion pump must be cooled down before opening the system.) Again, given my requirements (this is a pastime, not a production line), this is no problem and it keeps things simpler. Gauging is with a thermocouple guage in the foreline. A cold cathode (Philips) ion guage is used in the high vacuum side. The forepump is a Welch model 1402 belt driven rotary unit obtained from surplus. Figure 1.19 depicts the system. The remainder of this article will be concerned with the requirements of the cabinetry and the "rough" side of the apparatus. This will include, in particular the chamber.

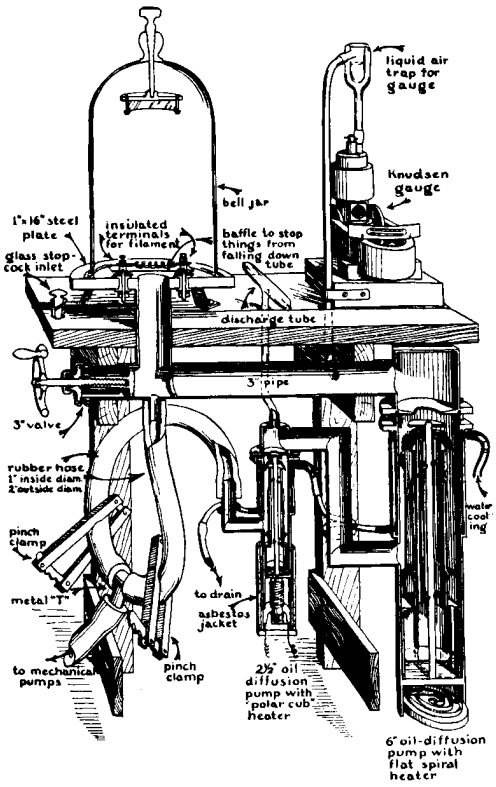


Figure 1.18 - Strong's Depiction of a Kinetic System. From Ref. 1, used with permission.

THE ENCLOSURE

Don't underestimate the value of a proper housing for your system. You may either construct a system that looks like an amateur built it or, with a little more in the way of thought and investment, make something that, although it truly was built by an amateur, will be more functional, safe, and pleasing to show off.

There are a few minimum requirements. First, the mechanical pump should be out of the way but easy to access when the need arises. Given the weight of a typical mechanical pump, mounting on a platform with casters is advisable. Due to the vibrations emanating from the mechanical pump, it should also be mechanically isolated from the rest of the system. Second, the enclosure should be stable. Third, there should be a good, ample and clean work surface. Fourth, the "nasty" parts of the system (these would include the forepump drive belt, the diffusion pump boiler, high voltage lines, etc.) should be concealed from idle hands and wandering pets. I met these requirements with a pair of steel tool stands (each about 21 inches wide by 16 inches deep by 34 inches high) which I obtained from W. W. Grainger Co., a wholesaler in industrial supplies. I bolted the two cabinets side by side with the intervening panels removed. I also cut out one of the bottom panels to fit the mechanical pump; it sets directly on the floor. As the cabinets are made from flat and angle stock and are assembled with machine screws, the side panel next to the mechanical pump may be easily removed to extract the pump for servicing. The top surface is 3/4" plywood with a plastic laminate (i.e. Formica™) surface. As there is some amount of high voltage circuitry within the enclosure (including charge storage capacitors) the cabinet is well grounded. The resulting six legged cabinet is solid if a bit weird to level.

Some other ideas for the cabinet system include printer stands (often framed with square steel tubing), wooden typing tables, copier and overhead projector stands, and other office related furniture. New, these can often be obtained at reasonable prices. Used items suitable for the application are commonly available at surplus office equipment supply stores. Other possibilities could include microwave oven and some TV stands. Refurbishing and modification usually will entail cleaning and repainting as well as adding a new or modified top and some plywood or sheet metal shields to cover the system's guts where needed. Needless to say, you may build the cabinet from scratch.

THE CHAMBER

For a general purpose system, a large chamber is - if not essential - certainly handy. Mine is used primarily as an experiment chamber for a coaxial plasma accelerator which is flanged onto the base plate as well as for demonstrations for the local kids. There are a couple of choices for the chamber. The first would be the traditional bell jar. These tend to be expensive except in the smallest sizes. The alternative is a glass cylinder. A disadvantage of the cylinder is that it requires two baseplates. (This is sort of like comparing a punt with a conventional pointy bowed boat: the punt requires two transoms, is not generally regarded as "handsome", but will carry a heavier load for a given length.) The advantages are several: cylinders seem to be easier to come by in moderate sizes, cost is lower, ports may be brought into either end with equal ease, and there is a higher useful volume for a given size. I opted for the latter even though it conflicts with the title of this journal (imagine *the* Cylinder!!). I do however have a small bell jar in my inventory. Whichever you opt for, the following will apply for the most part. My chamber is depicted in Figure 1.20.

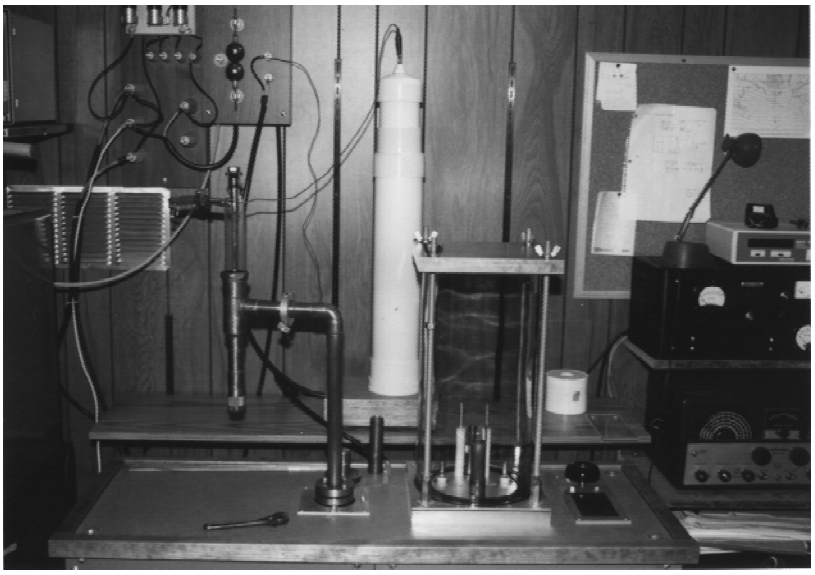
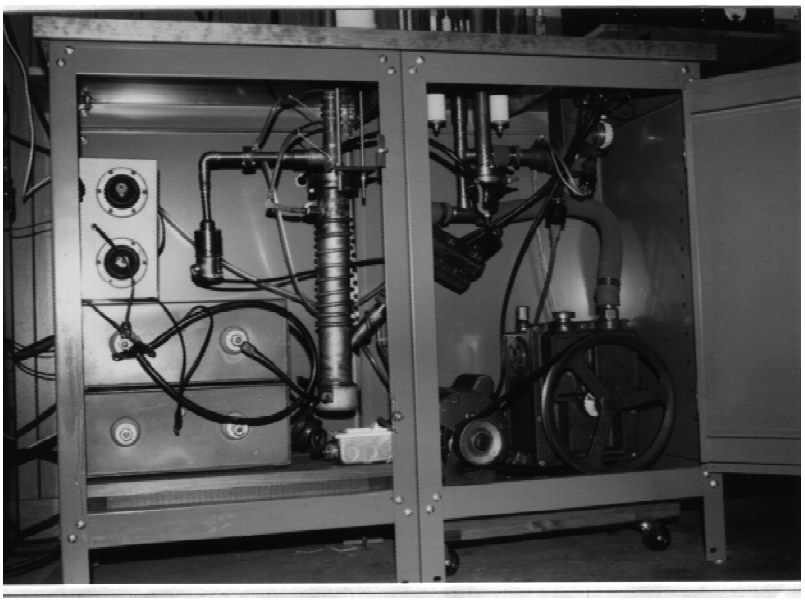


Figure 1.19 - The Editor's System in 1991. Above, exterior top. From right forepump switch & vent valve handle, large rough pumped chamber, high vacuum port with small experiment chamber. The gauge controller is at the far right. In the back is a 250 kV impulse transformer. Below, the guts of the system. Note the wood clamp that is used to isolate the diffusion pump. The Philips gauge is connected to the diffusion pump. Capacitors are for the plasma gun, located above the wood clamp.



The cylinder I obtained (from E. McGrath, a dealer in surplus vacuum and laboratory goods) was of Pyrex, dimensions as stated above. The ends are flame polished and ground just enough for flatness. (When buying surplus glassware for vacuum use, make sure that there are no scratches or other defects which would weaken the chamber.) The base plates are made from 1 inch aluminum obtained from a local scrap metal yard. One large plate had enough undrilled area to fabricate the two 10 inch square plates. The leftover plate was sold back to the dealer. A friend who has access to a machine shop (another route is to try your local trade school for machining jobs) milled the plates to size. As this is not a critical step, a thoroughly acceptable job may be done with a metal cutting abrasive blade in a circular saw. Take it slow and make several passes. Finish up with a file and use a machinists square to check accuracy. The plates you select must be free of large scratches and dings. These will impede sealing.

Do acquire your jar or cylinder before fabricating your plate or plates. Heavy wall borosilicate glass tubing suitable for vacuum service seems to be hard to come by in sizes larger than 7 inches in diameter (for reference, a standard 4 foot length of 7 inch tube will weigh in at 30 lbs and the new cost is around $13 per lb). A useful item to consider is glass process pipe. Sizes to 6 inch id are readily available new (e.g. from Ace Glass) in various increments of length from a

foot to several feet. Each end is "beaded" (i.e. flared slightly for joining with compression fittings and gaskets). The flare of a 6 inch id pipe is 7-1/8 inch. New, an 18 inch length of this process pipe will cost a bit over $100 (1992). Another nice aspect of process pipe is that you can obtain a variety of standard configurations including Tee's (if, for example, it were desirable to have a side arm on the chamber). Needless to say, surplus is a logical place to check for these items.

To aid in sealing during the early stages of evacuation, I have provided 4 half-inch threaded rods, one in each corner of the base plate. The lower, permanent, plate is threaded to accept the rods; the upper, removable, plate slides over the rods. Compression is provided by hex or, for more convenience, wing nuts. Gaskets are neoprene sheet (gasket material) cut to the size of the base plate with a circular hole slightly smaller than the id of the cylinder. There is also a 1/8 inch thick aluminum aperture plate over the gaskets (outside dimensions the same as the overall dimensions of each end plate with a sawn opening just larger than the cylinder). This is largely cosmetic but it does keep the gaskets in order. The only other machining step for the plates is the drilling of feedthrough holes and several 1/4-20 threaded holes (not through drilled) for the flanges (underside of the plate) and for the mounting of fixtures (topside of the plate/inside the chamber). The drawings of Figure 1.20 show the details of this chamber.. Note that there are 4 ports. One is for the vacuum connection, two are for high voltage electrical feedthroughs, one is a spare.

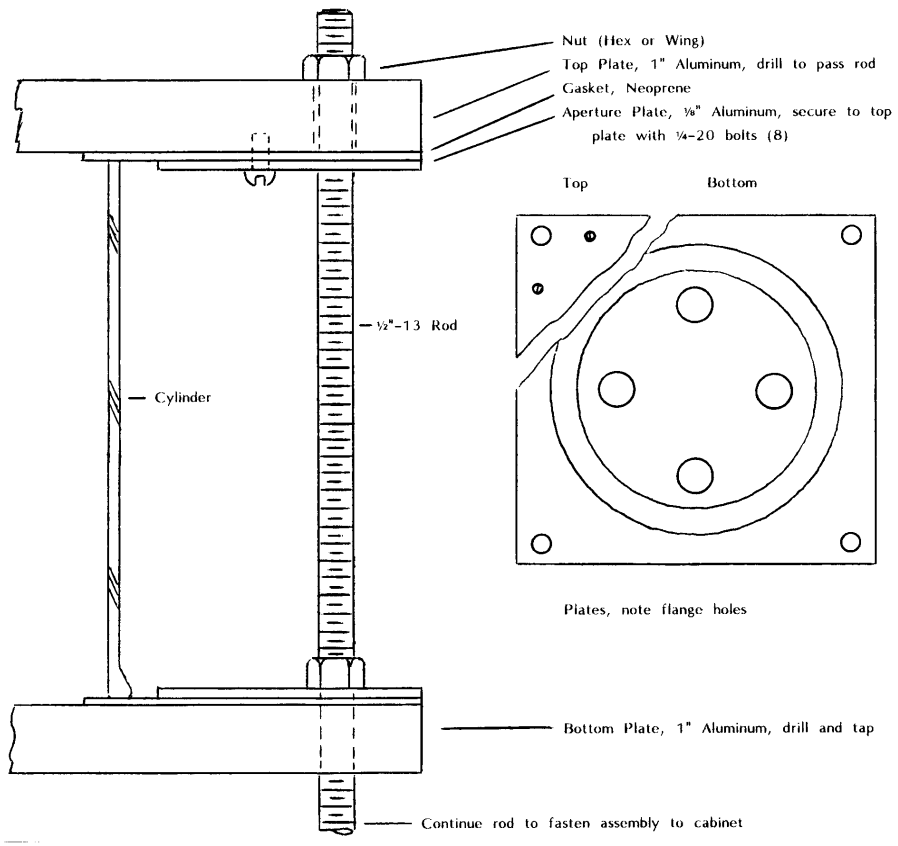


Figure 1.20 - Chamber and Baseplate Detail

From a safety perspective be sure to provide a shield around the chamber. I have a three sided Lexan™ shield. The corners a joined with light aluminum angle and pop rivets. Your shield should be transparent and easy to remove/replace (otherwise you won't use it).

A SIMPLE FITTING FOR TUBE TO PLATE CONNECTIONS

Here is a simple design for a fitting which uses standard components (a stainless KF (or QF by some suppliers) adaptive ring and an ordinary steel washer) to couple 3/4" copper water tubing to flat plates. The same approach may be used to provide electrical feedthroughs into vacuum apparatus. A standard KF assembly consists of two flanges, a centering ring (which holds the o-ring in place), and a clamp assembly which compresses the component parts together. Used this way, the KF system is for coupling one tube to another. The mating flanges are usually of the same size and will use the normal brass or stainless centering ring. However, an adaptive ring may be used to couple two adjacent sizes of KF flange. These rings are available from suppliers such as Lesker or Duniway. As it turns out, one size of KF adaptive ring will mate directly with the od of standard 3/4" copper water tubing (0.875" od). Lesker carries this as part number QF20-25. The joining may be performed by soldering with tin-silver solder. Pressure is applied to the assembly with a

standard 1" steel or brass washer (1-1/16" id by 2-1/2" od), drilled for 3 or 4 screws (#10 to 1/4"). Total cost for the assembly is about $12.00. If an electrical feedthrough is desired, the same components are used. The copper tube is cut to about an inch or so and is filled with epoxy (the white hardware store variety will do) after soldering to the ring. When cured, drill a hole just large enough to pass a threaded rod (brass or stainless) of the desired size for the conductor (1/4" is a handy size). Degrease the rod and insert it into the drilled plug about 1/2" higher than the final position. Smear some more epoxy on the threads just above the plug and then slowly push the rod to its final position. Turn the rod a bit to ensure that you get rid of voids. Then apply some more epoxy at the surface. (If the feedthrough is for high voltage, you can coat the rod for some distance for extra insulation. A glass or ceramic tube with the lower end embedded in the epoxy will do the same.) It is also possible to coat the epoxy surface with a low vapor pressure hard wax such as Apiezon W. Figure 1.21 shows both of the configurations.

REFERENCE

[1] John Strong, *Procedures in Experimental Physics* (Lindsay Publications, Bradley, Illinois, 1990).

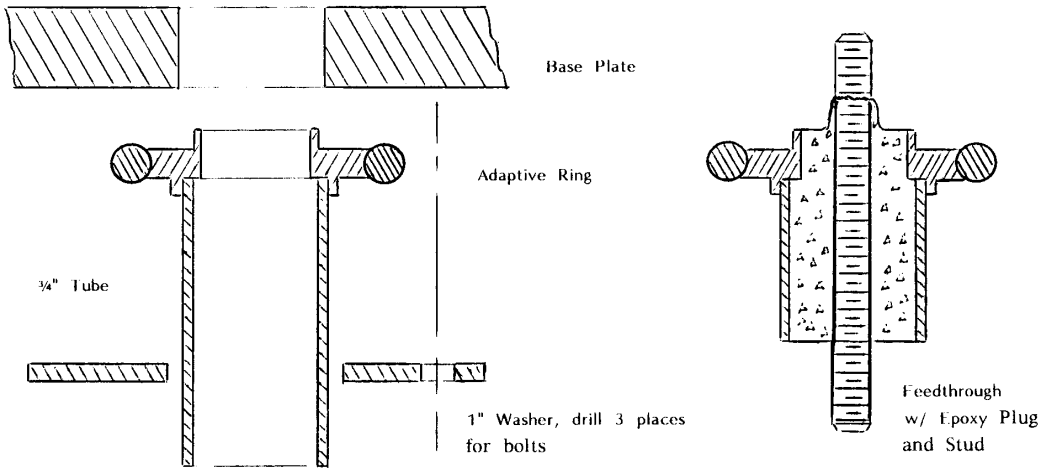


Figure 1.21 - Vacuum Fitting/Electrical Feedthrough

Diffusion Pump Basics

Steve Hansen

PRINCIPLES

It was noted in an earlier article that the mechanical displacement sort of pump (e.g. rotory piston or vane type), while entirely suitable for pumping in the viscous flow regime (where mean free path is considerably shorter than the sizes of the lines and pumping chamber), is not effective in the molecular flow regime where the mean free path is larger than the pumping chamber. Simply put, at very low pressures a gas such as air does not behave in the manner we normally associate with a fluid: you can't push it around with a plunger. At such low pressures another set of approaches is needed. One of the most common is to provide a mechanism which “whacks” each gas molecule that happens to wander into the pump. The direction of each “whack” is uniform and toward the pump outlet. Getting technical again, this action provides a preferred direction to each entering molecule by the process of momentum transfer.

Where the crossover between where conventional mechanical pumping becomes ineffective and where high vacuum pumping techniques such as momentum transfer are required is not sharply defined, at least in practice. In general, displacement pumps give up at about 0.1 mTorr. However, these pumps are comparatively slow (relative to even a small momentum transfer pump) and tend to surrender to other losses (such as outgassing) well before this limit. Thus, even

though a two stage rotary pump may have a rating of 0.1 mTorr, in reality (with not quite pristine oil and evacuating a "real" system) it may not be able to easily get below a few mTorr with any regularity. Hence, it is desirable to have some overlap so that the high vacuum pump can take over well before the displacement pump gives out.

EVOLUTION OF THE DIFFUSION PUMP

The first and traditional method for achieving this momentum transfer is embodied in the diffusion pump. The pumping mechanism here is very simple: a stream of vapor flows through a jet which is located in line with the plumbing of the vacuum system. The high speed stream of vapor molecules pushes the incoming molecules away from the inlet where they then become compressed in the outlet region. As the pressure is higher in this area a normal mechanical pump may be used to "back" the diffusion pump. The selection of the working fluid for this type of pump is critical. It would be counterproductive to use something which releases as much gas into the vacuum system as one is trying to take out. The way of getting around this is to boil a low vapor pressure liquid, pass the resulting vapor through the jet where the work gets done, and then condense the vapor as quickly as possible and return it to the boiler. The first working fluid which was shown to be effective was mercury and the first practical mercury diffusion pump was developed by I. Langmuir in the second decade of this century.

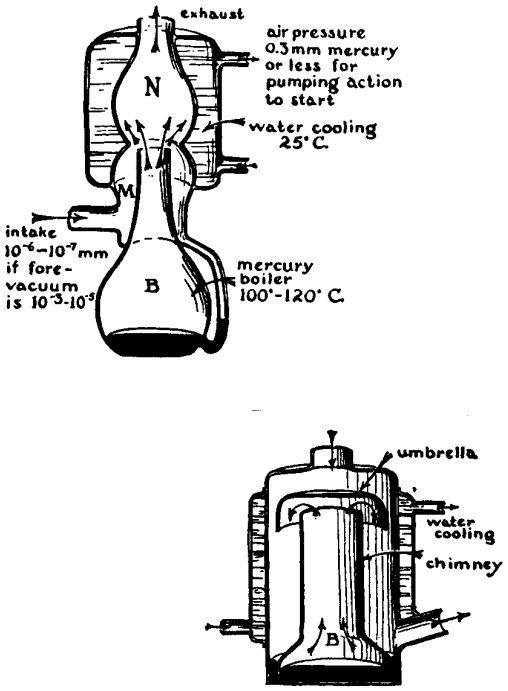


Figure 1.22 - Two of Langmuir's Pumps. From Ref. 1, used with permission.

Figure 1.22 shows two of Langmuir's pumps. Each has the required parts: boiler, jet, condenser, return path, inlet, and outlet. The differences are in material of construction (glass vs. metal) and jet configuration (up-jet vs. umbrella).

As with mechanical pumps, additional pumping stages will improve efficiency and capability. Figure 1.23 shows a two stage pump as developed by Kurth and Ruggles. Here the vapor stream is split between two sequential sets of jets. This particular design has persisted to this day and models of varying sizes are available as stock items from several laboratory supply houses.

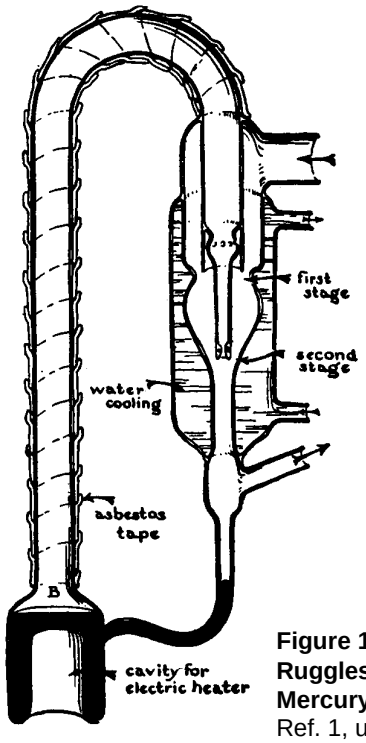


Figure 1.23 - Kurth-Ruggles Two Stage Mercury Pump. From Ref. 1, used with permission.

A disadvantage of mercury is that it has a relatively high vapor pressure. A cold trap (at liquid nitrogen temperature) is always required to prevent mercury from backstreaming into the vacuum system and contaminating the apparatus. A variety of hydrocarbon oils were shown to be effective alternatives to mercury and these (including some later developed synthetic oils) have since become the usual working medium for diffusion pumps.

Strong [1] points out some advantages of oil in diffusion pumps. These include (1) a wider choice of compatible materials for pump construction (in addition to welded stainless steel and glass, aluminum, brass, copper, mild steel, and solder joints are acceptable. Strong does caution against unplated copper and brass coming in contact with hot hydrocarbon oil, these areas should be nickel plated.) and (2) cold (LN_2) traps are not necessary for many applications - chilled baffles or charcoal traps are generally adequate.

There are some disadvantages. Oils deteriorate over time and *very* quickly if hot and exposed to atmospheric pressure (the

synthetic oils are much better if one can justify the added cost). Also oil pumps are more demanding of a good fore vacuum. 100 mTorr is a typical value whereas some mercury pumps can operate against a forepressure in excess of 1 Torr. In appearance, early oil pumps looked much like their mercury counterparts.

Figure 1.24 shows a two stage glass pump with a metal chimney and an inlet fractionating column by Hickman (ca. 1936). Except for a transition to all metal construction, the usual addition of a third stage, and some scaling up, most modern diffusion pumps are configured very much like this one.

With the exception of some laboratory applications, oil pumps now dominate where diffusion pumps are used. Standard commercial models span from small (1" diameter inlet and 10 liters/sec) to 48 inch monsters. The major modern competition to the diffusion pump is the turbomolecular pump where, instead of a moving vapor giving a preferred direction to the incoming gas, that preferred direction is imparted by a set of fast moving angled blades. The tradeoff is cleanliness vs. mechanical simplicity.

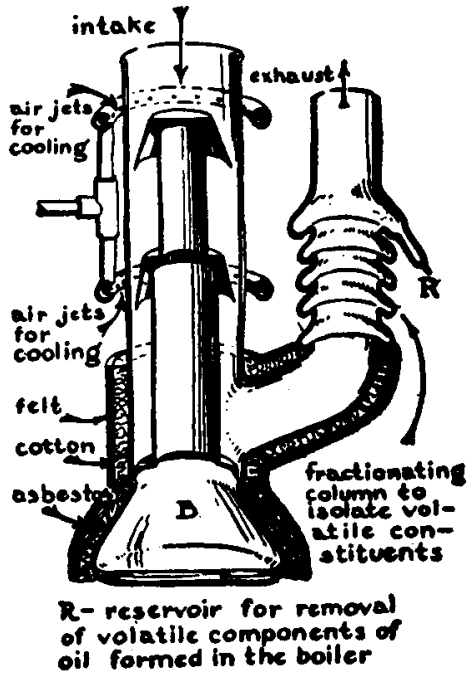


Figure 1.24 - Hickman's Two Stage Glass Oil Diffusion Pump with Metal Chimney. From Ref. 1, used with permission.

For the amateur, I think that the oil diffusion pump is hard to beat. They do take some care but goofs (like letting in atmospheric air at the wrong time or bits of glass) usually result in no more than a tedious cleanup job. On the other hand, the turbo pump hates dirt and will repay mishandling with the need for a costly rebuild job.

If you are building a system from scratch, a first order concern is the size of pump needed. It's most common to size a pump by its inlet opening. I've found a 2 inch pump to be acceptable for all of my needs i.e. the evacuation of small ion and electron beam devices to pressures on the order of 10^{-4} to 10^{-5} Torr. This size pump (it's rated at about 100 liters/sec at the inlet flange) will also handle a small bell jar. About the largest pump that an amateur would want would probably be in the 4 to 6 inch range and then only if one were working with a larger chamber or had an application such as sputtering where a fair volume of gas is continuously being admitted to the chamber. A good way of judging is to simply look in a catalog of vacuum apparatus and note chamber size and intended purpose along with the size pump which the supplier has built the system around. In general, where little if any gas is to be admitted, a 2" pump (about 150 liters/sec) can handle a bell jar (or equivalent volume) of 12" diameter by 12" high. A 12" by 18" chamber is well matched to a 4" pump (about 500 liters/sec) and chambers up to 24" by 30" should have a 6" (about 2000 liters/sec) pump. (Depending upon manufacturer and model, these throughputs may vary considerably for a given size; consider them as an an order of magnitude estimation only. Plus, as soon as you add any fittings to the pump inlet, the speed will suffer.)

Most amateurs can't afford to be picky - availability and a good bargain are reigning parameters. My general recommendation, however, would be to get as small a pump as will be suitable to your needs. (Too small is, of course, a worthless pump.) Bigger means more costly accessories, more oil (never cheap), more power, more water, and more physical exercise when the thing gets crudded up and needs to be cleaned.

GETTING YOUR PUMP IN RUNNING ORDER

A good surplus pump should have a working heater, no obvious leaks in the water cooling lines, no dents in the barrel, a high vacuum flange that looks ok and can be mated to something, and no obvious signs of abuse to the chimney and jet structures. Chances are, unless it is a rebuilt unit, it will be real dirty.

One important thing to remember is that diffusion pumps are meant to be taken apart; repeatedly. Don't hesitate to dismantle everything that is tied together with screws. Just make sure you keep everything and remember where it all went.

Cleaning is best done with alcohol and/or soap (pure) and water. An abrasive cloth like 3M makes is quite handy. Avoid steel wool as the fibers can get embedded in the softer parts of the pump (e.g. aluminum jets). A final rinse can be

done with distilled water. At this point it's best to start using latex gloves (the non sterile kind available at drug stores) to avoid depositing fingerprints as the parts begin to dry. Lint free disposable lab towels (Kimwipes™) should be used to dry and prevent spotting. When I clean my 2" pumps (both old NRC units) I pop them into the kitchen oven at 150° C for a bit to dry everything out.

When I acquired those pumps they each had several items besides cleaning which required attention. First, the cooling coil had been damaged at the upper end. This I fixed by filing away the damaged section (about 2 inches worth) and patching in a new hose fitting. I lost that amount of cooling but I don't think the pump is any the worse for it. Next, the jets were dinged up a bit. These are thin aluminum and the fix was to take the chimneys apart and, with a metal rod, carefully work the lips back into shape. Last, the heater was insulated with flaking asbestos. Having done this in the 60s I just chucked the asbestos and ran the heater bare. It works ok. A better approach, if you were to come across a pump with asbestos insulation, would be to carefully remove it, bag it, and take it to your local recycling center on "toxic waste" day. Then replace the asbestos with ceramic wool.

If you have a small pump with a low wattage heater (200 W range) it is worthwhile to add a variable control. I use a slide type light dimmer switch rated at 600 W.

OPERATION

At least at the outset, use whatever oil the manufacturer recommends. Also, the right amount. If you don't know how much oil to add use the following guidelines:

- Enough to cover any recesses in the bottom of the pump (sometimes there are concentric rings which serve to shroud the base of the chimney).
- Not enough to reach the inlet tube.
- In general about 70 ml for a 2" pump, 100 ml for a 4", and 200 ml for a 6".

The water cooling line, with a series valve for regulating flow, should be connected to the top of the pump, where the pump should be coldest. The outlet line can go to any convenient drain but you should have a way of measuring flow. (I use 1/4" id plastic tubing stuck in a drain line in the basement where the washing machine also dumps. For gauging flow I put the tube in a quart bottle and note the time to fill.) When starting up the pump, make sure that you have a tight system and you that you have achieved a vacuum of 100 mTorr or better. At this point turn on the water (not before pumping down, otherwise you will get water condensation inside the pump) and then the heater. If you are not sure how much water is needed, start at 1 liter/minute for a 2" pump and 2 liters/minute for a 6". There should be enougn flow for the bottom of the cooling jacket to be cold when the oil is boiling.

Once the oil begins to boil (it's audible) you should note the pressure at the high vacuum end of the system begin to decline. I don't know what to do if that does not happen, the pump has always worked.

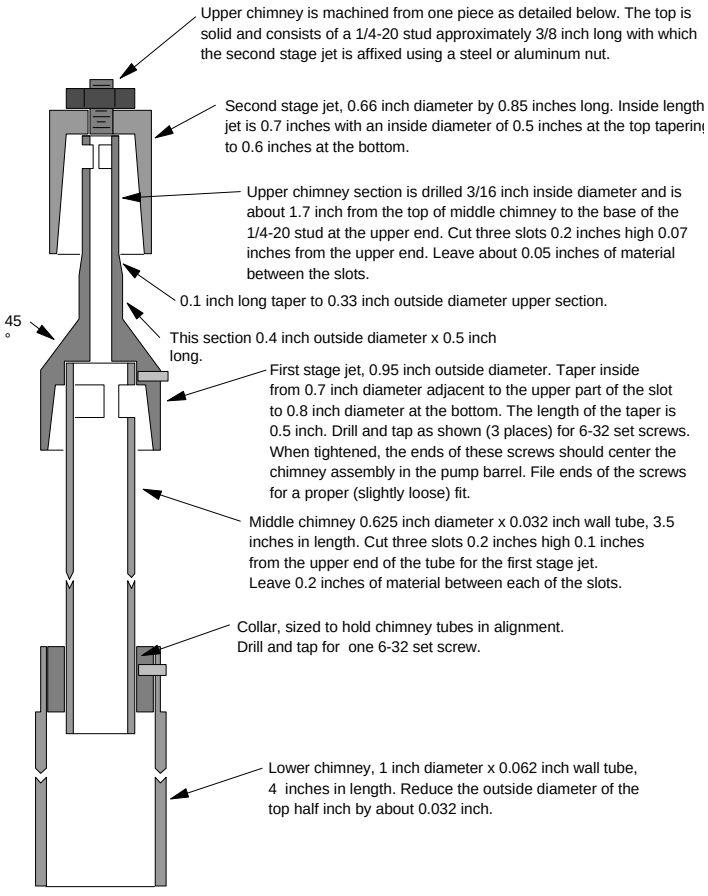


Figure 1.25 - Franklin Lee's Design for a 2-Stage 1-inch Diffusion Pump Chimney.

MAKE YOUR OWN DIFFUSION PUMP

While I have yet to try it, I believe that making your own small metal diffusion pump would be well within the realm of possibility. I'm not alone in this sentiment. Franklin Lee provided me with a information on a small Edwards pump, model EO1.

Franklin notes “I think this pump sells for $700 new which is absurd. Any machinist could make one in one day if materials were at hand. I made one once for somebody. He said it worked ok but I have no data.”

The pump has a one inch bore and two stages. Speed is 9 to 10 liters/sec with an ultimate vacuum of $5x10^{-6}$ Torr or better. The critical backing pressure is 0.5 Torr (high!) with an 11 ml charge of Dow Corning 702 silicone oil.

Franklin has provided a drawing of this pump dimensioned well enough for a person reasonably skilled at machine work to make a duplicate. He also has developed a design for a 2-stage chimney that uses standard aluminum tube and rod stock throughout. This is shown in Figure 1.25.

REFERENCE

[1] John Strong, *Procedures in Experimental Physics* (Lindsay Publications, Bradley, Illinois, 1990).

Organization of a Diffusion Pumped System

Steve Hansen

See also Roy Schmaus’ article on page 1-23.

VACUUM SYSTEM STRUCTURE

Figure 1.26 is a schematic diagram of a typical high vacuum system. Any given system may be either more complex or simple than this example based upon the final application or degree of flexibility desired.

There are two pumps in the system. The first is a mechanical fore pump. This pump is used to get the entire system down to a pressure regime in which the second pump, the high vacuum diffusion pump, will operate efficiently. The two pumps, connected in series, will evacuate the chamber. A bell jar is shown. However, the chamber could be any apparatus which is to be evacuated. Connecting these major elements are a variety of tubes or lines, some of which include vacuum valves. Inserted at several points are gauge tubes of various types (depending upon whether the gauge is to be monitoring the low vacuum (rough) side of the system or the high vacuum side). Traps and baffles are included to prevent backstreaming of oil and water vaper from the pumps to the vacuum chamber. Finally a provision is shown for introducing a controlled flow of gas into the chamber for specific applications. The gas could be argon for sputtering, oxygen for plasma ashing, and so forth.

This system is designed to be able to have the chamber cycled between atmospheric pressure and high vacuum without shutting down the oil diffusion pump. This is accomplished by having a high conductance gate valve between the chamber and the diffusion pump. The split line from the fore pump permits the diffusion pump to be bypassed so that the chamber may be evacuated by the fore pump. Once the chamber is down to a pressure at which the diffusion pump will operate, the valves are adjusted to bring the diffusion pump back into the circuit.

HIGH VACUUM SECTION

The “guts” of the system is the diffusion pump and its associated baffle, trap, and high vacuum valve. The baffle is a cooled (to the temperature of cold tap water or less) device which has a set of internal plates which are arranged such that there is no line of sight path through the baffle but which are also open enough to minimize any added resistance to gas flow. The purpose of the baffle is to condense pump oil vapors. As such, the baffle should be placed as close as possible to the inlet of the diffusion pump. The trap is a device which is used for condensable vapors including backstreamed oil as well as for water vapor and other condensables. Typically, the trap is chilled to a very cold temperature. The most common cooling fluid is liquid nitrogen. The basic difference between a trap and a baffle is the temperature it is designed to be used at. If really cold fluid is run through a baffle it then begins to act like a cold trap...for obvious reasons.

In some situations a chilled baffle is all that is needed to attain adequate (user defined) purity in the vacuum chamber. Furthermore, the correct choice of pump fluid can reduce oil contamination considerably.

In the design of a system, consideration has to be given to the locations of the baffle and cold trap. These need to be kept under vacuum whenever there is cooling fluid in or flowing though the baffle or trap. Otherwise, water vapor (and

other condensables in the atmosphere) will rapidly condense on the interior surfaces and load up the system. Thus, as shown in the figure, the high vacuum valve is located above the chilled areas. Cooling fluid is never introduced until this region is under vacuum. (Likewise, cooling water to the diffusion pump is not started until the system has been evacuated by by the mechanical fore pump.)

ROUGH VACUUM SECTION

The plumbing in the rough vacuum section of the system is arranged so the the fore pump may work either through the diffusion pump (normal operating mode) or in a bypass mode when the chamber is being evacuated and the diffusion pump is under vacuum. To do this, one branch (the roughing line) is brought to the chamber above the high vacuum gate valve. The other branch (the foreline) goes to the diffusion pump outlet. Two in-line valves control the flow paths. Additionally, a vent valve is used to admit air prior to opening the chamber. In operating the system there are three distinct phases: startup of the cold system, letting up the chamber to amospheric pressure (venting), and returning the chamber to vacuum. Proper juggling of the valves (in a manual system) is required or some unhappy outcomes may result.

The materials which are used in the rough vacuum section of the system are not as critical as those used in the high vacuum portion. For example, rubber hoses are totally acceptable in the foreline but do not represent a good choice for any serious work in the high vacuum sections. One problem that is not completely obvious is the issue of contamination from the fore pump. As the figure indicates, there is a rather direct path from the fore pump to the vacuum chamber which bypasses all the high vacuum traps and baffles. Unless some precaution is taken to prevent the flow of mechanical

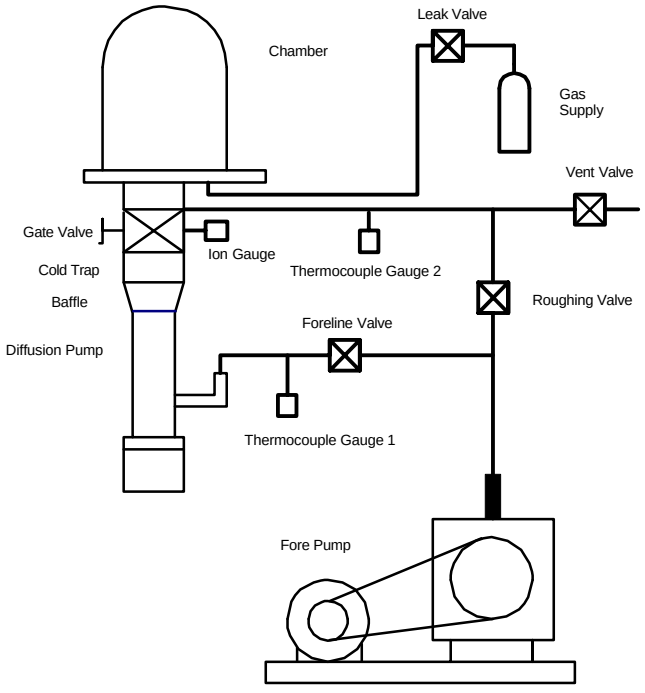


Figure 1.26 - Diffusion Pumped High Vacuum System.

pump oil vapor from that pump, the high vacuum region may become contaminated. This may be prevented or minimized by any of several methods including:

- Using a low vapor pressure mechanical pump fluid.
- Changing the mechanical pump fluid on a regular (frequent) basis.
- Using a foreline trap.
- Using care in the operating procedure.

Foreline traps were treated in an earlier article. While not shown, the proper location for the trap would be in the roughing line. With regard to operating procedure, the proper selection of pressures for switching between the various phases of the evacuation cycle can make a difference in the amount of oil vapor travelling through the roughing line to the vacuum chamber. This will be treated in more detail later.

GAUGING

During operation it is important to know the pressures in the various parts of the system. This is done by placing gauges of the appropriate types in specific places. Foreline pressure is typically monitored with a thermocouple (T/C) gauge located in the foreline. This should be located between the foreline valve and the diffusion pump outlet. It is also essential to know the pressure in the roughing line. Thus, two gauges are required for this basic system. A common alternative to the thermocouple gauge is the Pirani gauge. Another alternative would be a simple discharge tube. In fact, even if a T/C or Pirani gauge is used in the roughing line, a discharge gauge is useful in terms of providing a failsafe backup and to help determine what kinds of gases are in the vacuum chamber (by means of observing the coloration of the positive column).

None of the the above gauges are suitable for the measurement of the pressure in the high vacuum section once the diffusion pump has attained a vacuum of 1 mTorr or better. The preferred gauge for high vacuum measurement would be an ion gauge of either the hot cathode or cold cathode type. For an amateur's system the cold cathode (e.g. Penning or Philips type) offers a certain degree of robustness and easy maintainability that the hot cathode type of tube lacks.

The ion gauge should be located in an area that will be under high vacuum most of the time. Between the gate valve and the cold trap is a good choice.

GAS BACKFILL

For some applications it is necessary to have a gas other than air in the vacuum chamber. For example, if the system is for sputtering, argon is commonly introduced. Admitting the gas in a controlled manner is one issue. The other is the maintainance of the proper pressure (usually in the tens of milliTorr range but sometimes as high as a few Torr) without stalling the diffusion pump. The normal way of accomplishing this is to connect the gas supply to the vacuum chamber via a precision leak valve (which, due to the small quantities of gas needed, may often look quite unlike a standard valve) and then to throttle the diffusion pump by almost fully closing the connection between the chamber and the cold trap. By doing this the chamber will be at a pressure higher than that at which the diffusion pump will stall (which occurs in the 150 to 200 mTorr range) but the pump inlet will be at a much lower pressure - in the efficient operating range of the pump. In a simple system the gate valve can work as the throttle valve. In larger commercial systems it is common practice to have a separate throttle valve above the gate valve. These valves are not very good at sealing but the degree of conductance (controlled by varying effective open area) is adjustable in fine increments. In contrast, the normal gate valve is usually meant to be either fully open or fully closed.

OPERATIONAL SEQUENCE

The following sections outline the operating procedures to be followed when operating a system which is configured as shown in the figure. The operating procedure is divided into the three phases previously mentioned.

Startup. Before starting the system, all of the valves should be closed and all pumps, water lines, and gauges should be off. If the system has not been used for a while, check to be sure that there is adequate oil in the pumps. Then:

- Turn on the forepump.
- Open the roughing valve and turn on T/C Gauge #2.
- When the pressure in the chamber reaches about 100 mTorr*, close the roughing valve and open the foreline valve.
- Turn on T/C Gauge #1 and pump the diffusion pump section to about 50 mTorr.
- Turn on the diffusion pump heater and the water to the cooling coils. If the baffle is fed through another line, turn on that line as well.
- If using a cold trap, fill it with the cooling medium.
- Allow the diffusion pump to warm up while monitoring the T/C gauges. The indicated pressure should remain essentially constant. If the roughing line pressure (per T/C #2) rises too high, close the foreline valve and open the roughing valve to restore the rough vacuum.
- When the diffusion pump is operating properly turn on the ion gauge and open the gate valve. The pressure as indicated on the ion gauge should drop as the diffusion pump begins to evacuate the chamber.

* To minimize oil contamination from the fore pump, O'Hanlon [1] recommends crossing over as quickly as possible at a pressure between 100 and 170 mTorr even though the diffusion pump may initially be overloaded at this pressure. This keeps the line well out of molecular flow where backstreaming will occur.

Letting Up to Atmosphere. The chamber may be returned to atmospheric pressure without cooling down the diffusion pump or emptying the cold trap. This is done by isolating the region between the diffusion pump outlet and the high vacuum gate valve. Improperly sequencing the valves during this procedure can result in the exposure of the hot pump oil and the cold trap to air which will degrade the oil and lead to the formation of ice on the trap. If this happens, the system will probably have to be shut down and cleaned.

- Close the gate valve.
- Ensure that the roughing line valve is closed. (The foreline valve will remain open.)
- Turn off T/C Gauge #2.
- Open the vent valve slowly.

Returning to High Vacuum. After doing whatever needs to be done in the chamber (changing samples, etc.), first ensure that the seals on the chamber are properly seated. When ready to begin the pumpdown cycle:

- Close the vent valve.
- Close the foreline valve. Keep an eye on T/C Gauge #1 for any significant pressure rise.
- Open the roughing valve and turn on T/C Gauge #1.
- The pressure in the chamber, as indicated by T/C Gauge #1 will decline to the crossover pressure of 100 mTorr.
- When the crossover pressure is reached, close the roughing valve and, as quickly as possible, open the foreline valve and then the high vacuum valve. The note (*) above applies here as well.

SYSTEM SIMPLIFICATION

After all of the above, a natural question is “Is there any way that a useful but simpler system may be assembled?” The answer is “yes” if you are willing to put up with long cycle times. This is not really an issue unless you need to access the vacuum chamber frequently as would be the case when making multiple evaporation runs, etc. For things such as particle accelerators where the apparatus will be kept at vacuum for extended periods, a straight through system would be entirely adequate.

The reduction of the system in Figure 1.26 to such a straight through configuration involves the elimination of the roughing line and the roughing and foreline valves. A gauge (T/C Gauge #1) should be kept in the foreline and the ion gauge will still be required as are the diffusion pump baffle and, optionally, the cold trap. The gate valve is not needed unless gas is to be introduced, whereupon this valve would be used as a throttle. In this instance, it would also be desirable to have a T/C gauge (or equivalent) appended to the chamber for monitoring the process pressure. Finally, the vent valve may be tied to either the chamber or to the foreline. The latter would permit a simpler valve to be used (e.g. as simple as a pinchcock) as there would be no issue with high vacuum compatibility.

REFERENCE

[1] John F. O'Hanlon, *A User's Guide to Vacuum Technology* (John Whiley & Sons, NY, 1980). The title tells it all; an excellent book for those who use vacuum systems but be prepared for a good dose of theory with all of the associated math. O'Hanlon is also strict in adherance to *SI* units, necessitating some head scratching for us Torr-heads.

A Simple Mini System for Evaporation

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This article was originally presented in Volume 1, Number 3.

The following is a description of a simple evaporation chamber that was developed to make small optical components. This system has proven adequate for the coating of small mirrors, depositing antireflection coatings, and - with a simple magnetically coupled motion feedthrough - fabricating graded neutral density filters.

The incentive for building this system came from the need for a linearly graded optical filter. Commercially manufactured filters were too expensive, but given the availability of a small vacuum system, making the required fixturing and a small chamber seemed to be a reasonable and affordable solution.

The vacuum system itself consists of a 2 inch CVC MCF61 diffusion pump. Backing this is a small (2 cfm) Leybold mechanical pump. An Edwards thermoelectric cold trap is used to prevent backstreaming of the oil, Inland perfluoropolyether. The thermoelectric element maintains the trap at about 0° C.

For the evaporation chamber, a small bell jar was improvised from a glass cover which came from a surplus aircraft gyro. The jar is about four inches in diameter and six inches high; walls are a quarter inch thick. The only marking on the jar is the manufacturer's name, Kearfott. The base plate was fabricated from half inch aluminum plate.

An O-ring, mounted in an aluminum split ring, seals the jar to the base plate. An alternative to the gyro cover could be the sort of covers used for electric meters or explosion proof lamps. As these vessels are not designed for vacuum use, care should be exercised. A shield (e.g. fabricated from plexiglas) should always be used with glass chambers, even ones designed for vacuum. Figure 1.27 shows the chamber with its associated fixturing. There are two feedthroughs. One is for a high voltage electrode which is used for outgassing during the pumpdown cycle. The other is a high current feedthrough for the "hot" lead to the source filament. Both of the feedthroughs are fabricated from standard stainless steel Cajon brand fittings: 1/4 inch male pipe thread to welded tube. For the high voltage feedthrough, a piece of 1/4 inch od glass capillary tubing is epoxied into the fitting. A piece of bare nickel wire is passed through the bore of the tube and is likewise sealed with epoxy. The high current feedthrough is made in a similar manner: a piece of 1/8 inch square copper bar is first coated with a thin coating of epoxy (where it will pass through the fitting); once the glue has set, the bar is inserted into the fitting and more epoxy is applied. An important point to remember in the fabrication of the feedthroughs is to confine the epoxy to the area above the threads: epoxy in the threaded area, which is subject to strain when the fitting is tightened, has a tendency to crack.

For providing uniform coatings, the item to be coated is suspended a short distance from the evaporation source, a coil of tungsten wire. The means for producing the variable graded filters is also shown in the illustration. Here the substrate (typically a microscope slide) is held by a string below sort of a windlass. At the end of the shaft is a magnet which provides coupling to a rotating steel bar (driven by a small gearmotor) outside the chamber. A baffle is placed in front of the substrate. Initially the substrate is in the low position, behind the baffle. Once the evaporation cycle has begun, the motor is activated and the substrate rises from behind the baffle. Thus the deposit will be thicker (more opaque) at the top and thinner at the bottom.

While a thermocouple gauge is incorporated in the system, its use has not been required as the high voltage outgassing discharge gives a good idea of what is going on in the chamber. During the pumpdown cycle the heater is also degassed. Once blackout of the discharge occurs the evaporation cycle may be started. It is important to quickly progress from the outgas phase to the start of the evaporation cycle.

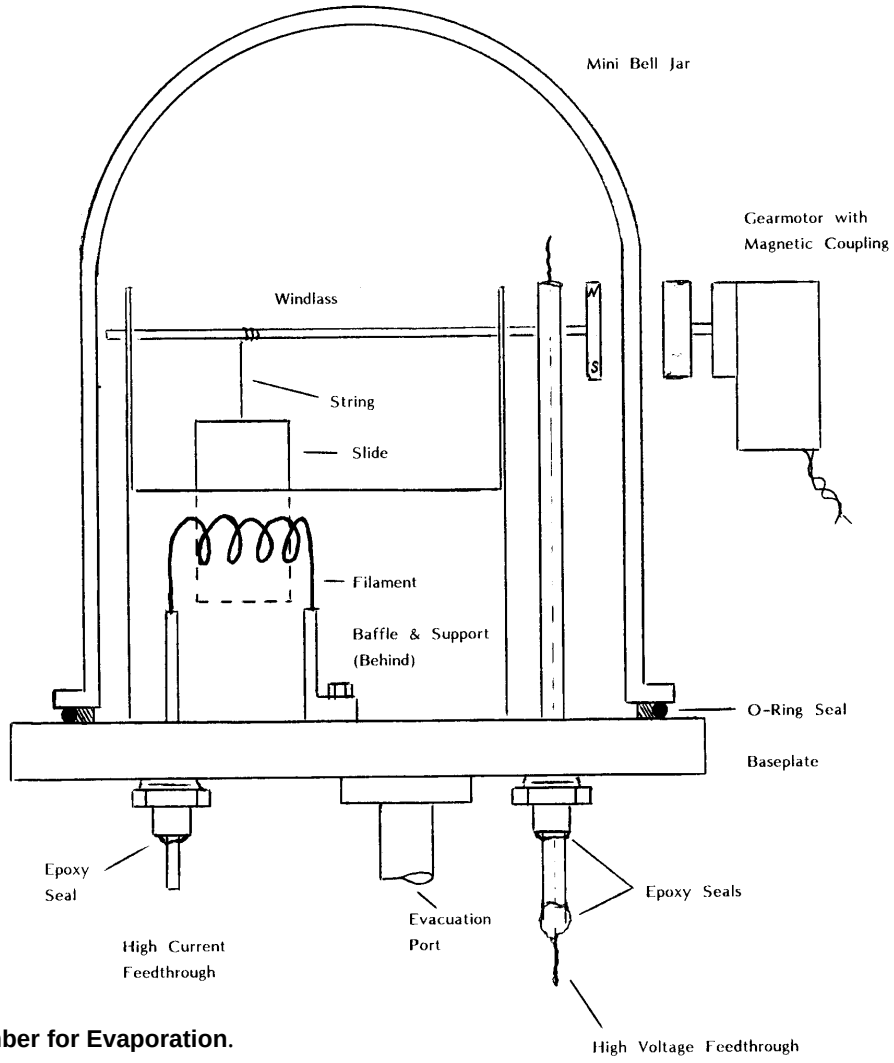

Figure 1.27 - Mini Chamber for Evaporation.

The Restoration of a High-Vacuum, Thin Film Deposition Machine

Alan K. Ward and John H. Moon

This article was originally presented in Volume 3, Number 4.

MOTIVATION

For a number of years there has been considerable interest in constructing astronomical telescopes here in Northern Ontario. Most home-made telescopes use a concave mirror to collect starlight which is then brought to a focus for a visual or photographic image. The optical demands on the mirror are considerable; it must have a paraboloidal surface accurate to 100 nm or so, it must have high reflectivity, and its shape must be temperature-stable.

The easiest way to meet these demands is to grind a concave shape in a thick disc of glass, polish and parabolize the glass, then coat the surface with a thin (50 nm) layer of aluminum. The layer of aluminum must be evenly deposited to

avoid altering the surface accuracy of the paraboloidal surface. This is accomplished by heating metallic aluminum in a high vacuum until it vaporizes, which then deposits on all surfaces in line of sight of the aluminum vapour source.

The effort to build our own vacuum coating facility began a year ago, after contemplating the 800 km drive required to aluminize our latest telescope mirrors. The aluminizing equipment, located in Southern Ontario, was available outside business hours on an ad-hoc basis and required that one of us accompany the mirrors.

We set out to build our own high vacuum deposition system. A small 2 inch oil diffusion pump and mechanical pump were appropriated from storage in an institution that had no more use for them, but it was soon learned that we would require a cold trap and a plate valve, which we had no easy means of acquiring.

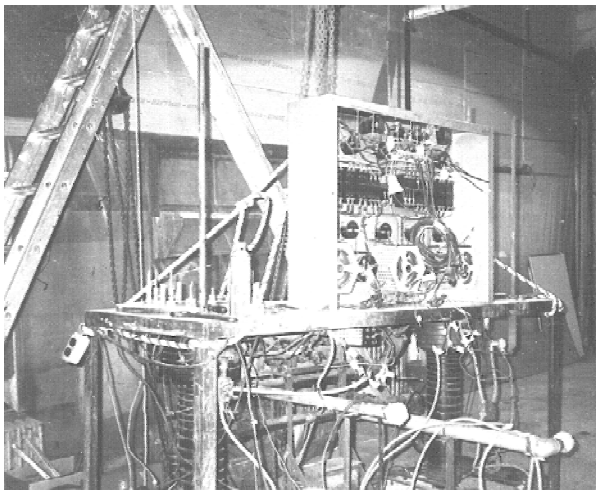

Figure 1.28 - The Machine 'As Received'

At this point a classified advertisement in one of the amateur astronomy magazines offered a large, dual bell jar coating machine for sale in New York City. A telephone inquiry confirmed that the machine had been used not only for metal film deposition, including aluminum, but also for the deposition of dielectric thin films, which are used for overcoating metallic films or as anti-reflection coatings on lenses. A deposit was sent to hold the machine until transportation arrangements could be made.

It was finally decided to enlist the help of an acquaintance with a truck. The three of us arrived in New York in mid-October 1993, pulling a flat-bed trailer to carry the machine. It would be inappropriate to include a reminiscence of our adventures with the inhabitants of the great city of New York, but much transpired to instill awe in three so far from the northern Ontario wilderness.

After some searching for mostly obscured address numbers, the former optical fabrication shop which housed the coating machine was located. The reaction to seeing the machine in its state at that time was, "Will it survive the trip home?", followed by, "If we can get it as far as the Canadian boarder, what is the rate of duty on rust?"

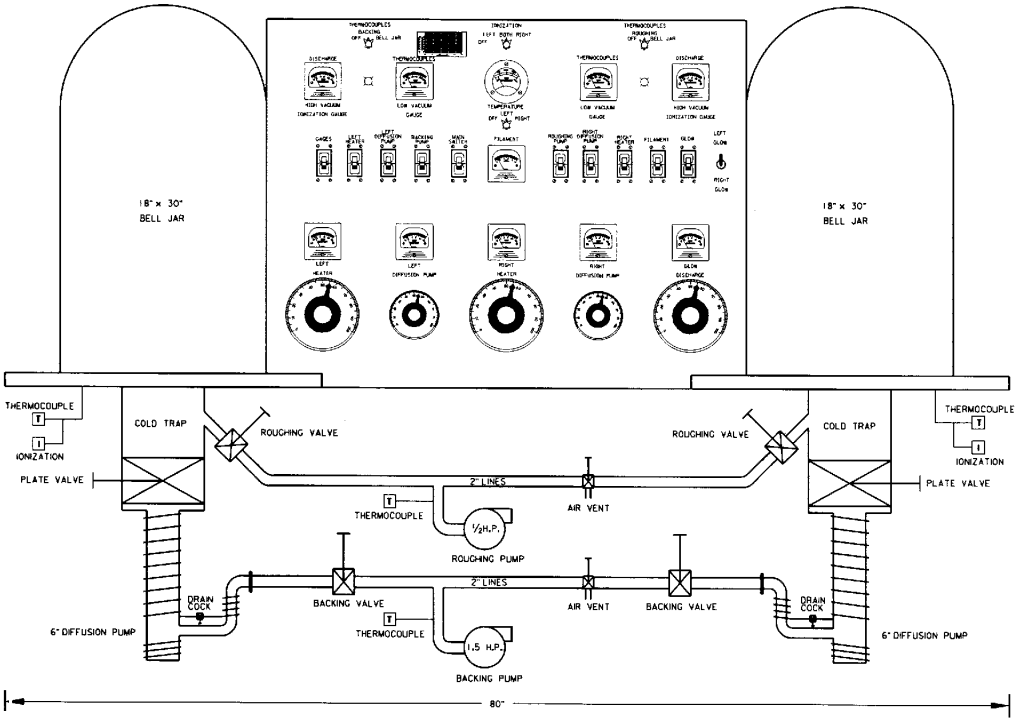

Figure 1.29 - System Schematic

However, the machine was loaded onto the trailer with a chain hoist, transported across the Canadian border very late at night, and safely unloaded at our optical shop in Sudbury. The long process of rebuilding the machine then commenced.

REFURBISHMENT (by Alan Ward)

"What did I get myself into?" was what went through my mind for a week after acquiring this machine. Anticipating our arrival from the states, my friends were not all too impressed with what we hauled in from out of the rain one late Sunday evening. It was hard not to laugh at it, for surely it looked like a mass of 'irrepairable damage.' I declared that in one year (!) the machine would be up and pumping. (With just one month left I can say that it's still on schedule).

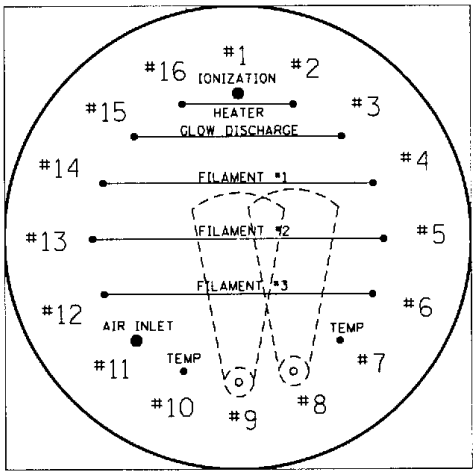

Figure 1.30 - Baseplate Layout

After being left to dry for a week, suspended from the ceiling on its chain hoist, the first job was to move the 2000 lb. monolith from its position of helplessness into John's work shop. Fortunately, John's room had a 10 foot ceiling which was required for the machine's bell jar pulley systems. No one figured it was possible to move the machine to where we wanted it. The idea of having to torch the machine in half was something to be avoided at all costs. To see, in fact, if it could be manouvered around and into the room, we made a framework from 2x4s to match the machine's dimensions. It went through, mind you, but with not a millimeter to spare.

Not knowing much about high vacuum systems at the time I figured I'd first start on some facet that I have some familiarity with. The control panel, being a disarray of wiring, was something I could begin with, knowing that it would keep me occupied while I perused some literature on high vacuum systems. Interestingly enough, it was at this point that I came across an ad in Sky & Telescope Magazine on 'A Newsletter On Nothing.' Without delay I had a money order in the mail to cover my subscription and all past issues. The "Bell Jar" articles were what really enhanced my understanding of vacuum basics and processes, and for that I have Steve Hansen of *t*BJ and the readership to thank.

For one week I traced out every wire (the ones that were still there) and connection to each device and drew up a wiring diagram. As some of you electrical buffs know, looking at a wiring diagram does not really tell you too much about how an electrical circuit works, so from the wiring information I created a control schematic diagram. From a control schematic, the logic of how a device or a piece of equipment works can be easily understood, even if there is lacking information from the wiring diagrams. From the control schematic I then constructed a new wiring diagram for reconstruction purposes. Once this was done I dismantled the panel into its components. The panel was then sanded, re-painted, and new meters and wiring was purchased and installed with new labels (Letraset). This was completed between November and the end of January.

After reading tBJ issues on diffusion pump basics, I felt that the next step might be to dismantle the two 6" diffusion pumps and investigate their integrity. I called John over because I didn't want to tackle this alone. We carefully un-bolted the pump from the so-called cold-trap (worrying that a bunch of springs and parts would pop out! Hey, don't laugh, we were new at this!) We then drained the contaminated 'black' oil from the pumps and lifted out the jets. I remember John saying "Al, is this all there is to it?" "I guess so" I replied.

The big job ahead, though, was to clean out the jets and replace the cracked tubing. My friend Randy Hoop, (who had also gone to New York with us) gave me a hand with this job. There is a little bit of black art involved in working with tubing as we have discovered. The first thing was to remove the old tubing that had been brazed on. I used a hammer and chisel to take care of that. Next, I ground off any residual welds on the pump to prepare for sandblasting. To retube, we used low grade 4% silver solder, and a high heat brazing torch to solder the tubing around the pump at 180 degree intervals. We also put a drain cock between the pump and the backing line outlet so that dirt and scaling won't accumulate there over time.

The cold-traps were next. We un-bolted the trap from the bell jar base. Once the bolts were off, the trap wouldn't let go. We had put a stool underneath so that it wouldn't come crashing down onto the floor. We tried pounding it out but nothing would give. Applying heat using a propane torch was the only way to break the seal. The trap was in a real mess. The baffles were all black with soot, the plate valve was broken and the tubing inside was zinc plated copper tubing

(!???) that was showing signs of peeling. We didn't want this exposed in the trap so we replaced the peeling copper with stainless tubing that was welded to the trap body which is made of 10 inch steel pipe. We had to bring the traps to a welder to weld the stainless to the outside wall of the trap. We used #309 rod for welding stainless to steel.

Literally every bolt on this machine has now been replaced with stainless. Protective parents would not allow their small children to visit when the mild steel hardware was being removed with chisels and sledges. A good majority of the bolts had to be either ground or hacked off. I'm sure this gives you an idea of the state that this piece of filth was in when I began refurbishing.

New valve seats were purchased and new gaskets were used in joining the lines together. New air and water valves were purchased along with filters. Refer to the diagram that shows the process schematic of the vacuum lines and valving on the machine.

The feedthroughs were next on the list. The insulators were made from a porcelain or ceramic material, and half of them were cracked. They required replacement with something, but with what? I couldn't find anything in ceramic or porcelain that would suffice. I then came up with the idea of using ultra-high molecular weight (UHMW) polyethylene for the insulators. UHMW poly, while not a high temperature material, has very good outgassing characteristics and it is fairly inexpensive. Acquiring some 1-1/2" rod material, I had it machined to make 32 insulators. Figure 1.30 shows the feedthrough arrangements.

A secret that we would like to share with all you vacuum buffs when it comes to cleaning your feedthroughs, holders, baffles, baseplates etc. - use 'AUTOSOL' - a metal polish that does wonders. It removes metal oxides and imparts a shine to brass, stainless, and aluminum unlike anything you've seen before. We have already used 5 tubes, and it's unbelievable! After using, be sure to wipe with alcohol to remove any films. (John and I haven't witnessed any outgassing problems from using this agent on another 12" coating machine we've acquired recently).

Figure 1.31 - The Refurbished System - Alan Ward at Left with John Moon. (Note the attractive *Bell Jar* T-shirts.) Photo by Robert J. Staple.

CURRENT EFFORTS

We are now checking the integrity of the mechanical pumps to see what may have to be done with them. The machine is now basically re-assembled and once power is connected we'll be checking for leaks.

Once the machine will pull a good vacuum using only tap water for cooling, the next objective will be to reduce the water bill by using a closed-loop cooling system. I obtained a 21 cubic ft. deep freeze and plan to fill it with antifreeze, cool it to -25 degrees Celsius, and pump antifreeze through the system. Has anyone experimented with this idea? Please let us know.

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John Moon may be reached at Site 10 Box 7, Skead, Ontario, Canada P0M 2Y0

Adding Instrumentation to a High Vacuum Deposition Station - A Student Project at Cambrian College

H. John Earle, Jean-Guy Imbault, Alan Ward and John H. Moon

This article was originally presented in Volume 5, Number 1.

INTRODUCTION (John H. Moon)

As was described in the previous article, Alan Ward in October 1993 purchased a used, high-vacuum, dual chamber thin film deposition station. It required about one year to refurbish the machine and deposit our first thin film of aluminum. By October 1994, it was clear to us that the machine would be more useful if it enjoyed better instrumentation. In particular, we wanted a means of measuring temperature at various points on the diffusion pumps, improved gauges for both high vacuum and roughing/backing vacuum, and a more accurate measurement of the current used to heat the filament or boat which evaporates the coating materials. In addition, we felt that it would be prudent to archive these data for diagnostic purposes on an IBM compatible computer. We decided that all data collected would be standardized to a 0 to +5 volt analog level. An A-D converter would then be part of the computer interface card, which we intended to purchase.

In 1994, I was on the faculty at Cambrian College in Sudbury, Ontario and had students who would be graduating in May 1995 in Electronic Engineering Technology. As a requirement for graduation, all students must complete a project involving electronics in their final semester. Alan and I felt that our requirements for instrumentation would also be about the right level of difficulty for student projects. I outlined the project possibilities to my class in the fall of 1994. Two students accepted the challenge. The results are described below.

The project approach worked well for all involved. Alan and I have a computer-interfaced deposition station; the students learned about high vacuum techniques and about how to apply electronics to a real machine. For grading, each student was responsible to a committee of three faculty members. They each had to submit a paper and prepare a presentation/ demonstration at the end of the semester. Each student received an "A."

Ordinarily final projects are done "in house" at Cambrian College, but these projects, completed off campus, provided students with a much different perspective than they might have had sitting at a workbench at the College. Readers who could use help in electronic interfacing might explore the possibility of making use of community college students who perhaps right now are trying to come up with an interesting project to satisfy a graduation requirement.

PROJECT ASSIGNMENT #1 (Jean-Guy Imbault)

My part in the project was to design two circuits and, with my partner, to write some software that would graph the various processes that occur during the system's pump down cycle. Circuits were needed to monitor diffusion pump temperature and evaporation filament current during evaporation. All circuits were required to provide an analog output that would be compatible with a 0-5 volt interface.

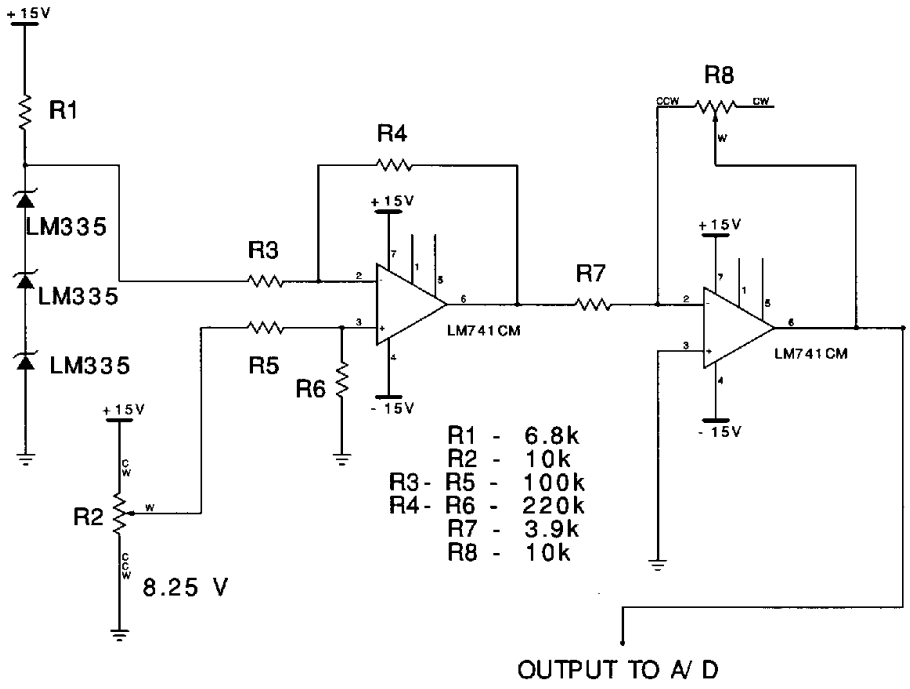


Figure 1.32 - Temperature Sensing Circuit

Diffusion Pump Temperature Sensing Circuit

For optimum performance, the 6-inch diffusion pumps require a temperature differential of 10 to 15° C between the inlet and backing line outlet. To adjust for cooling, a manually operated needle valve is used to vary the coolant (water) flow rate. Figure 1.32 is a schematic of one of the temperature sensing circuits. To monitor the differential temperature, two identical circuits were used.

Circuit Operation:

The LM335 is an easily calibrated, integrated circuit which sells for about $5.50 Cdn. The reason for using three sensors was to get an average temperature of both areas being monitored and to provide a better resolution for the A/D converter. Using an 8 bit A/D, the voltage required was calculated as follows:

$(5 / 2^8 - 1) \times (5 / 255) = 19.6mV$

Since the LM335 produces only 10mV/ °C it would take a change of 2° C (20mV) to show a change in temperature on the A/D. By using three sensors in series a 30mV/°C was obtained giving a more accurate temperature readout. R1, which is in series with the three sensors, sets the operating current for the sensors. R2 is used to adjust the sensing range of the LM335 which in this case was 0 to 50° C. The potentiometer was set to the voltage level of the sensors at 0 deg Celsius which was 8.25V. R3 & R4 sets the first stage of amplification of the sensor voltage which was 30mV/°C. R5 & R6, which are equal in value to R3 & R4, serve to balance the inputs of the op-amp. The signal leaving pin 12 was then passed through the second stage of the amplifying process. R8 is used to adjust the output to give 0 to 5V for the A/D converter.

Filament Current Monitoring Circuit

To ensure that the system could produce coatings with consistency, Alan wanted a means of monitoring the filament current. To accomplish this, I used a current transformer to monitor the primary current of a 2kVA low-tension transformer that supplies up to ~400A at 5 volts ac to the evaporative heaters used inside the bell jar. A precision rectifier, as shown in Figure 1.33, was used for this task.

Circuit Operation:

U1 is a 741 used in a unity gain configuration which has a high input impedance so as not to affect the shunt across the current transformer. The small signal from U1 is then rectified using U2 and U3. At the output of U3 the signal is now inverted and rectified (pulsating DC, a sine wave with the top flipped down). A resistor pack was used in the rectifying process to keep the signal as accurate as possible. The signal was then amplified through U4 and filtered at the output to get 0 to 5V which represented 0 to 15A. The potentiometer R4, was to fine tune the output to be filtered and R5 was used to smooth the signal to be read by the A/D.

PROJECT ASSIGNMENT #2 (H. John Earle)

This part of the project consisted of two tasks. The first was to modify the existing cold cathode ionization gauge circuitry to provide a 0-5V DC analog signal. The second was to co-write a data acquisition program in C to access serial data via an A-D converter.

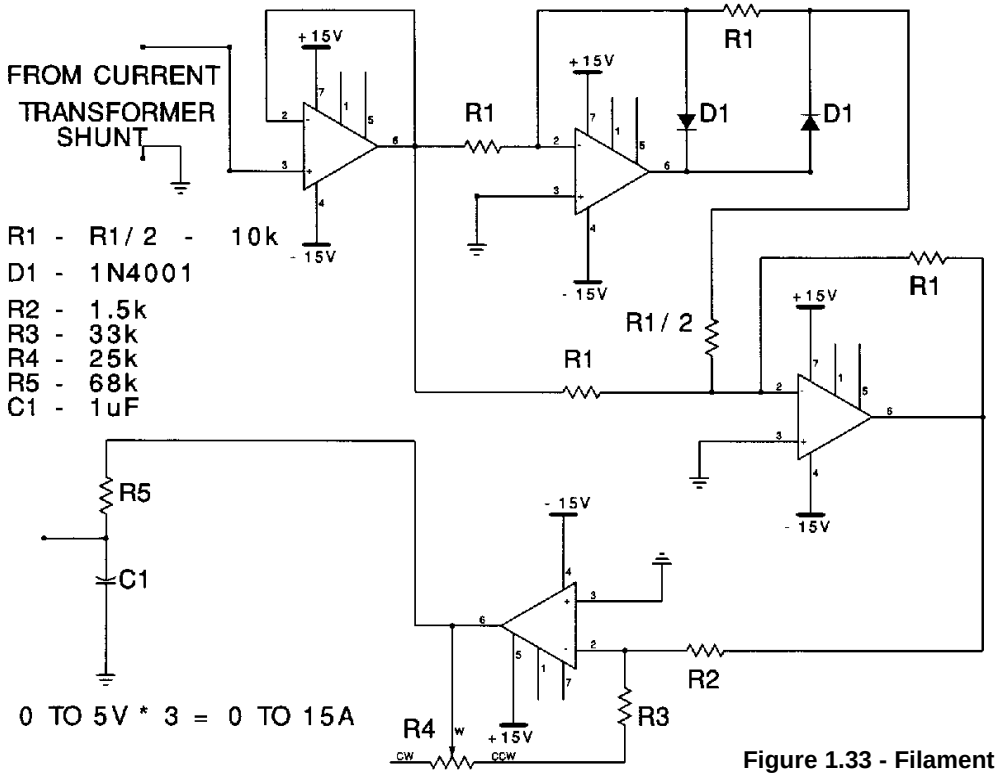


Figure 1.33 - Filament Current Monitoring Circuit

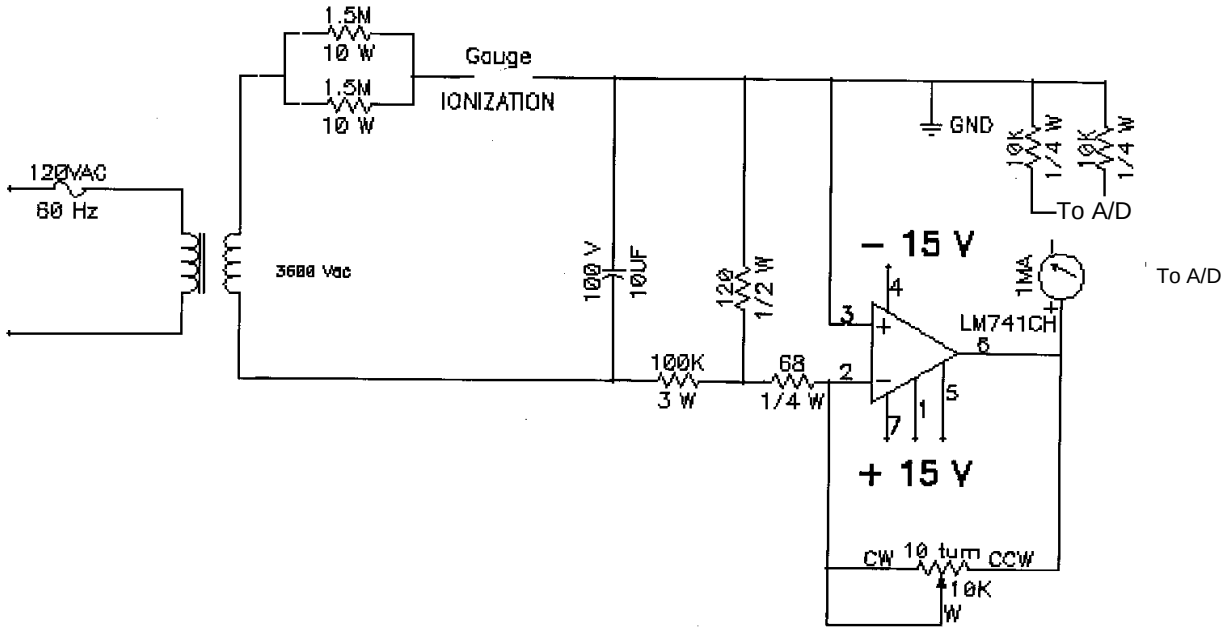


Figure 1.34 - Cold Cathode Ion Gauge Circuit

Ionization Gauge Circuit

The circuit diagram of Figure 1.34 shows the ion gauge controller circuitry with the modifications required to interface the cold cathode ionization gauge to the analog to digital converter.

The transformer is a step-up transformer which converts the line voltage to 3800 volts. The high voltage is required to initiate the ionization process. Ac is delivered to the ion gauge tube as the tube is self-rectifying. When the voltage at the anode becomes negative with respect to the cathode, a reverse action is initiated but is limited because the surface area of the anode is very small as compared to the cathode. This results in the gauge electrically acting like a rectifier.

Once ionization occurs, the voltage drop across the ionization gauge is lowered to approximately 2950 volts. The remainder of voltage is dissipated by the 1.5 M and 100 k resistors. The 10 μF capacitor filters the halfwave rectified waveform, smoothing the dc current. The 741 op-amp is used to provide drive current for the 1 mA ammeter and the voltage reference resistors. The 10 k potentiometer provides a calibration adjustment for the ammeter. The two 10 k resistors in parallel provide a 0 to 5 V reference for the analog to digital converter.

The op-amp is used in this circuit to provide an approximate no load appearance to the ionization circuit so as to reduce its effect on the circuit. A printed circuit board was made using the toner transfer method of etching PCBs. The circuit was then tested and calibrated against a calibrated ionization gauge with excellent results.

Interfacing Analog Signals Using the Serial Port

All of the measuring devices for the deposition station were redesigned to provide a 0-5 volts dc analog signal for interfacing purposes.

Interfacing:

The first step in interfacing an analog signal is to change it to a digital signal. This is accomplished by using an analog to digital converter. Common A/D converters require an analog signal that varies between 0 to 5 volts to produce a digital word. The word then must be sent one bit at a time into a serial port of a computer to be accessed by a program.

A circuit board can be designed or purchased for this purpose. In our case we chose to purchase the following interfacing components from Electronic Energy Control Inc., 380 S Fifth St. STE 604, Columbus, Ohio 43215-5491, tel: (614)464-4470, fax: (614)464-9656.

The price for the analog to digital converter (model ADC-16) was $99.95. Additional components included a terminal block (RCT-16) at $22.95 and a serial cable (CC-DE9S) at $8.95. The interface has a power requirement of 9-14 vdc at 300 mA. We found that this interface has many useful features that allow further expansion. Standard features included: 16 analog inputs (expandable to 32 analog inputs or 128 status inputs), 50 - 19,200 baud, RS-232 interface and capable of controlling 112 relays with an optional relay expansion port. The ADC-16 interface comes with some ready to use software that can be used with MSDOS or Windows. This can be useful in checking to see if everything is functioning correctly. The interface also comes with a booklet that documents how to access the interface in BASIC and C.

ADDITIONAL FEATURES & WORK IN PROGRESS (Alan Ward)

Aside from the above-mentioned circuits, work on two other circuits was initiated: a vacuum thermocouple controller and a thin film monitor. The first is used to monitor rough vacuum in the backing line and in the chamber; the latter is used to measure the thicknesses of the deposited films. Despite considerable effort spent on these, they still do not function as well as we had hoped. Descriptions of these are provided in the next two sections. Work is continuing on these and we will deliver a final report when we have produced satisfactory solutions.

Thermocouple Gauges

As noted, there are two T/C gauges: one is in the backing line, the other is connected to the chamber. The gauge circuits provide a current output. To provide a pressure reading, the currents are cross referenced to a look-up table. This function is done automatically in the computer. When I don't have a computer available, I can still rely on panel meter current readouts and a printed cross-reference to pressure.

While the circuits are functioning, the T/C pressure measurement circuits represent unfinished work. Jean-Guy had designed a thermocouple controller to work with a commercial grade gauge tube (KJL 6000). The circuits do work well, although they are not described in detail here because of the hard to obtain components needed to reproduce them. Jean-Guy is re-developing the circuit so that easy to obtain components can be used.

Thin Film Deposition Monitor

A crude thin film monitor has been built. It uses a an exposed oscillator crystal that changes frequency when a film is deposited on the crystal. The amount of shift can be related to the film thickness. While it does work, the crystal circuit is heat sensitive and requires further development to eliminate its heating effects. At this time it simply gives a 0 to 5 volt display which is then converted to a mass and thickness determination via a pocket calculator.

INTEGRATION (Alan Ward)

Figure 1.35 is a screen capture image of the output of the completed monitoring system. The image shows the output from the circuits noted above plus some other data. Starting with the tabulated data at the top of the screen, the outputs of two thermocouple gauges are shown: the backing line "BKG", and the chamber "T/C Current". The numerical figure is the unconverted analog current reading from the gauge circuit in mA.

Other displayed data include the diffusion pump coolant temperatures, ion gauge current, evaporator filament current, and manually inputted data including room temperature and humidity. The film thickness monitor output is displayed as a bar graph at the right of the screen.

The main part of the screen is a graphical output of the T/C and ion gauge pressure readings (converted to Torr) and the filament current. It is instructive to look at the gauge readings during the process cycle to see what's happening to the pressure in the chamber.

With regard to the two thermocouple gauges, the plot showing the higher pressure reading is the backing line monitor. Once the vacuum in the bell jar reaches the highest obtainable vacuum, via the rotary-vane roughing pump, the plate valve is then opened. At this point the chamber thermocouple gauge is turned off and the ion gauge is turned on. This explains why the chamber T/C plot drops to bottom at this point. The ion gauge then continues to measure

down to the system's ultimate vacuum. The backing line thermocouple gauge continues to be monitored during the entire process.
On the ion gauge plot, note the pressure spikes caused by degassing when current is applied to the filament.

To enhance the reflectivity of aluminum mirrors, I evaporate a $\lambda/4$ coating of MgF_2 followed by $\lambda/4$ film of ZnS. Degassing of ZnS is usually done during the pumpdown cycle. ZnS in its off-the-shelf form contains a considerable amount of adsorbed water. This must be vacuum baked at 900 °C to sinter it before evaporation can commence. The plot indicates a sudden pressure rise when the ZnS is baked. Normally it takes about 1 hour to completely sinter the ZnS. However here the pressure dropped back down very quickly. In this case, the material I was working with was already sintered and required little baking to thoroughly degas it, as is shown by the plot.

The sudden rise in pressure that was encountered when aluminizing took place was a sudden outgas of adsorbed water vapour off the walls of the chamber due to the heating effect from the filaments. These effects should be carefully monitored so that contaminated deposits of aluminum oxide do not form.

CONCLUSION (Alan Ward)

The students' contributions to this project have made a significant impact to the machine's capabilities. The new instrumentation will now make it possible to perform other more elaborate thin film processes which were not before feasible.

The project venture also was financially beneficial to everyone involved. Usually, students invest in their own projects. For this project, the students were exempt from all expenses except for those that were related to their commute. Approximately $600.00 Cdn. was invested in the project for materials and components. I knew that the return would far outweigh the expenses. If I had to purchase off the shelf' instruments commercially, I wouldn't want to even begin to imagine the cost. It is without a doubt that the cost would be well over the budget for the average amateur vacuum enthusiast.

Efforts in promoting vacuum technology among engineering technology students have also continued. A project scope outline is already in the works for next years' graduating students and we look forward to presenting it to the classes come this fall!

I have interesting plans in expanding the process capabilities of the machine in the future. Along with adding to the instrumentation, I would also like to do some analytical research utilizing thin films. An interesting thesis could be in store for graduating chemists and physicists. Lately, I've been feeling out this avenue with our local university.

(Please address any correspondence concerning this project to Alan Ward, 784 Churchill Ave., Sudbury, Ontario, P3A 3Z9, Canada; phone: (705) 675-2760).

The editor has received periodic updates on the evaporator and these have been included in more recent issues of the Bell Jar.

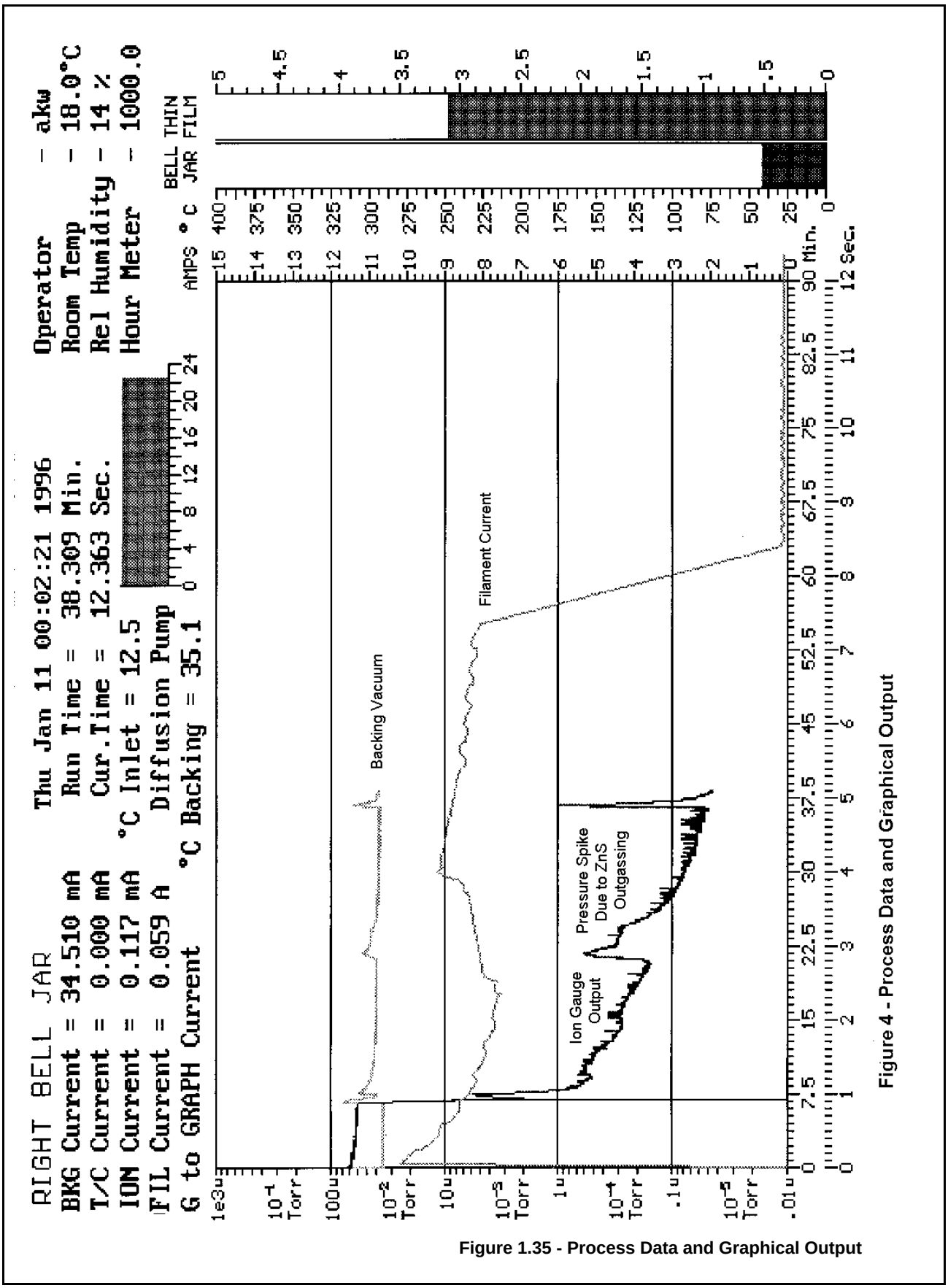


Figure 4 - Process Data and Graphical Output

Figure 1.35 - Process Data and Graphical Output

Part 2: Vacuum Components and Bits & Pieces

Refrigeration Service Vacuum Pumps

A couple simple modifications to make these inexpensive pumps more suitable for the vacuum experimenter.

Steve Hansen

I. INTRODUCTION

Some of the vacuum pumps used in the refrigeration service trade are well suited to the purposes of the vacuum experimenter and educator. These pumps may be obtained at relatively low cost, they have good vacuum capabilities, are fairly rugged and offer many features of industrial vacuum pumps. Normally these pumps are used by service technicians when recharging refrigeration systems. They should not be confused with the vacuum pumps that are incorporated within refrigeration systems.

Two such pumps have been evaluated. 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 somewhat lower prices may be had on occasion when the dealer has made a volume purchase agreement with the manufacturer.

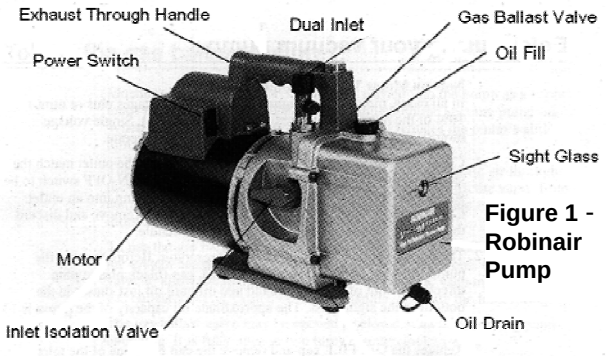


Figure 1 - Robinair Pump

Figure 1 shows the Robinair pump. It is a direct drive pump and incorporates a dual flare fitting on the inlet, an inlet shut-off valve and gas ballast. The oil drain is conveniently located and the exhaust is directed through the handle. The J/B pump is very similar in appearance and features. Both pumps have factory vacuum ratings of 20 to 25 mTorr.

As is, these pumps have one major limitation. That is the vacuum inlet and its compatibility with vacuum hoses of reasonable conductivity. This article will present a solution to that problem. Also detailed are a simple exhaust filter and some performance attributes.

II. INLET MODIFICATION

While these pumps have reasonably good throughput at the inlet, the hoses which are compatible with the flare fittings are of fairly small inside diameter (the ¼" id hoses actually measure about 3/16") and they are therefore quite effective at choking the pump. Why the manufacturers supply such skinny tubing is a mystery to me. The service tech undoubtedly feels that he is doing a great job because he has a high capacity 2-stage pump and the gauge, which is usually attached to the pump inlet, will read a nice high vacuum. Of course, the system being evacuated, which is what the tech should be caring about, is undoubtedly at a much higher pressure with the innards evolving water vapor like crazy. A constructive exercise would be to compare the conduction characteristics of a standard refrigeration hose (3 or 6 foot length, 3/16" id) to something more suitable in a small laboratory setup (1 foot length of 5/8" id tubing). Conductance equations are found in just about all vacuum texts as well as in the Kurt J. Lesker Co. catalog. Some material on gas flow calculations may also be found in the first section of this booklet.

If you don't want to bother with the look-up, just remember that, in viscous flow and with all other factors being equal, conductance varies with the diameter of the tube to the fourth power. For the tubes we are comparing, the difference in conductivities (again, same pressure, same length) amounts to a factor of about 123.

Figure 2 shows how a small system with a primary pump should **not** be organized along with the resultant effects. The hose should be short and wide and the gauge should be located on (or at least near) the experiment chamber.

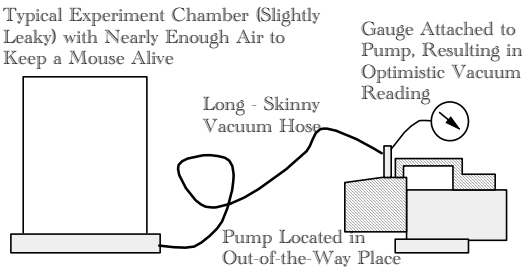


Figure 2 - How not to Connect a System

Enough preaching. Figure 3 shows a simple fitting that can be added to the stock pump to permit the attachment of standard 5/8" id reinforced PVC or rubber tubing. The main components are standard brass fittings

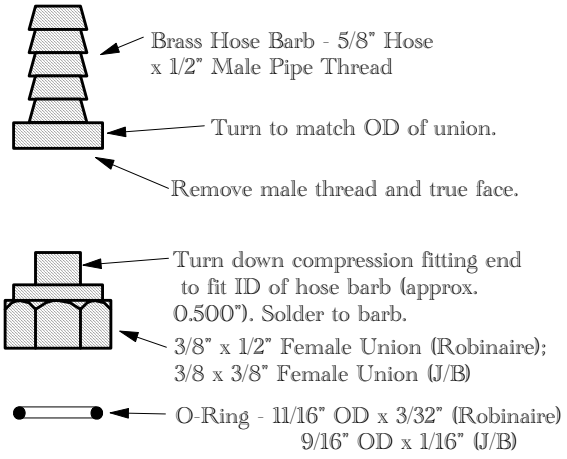


Figure 3 - Inlet Fitting

that are available from any well stocked hardware or plumbing supply store. The required lathe work is non-critical. After turning, join the two pieces with silver-tin solder.

The fitting goes on the top (larger) inlet port on the pump. In the case of the Robinaire, this is a 1/2" flare fitting. The J/B has a 3/8" fitting. With the O-rings, there is no need to really crank these fittings onto the pump. A gentle wrench tightening is all that's needed. The O-rings are from the hardware store's faucet fix-it section. Corresponding Moen part numbers are 14611 (1/2" fitting) and 14510 (3/8" fitting).

Each pump has a 1/4" side-arm fitting with an O-ring sealed cap. This is useful as a vent.

III. AN EXHAUST OIL FILTER

Any mechanical pump will produce a fine mist of oil during the initial pumpdown. Subscriber Jack Herron noted that a standard auto oil filter may be used to reduce the oil mist coming from the pump.

Figure 4 shows my adaptation for the Robinaire pump. This particular filter, the Fram PH3387A, has threads that match the threads on the compression fitting end of a 1/2" brass compression fitting. All fittings are 3/8" pipe thread. As it turns out, the bore of the Robinair handle is the same as the diameter of a 3/8" brass pipe nipple with a slight taper. A 1-1/2" nipple fits snugly into the handle. Check the fit first and then smear a bit of silicone caulk on the nipple. Let the silicone cure before using the pump.

The air flow through the filter is opposite to that of the normal oil flow in the automobile application. The one way rubber flapper valve in the filter is accessible with a sharp tool and must be suitably disrupted to defeat the one-way action. If this is not done, pressure will build up in the pump and a flying oil filter could result. I would recomment that, after modification, the filter be marked to indicate that it has been so modified.

The J/B pump has a galvanized steel pipe handle that is threaded with a standard 1/2" pipe thread. To add a filter to this pump, simply substitute 1/2" fittings. The nipple is not needed.

IV. OTHER COMMENTS

For some reason, the J/B pump is noticeably quieter than the Robinair pump. If noise level is a concern (e.g. in a classroom setting) and the throughput difference is not an issue, the J/B might be preferable.

Dr. Bruce Kendall of State College, PA tested both pumps with a calibrated gauge connected directly to the input ports. His measurements indicated an ultimate vacuum in the range of 15 microns. He also compared the 4 CFM Robinaire with a 6 CFM model and found the larger pump to be quieter.

Additionally, by changing the oil from the Robinair brand supplied with the pump to a standard commercial hydrocarbon oil, he was able to about halve the ultimate vacuum.

Note: I recently tested a 1998 vintage Robinair pump and found it to be considerably quieter.

Originally published in Volume 4, Number 1.

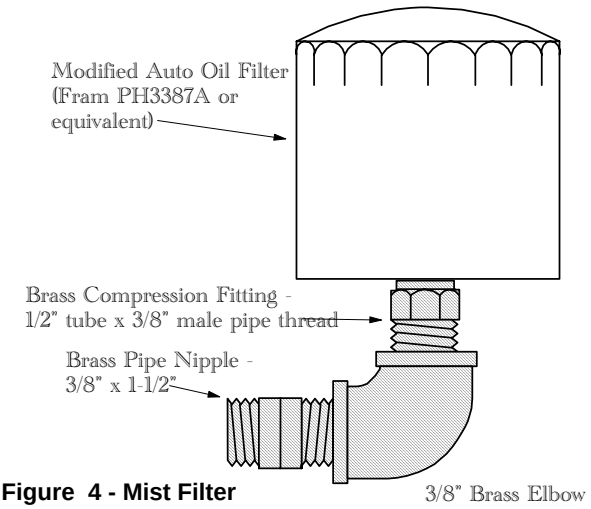


Figure 4 - Mist Filter

The Aerobic Workout Vacuum Pump

A Piece of Rubber Tubing, a Pair of Ink Brayers and Some Sweat are Used to Evacuate a Discharge Tube

This simplest of pumps was originally suggested by Nyle A. Steiner in his contribution to *The Amateur Scientist* column of Scientific American, August, 1966. The pump, whose action is identical to that of the peristaltic pumps used in biological and hospital applications (e.g. for transfusions), may be thought of as a linear version of the standard rotary vacuum pump.

The heart of the pump is nothing more than a length of flexible rubber tubing that is supported on a wooden base. The pumping action is achieved by the use of a pair of ink brayers, one in each of the operator's hands. Figure 1 shows the general layout of the pump. A piece of flexible tubing about 2 feet long is fixed on a plank with the ends held by metal clips or staples. The end toward the operator is the exhaust, the other end is the inlet. To pump, place one brayer at the far end and, while pressing, pull the brayer toward you. When the brayer has traveled the full length of the tube, place the second brayer in the start position. Only lift the near brayer when you have started the second stroke. Keep this pleasant action up until you have achieved the desired vacuum.

The tubing needs to represent a compromise between softness (unless you have the arms of Arnold Schwarzenegger) and ability to avoid collapse under vacuum. Steiner used 3/16" id x 3/8" od latex surgical tubing. This seems to be pretty good. I've tried 1/4" x 3/8" silicone rubber tubing which is too soft. Larger bores also pose difficulty in maintaining a seal. To keep the brayer parallel to the board, a small raised strip (about 1/8" high) is required. This helps to maintain even pressure across the tube and also helps to keep the rubber tube from wandering. Place this strip about 2 inches from the tube.

Don't get any ideas about being able to evacuate even small chambers with this technique. However, with a very tight system (leaks are killers with this pump) it is possible to light a small discharge tube.

Invariably, after a minute or two of stroking, you'll slip and all the air that you so diligently pumped out will rush back into the tube. Steiner developed a clever stratagem whereby the tube is converted into a two-stage pump. This is done by pumping until no improvement is noted and then, with the roller nearest to the operator still pressing the tube, placing a pinch-clamp on the tube just behind the roller. This seals the pump from the atmosphere. Pumping is then resumed except that the stroke is shortened. Now the pump is "evacuating" into a low pressure closed volume. If the entire system is leak-free, further pumping will lower the pressure well below that which is achievable by pumping into the atmosphere. By doing this I've been able to produce a nicely differentiated glow discharge showing the various dark and bright regions.

Steiner was actually able to achieve a "dark" discharge condition with the pump exhausting into a refrigeration compressor. The latter can achieve about 1 Torr. The hand pump was able to reduce the pressure another 1000 fold. I will take his word on this as I have not attempted it.

Originally published in Volume 4, Number 2

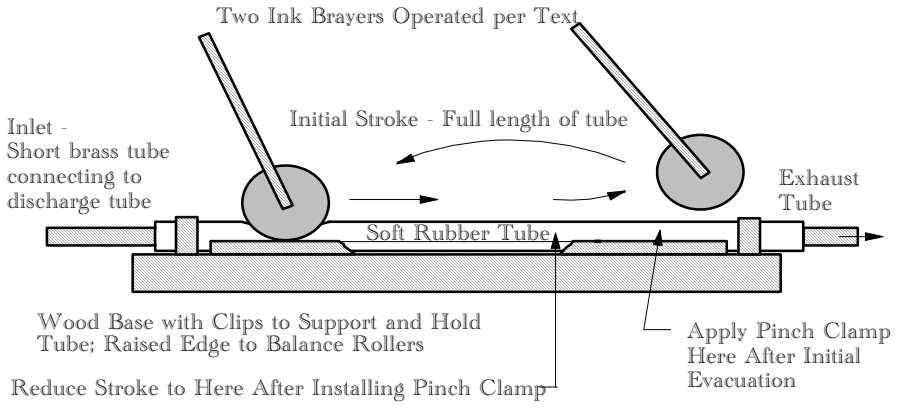


Figure 1 - Hand Operated Vacuum Pump

A Homebuilt Thermocouple Gauge Controller

I. INTRODUCTION

The thermocouple (or T/C) gauge is one of the more common and cost effective gauges for vacuum pressure measurement in the 1 Torr to 1 milliTorr range. The T/C is usually found in the forelines of high vacuum systems (i.e. between the roughing and diffusion pumps) as well as in single pump systems of the sort used to evacuate sign tubes.

Like most vacuum gauges, the T/C gauge does not measure pressure directly as do, for example, manometers of the McLeod or Bourdon type. Instead, these vacuum gauges depend on changes of a physical characteristic of the residual gas within the gauge tube. In the case of the T/C gauge, and all other thermal conduction gauges, that characteristic is the thermal conductivity of the gas.

A thermal conduction gauge may be thought of as a defective vacuum insulated thermos bottle (Figure 1). Each has a hot element (coffee for one, a filament in the case of the other) within a vacuum wall. There are two ways of removing heat: conduction (molecule to molecule) and radiation. For both coffee and warm filaments the primary path at atmospheric pressure is conduction. As it turns out, the thermal conductivity of air is nearly constant down to a fairly low pressure - about 1 Torr. Then it begins to change rather linearly with pressure down to a value of about 1 mTorr, whereupon conduction through the gas ceases to be a major factor. At that point, the dominant loss factors are conduction through wall and leads, and radiation. What might be surprising to many people is that a fairly good vacuum is needed in a thermos. With a bit higher pressure, you might as well have no vacuum. In the case of the thermal conduction gauge, operation will only occur within the sloped portion of the curve. An interesting experiment would be to nick open a thermos bottle refill and measure the cool-off rates for hot water with the bottle evacuated to a number of pressures. The result would be a useful, but very slow, thermal conduction gauge.

The T/C gauge contains two elements: a heater (filament) and a thermocouple junction which contacts the filament. With the filament current held constant, as the pressure within the tube is decreased the filament will become hotter because of the improved thermal insulation provided by the increasingly rarefied gas. This temperature is sensed by the thermocouple junction. Measurement is accomplished by reading the thermocouple junction voltage on a sensitive meter which has previously been calibrated against a manometer. Simple T/C gauges may be obtained from a variety of sources such as Duniway Stockroom or Kurt J. Lesker Co. These consist of the gauge tube itself, a power supply for the filament, and moving coil (d'Arsonval) meter for displaying the pressure. Tubes usually have a 1/8" male pipe thread for coupling to the vacuum line and an octal (vacuum tube) base for mating with a socket. In newer gauges, the power supply is usually nothing more than a plug-in type ac adapter with a potentiometer for adjusting the current. Each type of T/C tube has its own calibration curve. Also, as there are some structural variations from tube to tube within a type, each has its own filament current rating. The current at which the gauge will conform to the calibration curve is imprinted on each tube. Also, T/C gauges are calibrated for air. As different gases have varying thermal conductivities, the gauge will not be accurate when working with, for example, argon or carbon dioxide.

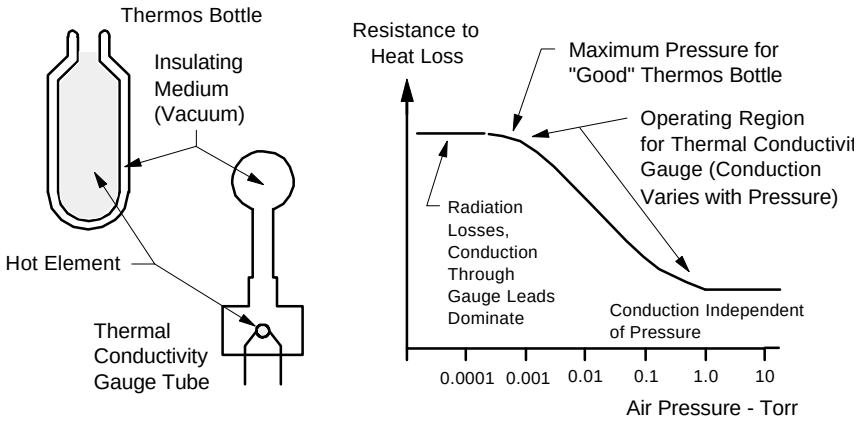


Figure 1 - Thermal Conduction in Gauges and Thermos Bottles

II. MAKING YOUR OWN GAUGE CONTROLLER

As was previously noted, complete basic T/C gauges are available from a variety of suppliers. Typical prices are in the $200 to $250 range, new. Given the basic simplicity of a T/C gauge, building one from available components would not seem to be difficult. To look at the price breakdown of a simple system I'll pick on a commercial gauge that is available through Duniway Stockroom Corp. This gauge is based on the 531 tube (stock number TCG-531/BOX). The complete gauge is $239. As the meter, tube, power supply, and box are available separately some idea may be obtained as to the cost split in the unit. The 1993 catalog lists the tube at $40, the meter at $59, the power supply unit at $115 and the metal box at $50. This gives a total cost for the separate components of $273. As is usually the case, one pays more for the separate components than for an integrated unit. However, it is instructive to look at how the costs are spread. In terms of building one's own gauge, the power unit and box are the easy targets; particularly so since the commercial power supply does not include the filament current meter - it's assumed that you have a multimeter with which to adjust the setting. Thus, for the supply just use a surplus ac adapter (rating not critical), a good wirewound pot (the 10-turn ones can be gotten at low cost through surplus outlets like Fair Radio Sales - 1k is an appropriate value), and a milliamp meter. If all of these have to be purchased, the cost (assuming surplus) should not exceed twelve dollars or so. A classy surplus box to put the stuff in should not go much over a few dollars. But, by all means, do make it look nice.

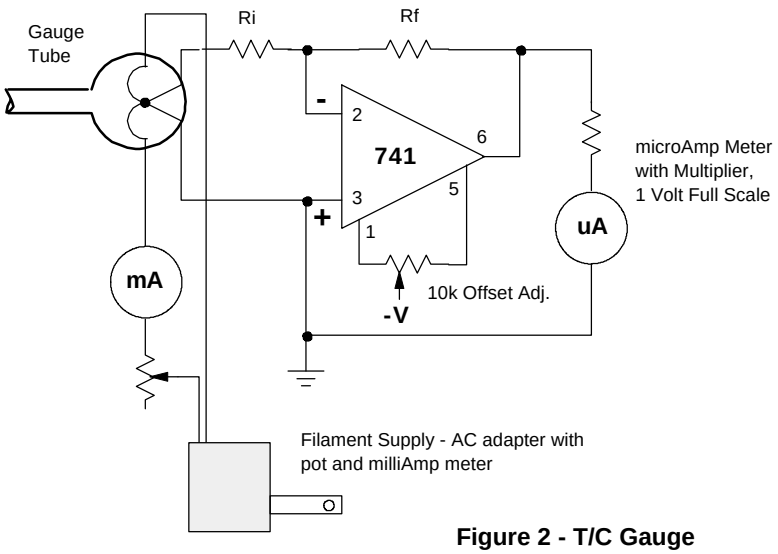


Figure 2 - T/C Gauge Controller

That leaves us with the gauge tube and the readout (thermocouple) meter. For the tube, I don't think that there is much purpose to trying to build your own. Buy one of the cheaper ones (some suggestions will follow below). The meters are specialized items in that they have to be compatible with the millivolt level, low impedance output of the gauge's thermocouple.The more commonly available milliamp/microamp meters have coil resistances many times the 55 ohms of the meter used in a typical gauge controller. Connect up a standard microamp meter to the gauge tube and it might budge, but probably not much. If you buy a meter as a subassembly you will get a very professional and calibrated readout as long as you use the tube for which it was intended. Even with this route, the complete gauge should come in at half the price of a new commercial unit.

A very satisfactory alternative involves the placement of an IC amplifier/buffer between the gauge and the meter. By selecting the right values of components, almost any meter can be coupled with any gauge tube as long as you know the tube's maximum output and calibration curve. The next section will detail how to build an op-amp based T/C gauge using either of two inexpensive tubes.

III. AN OP-AMP BASED T/C CONTROLLER

The meter side of this controller is based on a single stage op-amp amplifier configured in the inverting mode. To establish the component values in the circuit (the values of the input and feedback resistors) one needs to know the load resistance for which the T/C tube was calibrated and the maximum output voltage of the thermocouple at "full" vacuum. The latter corresponds to a full scale deflection of the meter and is taken at a pressure of 10^{-4} Torr. The tubes we shall consider are the DV-6M, 531 and 6343 (and their Kurt J. Lesker equivalents). Relevant data on these tubes is shown in the chart on the next page. The circuit is shown in Figure 2. Since the input impedance of an inverting amplifier is set by the input resistor, the value for this should be 55Ω. I elected to measure the output with a 30 kΩ/volt multimeter set on the 1 volt scale. Thus the gain of the amplifier would have to be set to

up the 14 mV T/C output to 1 volt, a gain of 71.4. As the amp's gain is set by the value of the feedback resistor, R_f, divided by the value of the input resistor, R_i, R_f should be about 3.9 kΩ. As it turned out, the closest values I had on hand were 47 kΩ and 3.3 kΩ which would give a gain of 70.2. Close enough, I figured.

The op-amp is a 741 and the circuit was assembled on a Radio Shack proto p.c. board, catalog number 276-159. I used a regulated +/- 15 volt supply but a couple of 9 volt batteries would work as well. Likewise a 50 μA meter (surplus of course) with a series resistor could be used in place of the multimeter. Do include the offset pot for zeroing.

On the filament supply side it does not matter which pin you select as the positive pin. However, it is essential that the filament supply be independent of the amplifier circuit (i.e. no common ground). Otherwise you will end up just amplifying the filament voltage (the filament and the thermocouple are electrically connected). The filament pot (as well as the offset pot) are 10 turn wirewounds. Fair Radio Sales and other surplus electronics houses have them for about three dollars per.

To get the gauge going, connect the tube to your system with the threaded connection (use Teflon tape or other sealant) or just slip it into a piece of tight fitting rubber vacuum tubing and tighten with a hose clamp. Octal sockets are available from Fair Radio and 4 conductor telephone type cable is good for the tube to controller connection (this should be no longer than 10 feet or so). Be sure to have the filament current control at the lowest setting so you don't burn out the tube. Begin to pump down the system and set the offset pot for a "0" reading on the T/C meter. Then begin to bring the filament current up to the value marked on the tube. The T/C meter should begin to creep up indicating that (1) the circuit is working and (2) that you are pulling a vacuum.

Most T/C tubes don't do well when operated at atmospheric pressure. To preserve your tube, don't apply filament power until you are sure that you are drawing a vacuum in the system. Also, avoid getting contaminants in the tube and position it at a location in the system plumbing where oil cannot back up into it.

IV. CALIBRATION

Now, all you need to know is the correspondence between the meter reading and pressure. The table on the previous page, with data points scaled directly from production gauges, gives a reasonably accurate set of points with which to develop a calibration curve. Even with the sloppy resistor selection, my prototype controller tracked a commercial gauge pretty well.

Thermocouple Tube Calibration Table

Tube type	DV-6M	531	6343
KJL Equivalent	6000	5311	1518
Heater Pins	3, 5	1, 3	1, 7
Heater Current (mA)	20	163/165	15/18.5
Heater Type	AC	DC	DC
T/C + Pin	7	5	5
T/C - Pin	3 or 5	7	3
Full Scale mV (into 55 Ω at 10^{-4} Torr)	10	14	10
Atmos.	.000	.000	.000
2000 milliTorr	-	.170	-
1000	.038	.201	.063
500	.084	.283	.106
300	-	.371	.166
200	.211	.450	.227
100	.374	.634	.367
75	.468	.700	-
50	.554	.776	.522
40	.613	.816	.574
30	.682	.854	.650
20	.758	.902	.736
10	.858	.953	.850
5	.926	.977	.917
1	-	-	.989
0	1.000	1.000	1.000

Note: 0.000 is meter zero. 1.000 is meter full scale.

Originally published in Volume 1, Number 4 with an update in Volume 3, Number 3. Another pinout/calibration table was published in which some figures were transposed. It is believed that the table above is correct. If you note any errors, please send a note.

Easy Gauging - In Praise of the Lowly Discharge Tube

Steve Hansen

I. INTRODUCTION

When you think of what sorts of gauge are useful for measuring vacuum in the region of a few Torr down to several mTorr, the ones that usually come to mind are manometers (like the McLeod gauge) or heat conductivity gauges (like the thermocouple type). Think of discharge gauges and the cold cathode Philips gauge may come to mind. However, one type, which has been rather neglected of late, is the simple discharge tube. Consisting of nothing more than a glass tube with an electrode at each end it is a very useful instrument for assessing conditions within a vacuum system. Note that I avoid saying "pressure measurement." The simple discharge tube gauge will give only a general indication of pressure. On the other hand it is pretty much foolproof, has no sensitive or limited life parts, requires no calibration, and will also indicate (by the color of the discharge) what gases are dominant in the system. Discharge tube gauges are too imprecise to be used on modern/commercial systems but where all that is needed is a semi-quantitative indication of vacuum quality, as in many amateur applications, this sort will do quite well. In fact, discharge tubes were once used in the forelines of high vacuum systems where all that is needed is an indication of when it is safe to turn on the diffusion pumps. The only "real" gauge needed would be a quantitative one connected to the high vacuum side.

II. DISCHARGE CHARACTERISTICS

For a two electrode system, as the pressure is lowered the voltage required to achieve breakdown across the gap also decreases. A glow discharge will form at around 100 Torr, first in the form of a thin streamer. As the pressure is decreased further the glow discharge forms several distinct regions. Figure 1 depicts these regions as would be seen at pressures of 0.1 to 1.0 Torr. Proceeding from the cathode (negative electrode) there is first a very narrow unlit gap called the *Aston dark space* (note that "dark" does not mean that there is no luminous emission at all, just that the level is dim relative to that of the adjacent "bright" regions). This is followed by the also thin but luminous *cathode sheath*, then the wider *cathode* (or *Crookes*) *dark space*, a broader *negative glow* region, the prominent *Faraday dark space*, and then the long *positive column*. The positive column usually bears a *striated* appearance. Judging the pressure in a system is performed by comparing the appearance of these various regions with descriptions appearing in the literature. Some guidelines will be discussed later.

III. JUDGING PRESSURE

As stated previously, the general appearance of the discharge will vary with pressure and this is how the discharge tube is used as a gauge. The literature generally gives the important "break points" as shown in the chart on the next page. Any of these are subject to change somewhat based upon tube geometry, applied voltage, etc., but I've generally found them to be reliable within the accuracy one would expect of such a subjective instrument. It is important to try to keep your tube's parameters (geometry, applied voltage, etc.) constant: its messages will be more meaningful over time.

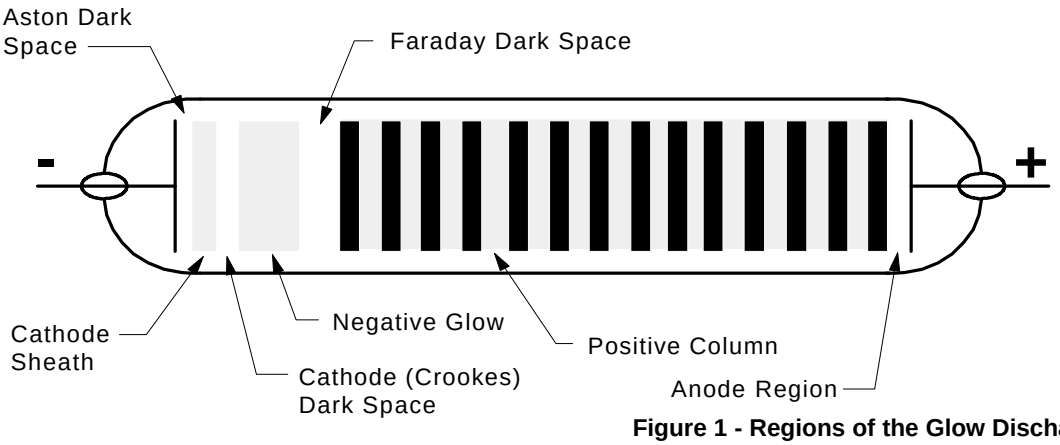


Figure 1 - Regions of the Glow Discharge

Appearance of Discharge at Various Pressures

50 Torr	Thin red/violet streamer
10	Streamer fills tube
2	Striations in positive column
0.5	Striations spaced 10mm
0.1	Crookes dark space forms
0.01	Green fluorescence of glass
0.001	Black out

IV. CHARACTERISTIC COLORS

The type of gas in the system may be judged by observing the color of the discharge. Depending upon the type of gas present, one region of the discharge may have a different color from another. However, for the range of operation we are considering, the most prominent region is the positive column. The table to the right provides a listing of the characteristic colors for that region.

By observing the discharge color, it is possible to perform some system diagnostics. Leaks may be detected by carefully swabbing sections of the system with a cover gas or with a volatile liquid while monitoring the discharge tube. The operation of leak valves used for the intentional admission of fill gases may also be verified with the discharge tube.

V. CONSTRUCTION OF A DISCHARGE TUBE

The basic elements of a discharge gauge are a glass tube (preferably borosilicate as the electrode regions can get hot), a pair of suitable electrodes, and an evacuation port for connection to the vacuum system. A very nice tube may be made using neon sign type electrodes with glass to metal seals. A demountable version made from a piece of chemical labware is shown in Figure 2. This might be considered to be overly complex but this particular design also serves as a good example of the type of vacuum apparatus that may be made from standard laboratory glassware.

Discharge Color with Various Gases

Air	Red to pink, paling at lower pressures
Water	Faint blue
Mercury	Blue
Carbon dioxide	Bluish green
Oxygen	Pale yellow & pink
Nitrogen	Red
Helium	Red/violet
Neon	Brick red
Argon	Dark red
Decomposed oil	Gray/green

The tube is what is known as a chromatographic column, commonly used in analytical chemistry. This particular column has threaded ends that are compatible with a number of plugs, connectors and bushings. This connection system has been patented by Ace Glass and, at this point, such labware is only available from them. (However, they do sell the threaded ends so that any glassblower can fabricate customized pieces with these fittings.) A wide variety of thread sizes and glassware shapes are available from Ace Glass and a well selected set of components may be combined into a wide variety of vacuum apparatus by the experimenter. (There is more information on the Ace Glass components elsewhere.) The particular tube is an 11 mm column, 1 foot in length, Ace catalog number 5820-04. The Nylon bushings are Ace catalog #7506-02. Each bushing comes with an O-ring. The Ace components will cost about $35.

One electrode is just a piece of 3/8" aluminum rod. The other electrode, which also serves as the evacuation port, is made from 3/8" od aluminum tubing. Aluminum is to be preferred over other common soft metals such as brass and copper as those metals tend to quickly deposit the electrode material on the wall of the tube through an ion bombardment process called sputtering. This coating of sputtered metal obscures the view of the discharge and will also lead to erratic operation since the coating is conductive. Stainless steel is an acceptable alternative.

Assembly consists of sliding each electrode through the nylon bushing and O-ring and then screwing thebushing into the end of the column. Finger tight will do it. Each electrode should project an inch or so beyond the bushing. A piece of rubber or PVC hose and hose clamps completes the tube assembly.

The necessary high voltage may be supplied by an old oil burner transformer with a suitable rectifier diode. These transformers are current limited at 5 to 20 mA and usually produce 5 kV rms per side (the secondaries are center tapped to the case). For a small tube such as this, a series resistance in the secondary or a variable transformer in the primary is advisable in order to control the current.

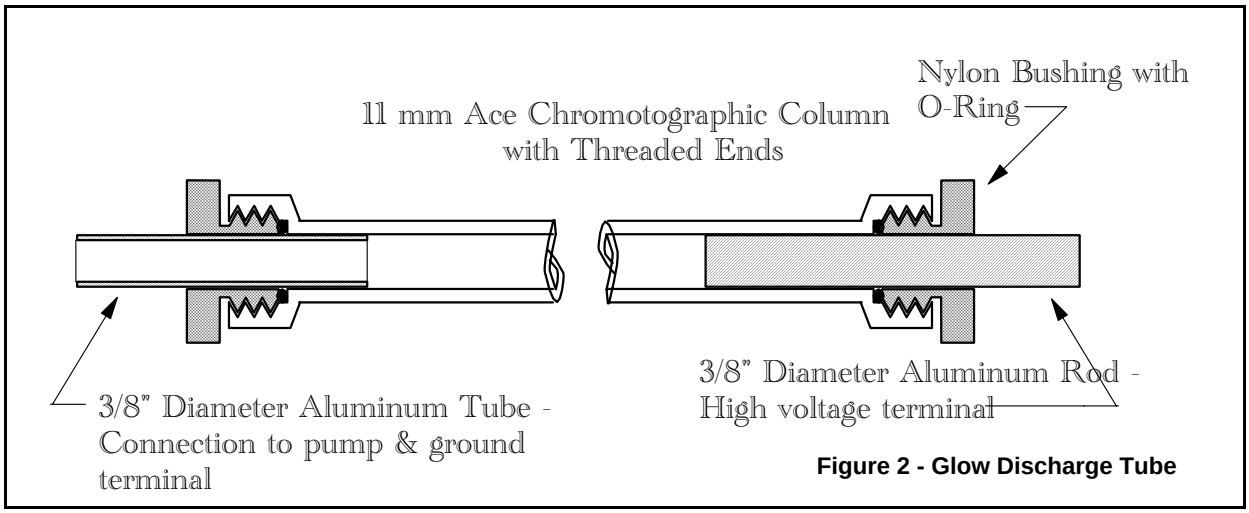


Figure 2 - Glow Discharge Tube

More on Thermal Conductivity Gauges

A Thermistor Gauge Circuit from Roy Schmaus and the Ionic Wind Voltmeter

I. INTRODUCTION

Thus far we have presented a single op-amp controller for a couple of low cost commercial thermocouple gauge tubes.

Another type of thermal conductivity gauge is the thermistor gauge. This is very similar to the Pirani gauge (see the next article) except that a thermistor element replaces Pirani's tungsten filament. Thermistor gauges are not commonly used. However, they are rugged and are frequently used in refrigeration servicing.

This article describes the construction of a thermistor gauge that uses a standard thermistor element with an op-amp based controller. This circuit was developed by Roy Schmaus of the University of Alberta in Edmonton, Alberta.

II. THE THERMISTOR GAUGE

Figure 1 shows the circuit for this gauge. Roy writes "This is a very low cost vacuum gauge that spans a range from about 10 microns to 10,000 microns Hg. It uses a small glass bead thermistor as a sensing element. The gauge is not super-accurate but is still useful for many rough vacuum applications.

"The thermistor is maintained at a constant 105 °C by a self-balancing bridge formed by the resistor network to the left of A1. The IRF520 power FET bridge output (see Figure 2) is level shifted by amplifiers A2 through A4. This results in a 0-10 volt output range as shown in the plot of Figure 3.

"The IRF520 requires a small heatsink. The +15 volt supply comes from a 7815 regulator and the -15 volt supply can be from a simple zener shunt regulator.

"The thermistor itself is mounted in a 1/2" od glass tube which is sealed with low vapour pressure epoxy with the epoxy enclosing the two feedthrough pins at one end. The other end mounts in a Cajon VCO fitting that is then attached to the vacuum system. Other mounting schemes are up to the user's ingenuity, but keep the thermistor maximum temperature rating of 300° C in mind.

"In our application the output of the gauge controller goes to an A to D converter and computer.

"The circuit will also function as a Pirani gauge control. In fact it was originally developed to replace our ancient CVC Autovac gauge controls. In this application, the wires leading to pins 5 and 6 of A1 are swapped and Rs was around 55 ohms, i.e. trimmed to match the salvaged Autovac meter. The Autovac is no longer made but we are still using three of these controllers in the lab with their original gauge heads.

"The CVC gauge heads have a room temperature resistance of around 88 ohms. Ten watt outdoor Christmas tree lights typically have a resistance of around 120 ohms and may be worth investigating as a source of Pirani gauge filaments.

"The calibration curve for our thermistor gauge is shown in Figure 3. Please note that the calibration will vary somewhat for each individual thermistor."

Roy Schmaus
Room 442 Civil/Engineering Bldg.
University of Alberta
Edmonton, Alberta
Canada T6G 2G7
email: schmaus@ee.ualberta.ca

The thermistor used by Roy is a Fenwal GC32J2. Fenwal has a large dealer network and one near you can be found by contacting Fenwal at 1-508-478-6000.

III. COMMERCIAL THERMISTOR GAUGES

If you look through the pages of the catalogs of industrial vacuum equipment suppliers you will be hard pressed to find thermistor gauges. However, they are the pretty much the standard vacuum gauge for refrigeration servicing. Typically these gauges are designed for portable use, much like multimeters and other instruments used in the trades. The sensors are integrated with the control and the connection to the system is made with refrigeration charging hose. These hoses are O-ring sealed and are compatible with the 1/4" male flare fittings supplied on the gauges.

W.W. Grainger supplies a gauge made by Supco for about $120. Instead of a meter the gauge has a row of 10 LEDs which indicate pressures of 5000, 1500, 1000, 700, 400, 300, 200, 100 and 50 microns Hg. Robinair makes a similar gauge as well as one with a conventional analog meter that measures over a range of a few microns Hg up to 25,000.

IV. CONVECTION-BASED THERMAL GAUGES

At pressures over a few Torr conduction becomes pressure independent and a thermal conduction gauge will no longer work. However, if air is allowed to circulate, the amount of convective air flowing past the heated element will give an indication of pressure. Indeed, some Pirani gauges are designed to also work in a convection mode thereby extending their range up to atmospheric pressure. This principle is also applied in such instruments as the hot wire anemometer which is used to measure air flow velocity in ductwork and to detect drafts around leaky doors.

Since most readers of this journal are also high voltage fanatics, an interesting variation on this theme is the so-called *Ionic Wind Voltmeter*. This instrument was described by Thornton, Waters and Thompson in 1931 (*Jour. Inst. Electr. Eng.*, 69: 533). The device is shown schematically in Figure 4. The following description is from the National Bureau of Standards' Handbook 77, Volume 1, *Precision Measurement and*

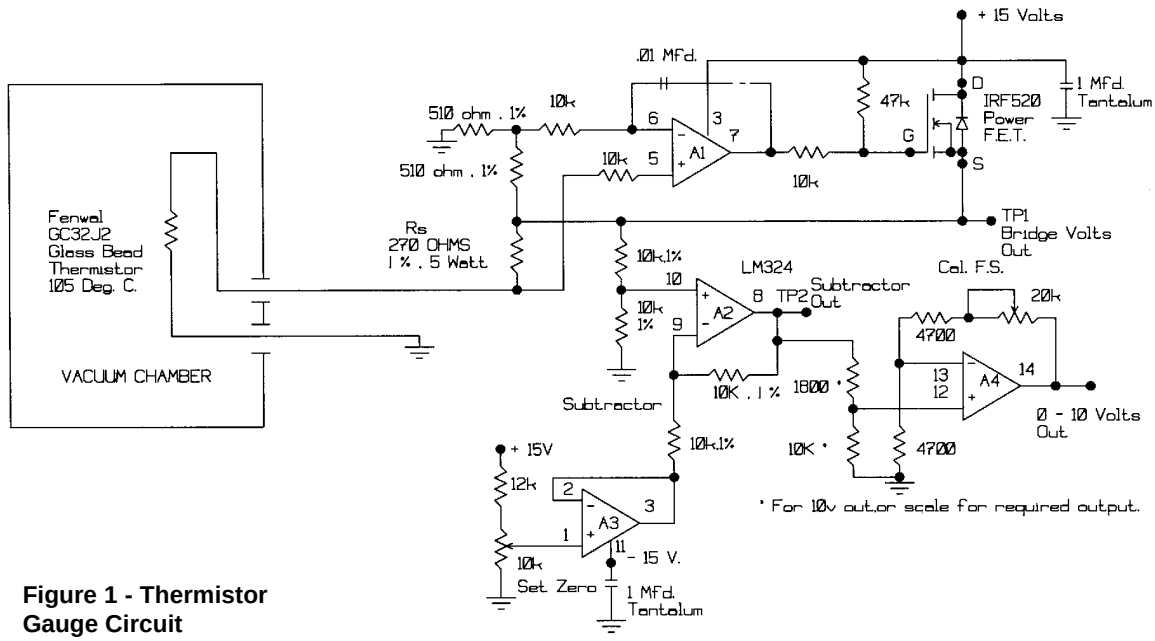


Figure 1 - Thermistor Gauge Circuit

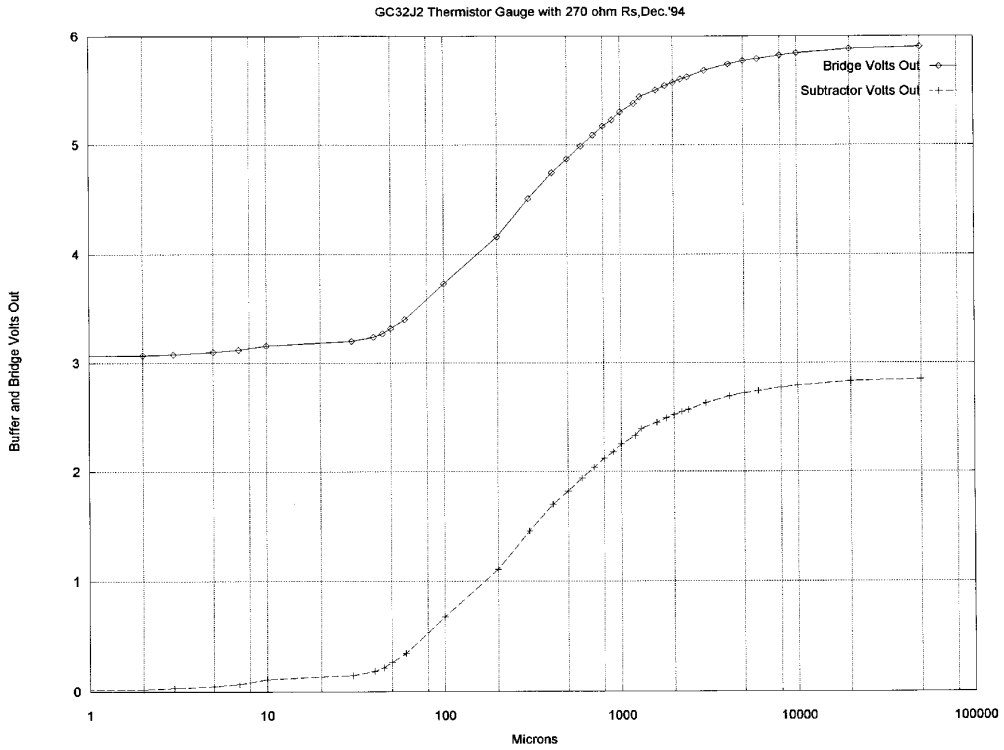


Figure 2 - Bridge and Subtractor Outputs

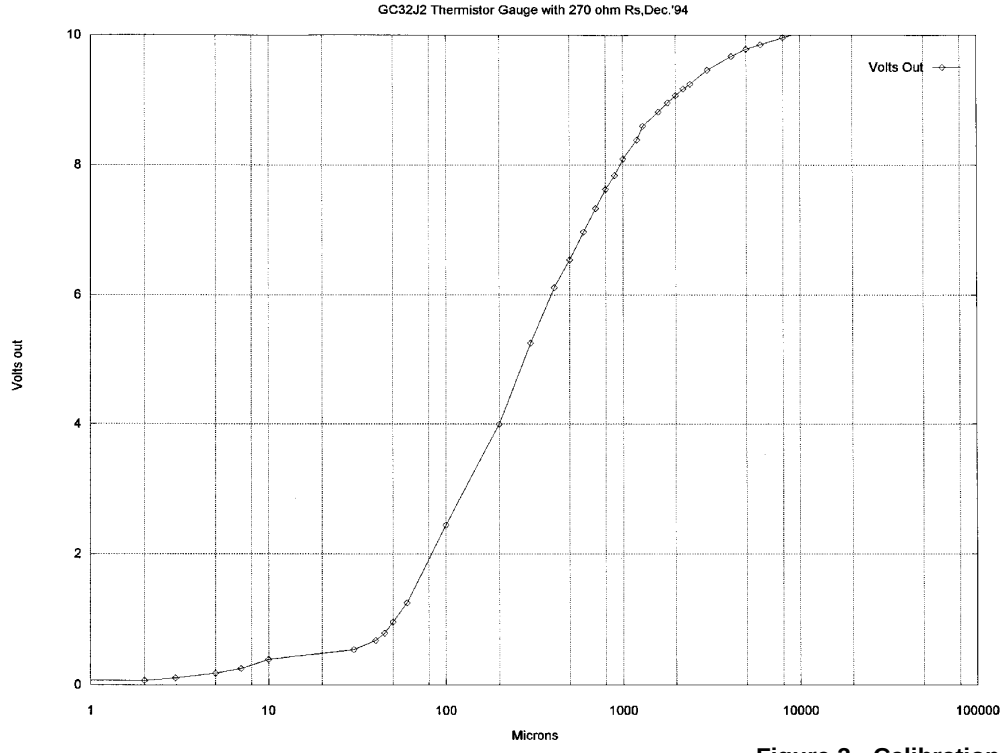


Figure 3 - Calibration Curve

Calibration - Electricity and Electronics as contained in an article on the measurement of high voltages by F.M. Defandorf.

"The Ionic Wind Voltmeter merits some attention because it represents a unique application of a thermal method to the measurement of high alternating voltage. Use is made of the cooling effect on a heated filament arising from "electric wind." The heated filament, with a suitable ground shield, is located at a distance from the high-voltage electrode in such a position that, although in the electric field, it will always be below corona-forming voltage. Ions that may be present move back and forth as a result of the alternating electric field and in striking neutral molecules increase the general molecular motion. This results in an increase in cooling effect on the heated filament proportional to the electric field. A filament that has a high temperature coefficient of resistance is connected in one arm of a Wheatstone bridge. The bridge out-of-balance indicator is then calibrated in terms of the high voltage applied to the Ionic Wind Voltmeter. Although this device may be constructed to have good sensitivity and is useful as a control device or relay, its indications are affected by change in wave form and an accuracy of only plus or minus 2 percent is claimed for it."

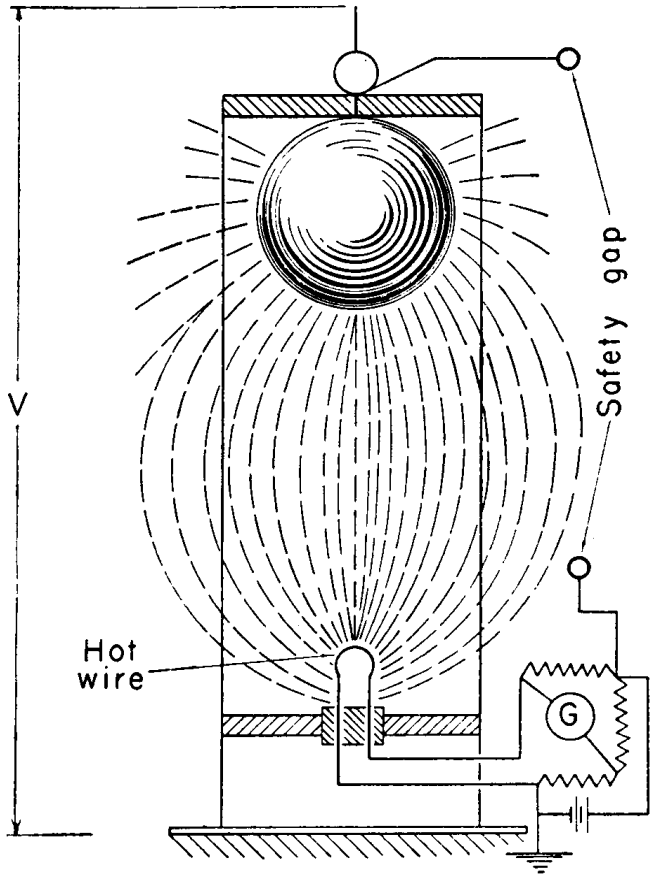


Figure 4 - Ionic Wind Voltmeter

Originally published in Volume 4, Number 2

Constructing a Pirani Gauge

F.B. Lee

The Pirani gauge is one of the most inexpensive vacuum gauges available and, in its simplest form, the easiest to construct. Its range of application, however, is limited. There are no vacuum gauges that cover the full range of pressure from atmospheric to ultra high vacuum, so most systems employ more than one type of gauge. The Pirani gauge is useful for the pressure range of 0.01 to 1 Torr. This is the (foreline) pressure range in which diffusion pumps operate, so a Pirani gauge is very well suited to measuring the pressure between mechanical pumps and diffusion pumps. The gauge responds well to vapors as well as gases, which is also desirable.

This gauge consists of a hot wire maintained at a temperature of about 400° C. (State of the art gauges operate at a lower temperature, about 120° C.) In a high vacuum, the wire loses heat only by radiation. In the presence of gases, the wire also loses heat by thermal conduction. Thermal conduction changes with gas pressure and type of gas. The higher the gas pressure, the greater the heat loss. The relationship between heat loss and pressure is not linear, and the gauge is usually calibrated against an absolute manometer (such as a McLeod gauge) which has a predictable and definite relationship to gas pressure.

Traditionally, Pirani gauges are operated in pairs. One of the pair is evacuated and sealed; the other is connected to the vacuum system. The heated wires in the gauge are electrically connected as shown in Figure 1. The sensing element of this particular gauge is a small lamp having a 12 ohm filament (e.g. #19 lamp). Two lamps connected in series and supplied with a voltage of 1.3 volts will heat to about 400° C. At this temperature the resistance will increase to about 25 ohms. In the bridge circuit of Figure 3, a voltage of about 0.25 volts will appear between terminals T1 and T2.

One can use a small power transformer with half or full wave rectification to provide the necessary power. One may also provide a 1 mA dc meter between T1 and T2. The meter should be provided with a shunt to provide a combined resistance of 20 ohms. (An 80 ohm resistance meter, for example, would require a 27 ohm shunt.)

To assemble the gauge, connect lamp sockets and resistors to produce the electrical circuit shown in the figure. The lamp to be used as the gauge should have long enough leads to reach the point of gauge connection. Apply approximately 1.3 volts across terminals T3 and T4 and observe the meter reading between T1 and T2. The reading probably will not be exactly zero due to differences in bulbs, resistors, or lead wires. Apply a suitable shunt resistor between T3 and T1 or T1 and T4 to correct the imbalance. Next, using a small file, file a notch in the end of the bulb to be used as the sensor. A large reading will now appear on the meter. Adjust R to cause the meter to read full scale. The bulb to be used as the sensor is now connected to the vacuum system with a piece of rubber tubing. The calibration curve for the bulbs used in the prototype is shown in Figure 2.

This gauge will retain its calibration for years unless contaminated by oil from the pump. To help avoid this, install the gauge where it would be unlikely to encounter pump oil.

Originally published in Volume 4, Number 1.

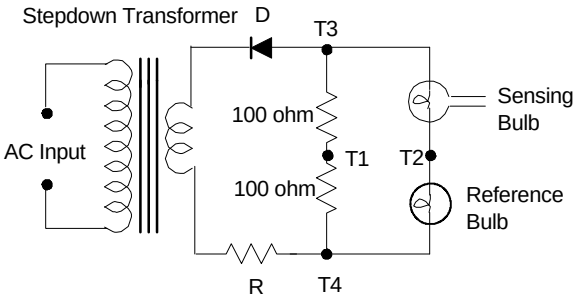
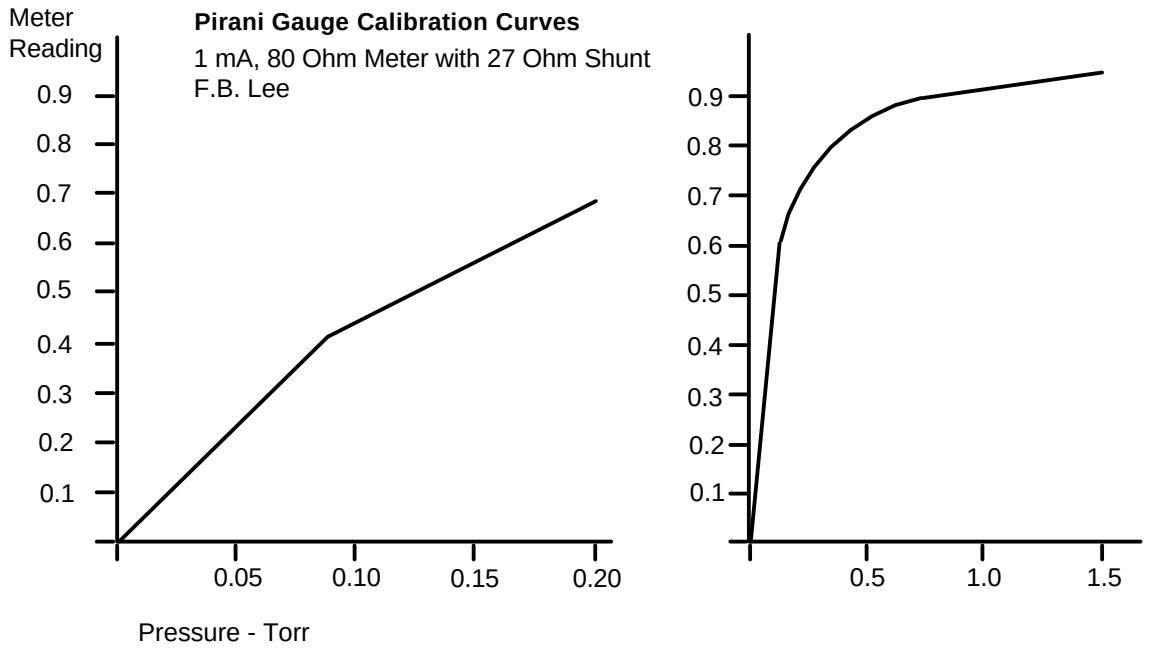


Figure 1 - Pirani Gauge Circuit

Figure 2



More on Pirani Gauges

In a recent communication, Frank Lee suggested the use of miniature Christmas tree bulbs. These are about ¼" in diameter by 1" long which makes them easy to attach to a rubber or pvc vacuum line. The seal, which is at the end opposite the base, is easy to break and the wire leads are easily straightened for connection to the circuit. Avoid the colored bulbs as these have a plastic coating. For obvious reasons, also avoid the type of light that blinks.

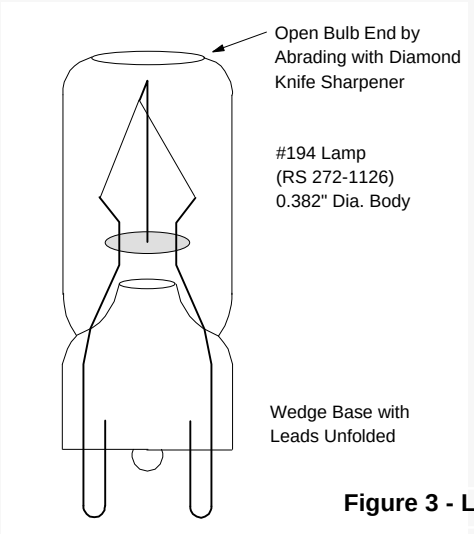


Figure 3 - Lamp

Another handy bulb is the #194, shown in Figure 3. I open these by abrading the end away with a diamond impregnated knife sharpener. A couple minutes of scraping makes a clean opening without jagged edges. Additionally, the diameter is just right for a good fit in a standard 3/8" brass compression fitting - just replace the standard brass ferrule with an O-ring. While the leads are not solderable, they are long enough to connect with crimped terminals. These bulbs are a standard item at Radio Shack.

Small opened bulbs such as these can also be used as cheap, low current (10 to 20 υA) electron sources.

Dr. Bruce Kendall of Penn State has had success using model airplane engine “glow-plugs” as Pirani gauge sensors. They are rugged and don’t require the cutting of glass.

In lieu of the traditional Wheatstone bridge circuit as shown with this article, the experimenter could try using a regulator IC (e.g. LM317) as a constant current source with a cheap digital voltmeter as the readout device. - *Editor*

Further Reading

Most good vacuum books have decent sections on the various vacuum gauges. I would recommend Saul Dushman’s *Scientific Foundations of Vacuum Technique* (John Wiley, 1949 or the later editions) for its detailed coverage of radiometers, radiometer-type gauges and thermal conduction gauges.

How to Make a Glass to Metal Seal

Kevin E. Bennet

Abstract: Using simple glassblowing techniques to fabricate a gas discharge tube with a tungsten-glass seal for the electrode.

I've tried glassblowing but have never been very successful. I think that much of the reason is that I've never paid much attention to getting a good torch. In this article, Kevin Bennet describes a good solution to getting a usable torch and flame and then shows how to do some useful glasswork. - *Editor*

Caution: Glassblowing requires working with sharp glass, high temperature flames, hot glass and hot metal. Be very careful keep flammable materials away from your work area and take care not to cut or burn yourself. Wear safety glasses at all times when working with glass, hot or cold. Also, keep a pan of cool water nearby into which to put your hand should you burn yourself. The sooner you cool the burn, the less damage to your skin!

I. INTRODUCTION

Many of the useful and most interesting things to be done with vacuum systems involve the introduction of electricity into the evacuated enclosure. This electrical feedthrough represents one of the most basic techniques in vacuum glasswork and also one of the most difficult to be done reliably on a routine basis. However, the selection of the correct materials combined with proper technique and practice will yield reliable seals. Industry has developed glass to metal seals on a production basis as may be seen in such everyday items as lightbulbs and electron tubes. As an example, each string of holiday lights requires 100 glass to metal seals and millions of these are made each year. In learning any new skill, expect failure at first. Practice is required to become successful and proficient.

The difficulty in glass to metal seals lies in the low strength of glass in tension combined with the differences in expansion between the glass and metal components of the seal. Without going into the theory of stress and expansion coefficients, the solution to the problem of glass to metal seals is to use a metal and glass combination where expansion is matched at all temperatures and where the metal forms an adherent oxide coating to allow the glass to stick to the metal and seal tightly. A variety of metals which approximate these needs are available for this service including platinum (for soda lime glasses), molybdenum (for silica glasses), tungsten (for borosilicate glasses), a copper/iron composite known as Dumet (for lead glasses), as well as copper and various iron alloys (such as Kovar) for a variety of glasses.

II. ABOUT GLASS

Of the commonly available glasses, the borosilicate variety (known by various trade names like Pyrex®, Kimax®, etc.) is the most forgiving of thermal shock during the glassblowing process. This is the material of choice for most laboratory glassware.

Soda lime glass is the glass from which most consumer products are made. Examples include bottles and light fixture globes. This type of glass cannot be worked by torch in any resonable manner. Soda lime laboratory tubing is made, but it is not recommended for scientific glassblowing.

Lead glass has a history of use in the fabrication of neon signs, incandescent lamps and electron tubes. Lead glass can be purchased from scientific or neon sign suppliers. This glass will crack very easily with the application of a torch and requires considerable patience to work. Dumet wire (copper coated iron) for making glass to metal seals may be recovered from any lightbulb. Lead glass requires a lower temperature flame such as from a propane or natural gas and air torch in order to be worked effectively.

Getting back to borosilicate glasses, thin walled glass (from about 0.75 to 1.0 mm) is the easiest to form and the easiest to flame anneal (remove residual stress). Since glass is a poor conductor of heat, the use of a torch on thicker glass results in the development of severe strain which then results in cracks. Thus, bell jars, which are made from heavy glass, are very difficult to modify or repair with a torch.

III. THE TORCH

Bunsen burners and the propane torches commonly used for plumbing will not work properly with borosilicate glasses. While these will melt the glass, the temperature of the flame simply is not high enough to

properly work the glass without considerable frustration. For satisfactory work an oxy-gas torch is required. While special glassworking torches are available, a good alternative (and one that is fairly easy to locate and buy or borrow) is the common oxy-acetylene torch. However, acetylene burns much too hot and will boil the glass and melt the thin metal electrode material. On the other hand, these torches may be used with propane (as, for example, is used with your gas grill). You will need to put together an adapter to connect the propane line (after the regulator, please) to the fuel line to the torch. Such adapters may be purchased from a welding supply house or well stocked hardware store. Another alternative would be a gas/oxygen "blowpipe" as may sometimes be found at dental supply stores.

Once a torch has been located and an adapter has been made to connect the propane, use a vise or other holder to support your torch so you will have both hands free to manipulate the glass and metal during the fabrication process. Whatever type of support you devise, make sure that the torch is held securely in place.

IV. THE EXERCISE - A DISCHARGE TUBE

For this exercise we will construct a discharge tube for mounting to a vacuum system to use as a basic but effective vacuum gauge. We will modify a borosilicate (Pyrex or Kimex) culture tube (this is a type of test tube that does not have a lip) to include an electrical feedthrough at one end and sealed to an NPT fitting (or other fitting of your choice) at the other end to fit your vacuum system.

For glass to metal seals in this type of glass, tungsten is the metal of choice. Fortunately, tungsten is available locally in the form of rod from welding supply stores. This is pure metal with a ground surface that is used as the electrode material in TIG (Tungsten Inert Gas) welding. Tungsten is very difficult to form and cut due to its toughness and fibrous structure, the latter a result of the wire drawing process. Tungsten is best cut into the lengths required for electrical feedthroughs (about 1¼ to 1 ½ inches long) by grinding through the rod with the edge of a grinding wheel (a small wheel in a Dremel tool is effective).

While at the welding supply store, also purchase a length of pure nickel (normally used to weld cast iron) to use in sealing the fibrous structure at each end of the tunsten electrode. After cutting the tungsten into lengths for use in feedthroughs, braze the end of the tungsten rod with a small amount of nickel using borax as a flux. This will seal the ends of the tungsten, reducing porosity. Untreated ends might result in leaks in the vacuum system. The nickel will also form a suitable base for the subsequent welding or brazing of electrodes or leads to the tungsten.

The smaller the diameter of the tungsten rod you are using, the more successful you will be. A diameter of 0.040 to 0.050 inch is a good starting point.

Now you are ready to make the glass to metal seal. For sealing tungsten to borosilicate glass an intermediate glass is frequently used to better match the expansion characteristics of the glass and metal. For example, uranium glass (Corning 3320) may be sealed to the tungsten followed by Nonex (Corning 7720) and then the borosilicate (Corning 7740). This structure is known as a graded seal with each glass representing an interim step in expansion matching. The end result is a low stress seal. However, with the small diameter wire we have chosen, the tungsten may be sealed directly to the borosilicate component. The steps required to make a seal are as follows:

- Clean the tungsten
- Lightly oxidize the tungsten
- Place a sleeve of glass over the tungsten rod
- Fuse the sleeve to the tungsten
- Bead the sleeve with more borosilicate glass
- Seal the feedthrough into the vacuum system

The sleeve is made by drawing out a larger piece of tubing. Take a test tube or other tube of borosilicate glass and heat it in the center while rotating to soften a band about ½ to 1 inch wide in the center of the tube. The tube is then removed from the flame and pulled apart in order to form a length of tubing with an inner diameter slightly larger than the tungsten wire. After cooling, the small diameter tubing is scratched with a triangular file and pulled apart at the nick to form tubes about 3/8 inch long. These tubes are set aside for sealing to the tungsten after the tungsten is cleaned.

The tungsten is cleaned by holding the tungsten with pliers or a pin vise and heating it with the torch at the place where the glass will be bonded until the tungsten reaches a bright red glow. The hot tungsten is then rubbed in sodium nitrite until well coated and then washed in distilled water to remove the sodium nitrite residue. The tungsten is then dried with a cloth or paper towel. The appearance of the treated and cleaned wire should be a shiny grey color.

After cleaning the tungsten is placed back in the flame and heated to a dull red for a few seconds to create a uniform film of tungsten oxide on the surface. This oxidation is important to the formation of the seal since too little oxide will not make the required glass to metal oxide bond and too much oxide will result in a seal which leaks.

Once cool, a 3/8 inch length of borosilicate tubing is passed over the wire and centered over the area which was cleaned and oxidized. Starting at one end of the length of tubing, the tubing is gradually melted and shrunk while rotating the tungsten. As the glass shrinks and fuses to the tungsten oxide, the color of the glass to tungsten interface will change to a golden yellow to copper color. A shiny grey interface will indicate there there was not enough oxide formed initially and a dirty black color will indicate that the oxide coating was too thick.

FIGURE 1 - **Glass Bead on Prepared Tungsten Wire.** Adapted from Ref. 1, used with permission.

Using one of the ends of the test tube you used to make the small sections of tubing, wind a ribbon of glass around the center of your tungsten to glass seal to form a bead about a quarter of an inch in diameter. This will make sealing to the discharge tube much easier. After forming the bead, turn off the oxygen and rotate the seal in the luminous gas flame for about a minute to slowly cool the seal. Then remove the tungsten seal from the flame and continue rotating for another minute to uniformly cool the seal. Then set it down on a fire resistant surface (not a metal surface as heat will be conducted away too rapidly and potentially cause stress).

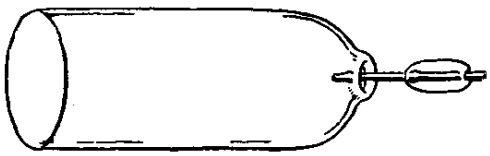


FIGURE 2 - Beaded Feedthrough Ready to be Sealed into Pierced Tube. Adapted from Ref. 1, used with permission.

In preparation for sealing the tungsten feedthrough to the closed end of the culture tube, connect about a 2 foot length of ¼ inch tubing which we will use as a blow hose to a one hole stopper (the stopper may be cork or rubber). Place the stopper in the culture tube and put the open end of the blow tube in your mouth (it helps to have a rigid mouthpiece such as a pipe stem) and, using a small gas/oxygen flame, soften the end of the culture tube in an area less than ¼ inch around. Once the area is heated to a pale red/orange color remove the culture tube from the flame and blow briskly. The heated portion will enlarge and may "pop" forming an opening in the end of the tube slightly smaller than your tungsten feedthrough. If an opening does not form, reheat the enlarged area and blow again. Strike off the excess thin glass with a file and clean up the edge of the hole by melting the ragged edge with the torch.

Taking the tungsten feedthrough in the pin vise, slowly warm the completed seal by passing the seal through the torch flame about 10 times, rotating about a quarter of a turn with each pass to heat uniformly. With the blow tube attached, hold the culture tube in the other hand and pass through the flame at the same time to get all of the working areas up to temperature. Once you see the flame colored by the sodium in the glass, the temperature can be increased and the tube and tungsten seal can be fused together. By application of heat and slight blowing and sucking, the glass can be smoothed out around the seal.

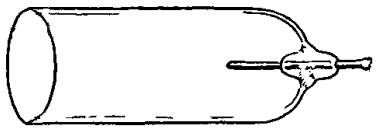


FIGURE 3 - Completed Seal. Adapted from Ref. 1, used with permission.

V. COMPLETING THE ASSEMBLY

A discharge tube normally requires at least two internal electrodes. However, if you have gotten this far in your initial efforts at glasswork, you get a break with this next step. After you have successfully fabricated the glass to metal seal, the next step is to modify the open end to serve as both the connection to your vacuum system and also act as the second (grounded) electrode. Here we will use a standard copper, brass, or stainless steel fitting which is compatible with a metal vacuum line. A fitting with a pipe thread at one end and a solder (sweat) fitting at the other is a convenient choice. Find a fitting into which the glass tube will easily fit. If the fitting is slightly undersize, enlarge it to a loose fit with a drill, Dremel tool, etc. When properly sized, clean all of the parts and attach the glass to the fitting with some epoxy cement. As a last step, affix a piece of wire to the fitting (centered and extending into the lower end of the glass tube) to act as the ground electrode. It is advisable

to use a material which does not sputter well for this electrode. Aluminum and tungsten are good choices, copper is not. Figure 4 shows the completed assembly.

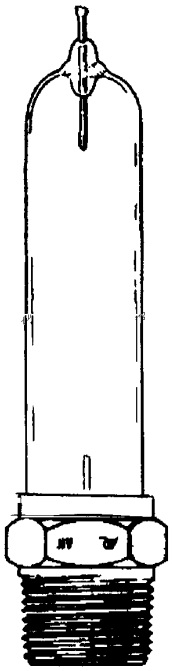


FIGURE 4 - Completed Discharge Tube Assembly.

The high voltage requirements for this tube are minmal. While an AC supply will work, a DC source will provide additional information about the the state of your vacuum system. Usable sources of high voltage include a small Tesla coil of the sort used in glass vacuum system leak detection, a small (2 kV) neon sign transformer (with a diode to give a DC output), or an induction coil.

VI. INTERPRETATION OF THE DISCHARGE

While the use of a gas discharge tube is not widely used to measure pressure, a good indication of the pressure and an idea of what gases and vapors are in the system may be discerned from the discharge appearance and color.

The relationship between discharge appearance according to pressure has been discussed in an earlier article. Another set of useful data is contained in the table below. This includes information for both the positive region as well as the negative.

REFERENCE

[1] John Strong, *Procedures in Experimental Physics* (Lindsay Publications, Bradley, Illinois, 1990).

SUPPLIES & MATERIALS

Gas/oxygen torch and holder (vise)
Ground tungsten rod, 30 to 50 mil diameter
Several borosilicate culture tubes
One hole cork/stopper to fit culture tube
2 feet small (~¼") ID latex (or equiv) tubing with mouthpiece and connection to stopper
Sunglasses or (pref.) dydinium glassblowing glasses
Sodium (or potassium) nitrite (or nitrate)
Nickel rod
Pliers and/or pin vise
Triangular file

Originally published in Volume 2, Number 2.

Discharge Glow Coloration

Gas	Negative Glow	Positive Glow
Air	Blue to Pink	Reddish
Nitrogen	Blue	Yellowish
Oxygen	Yellowish	Lemon
Helium	Pale Green	Violet-Red
Argon	Bluish	Deep Red-Violet
Carbon Dioxide	Blue	White
Mercury	Greenish	Greenish Blue
Neon	Orange	Blood Red

Reconfigurable Glass Vacuum Chambers

Steve Hansen

Abstract: ACE Glass Company produces a variety of standard glassware which incorporate a proprietary internally theaded connector (*Ace-Thred*). Used with the matching bushings and O-rings these glassware items may be easily configured and reconfigured to form a variety of vessels usable in vacuum work.

I. INTRODUCTION

For the amateur, the thought of making complex glass shapes or of joining glassware to metal tubing usually brings forth images of torches and out-of-control sagging glass or of rubber stoppers and various kinds of clamps and goop. The usual methods for configuration and joining consist of the following:

- Traditional glassblowing
- Glass to metal graded seals
- Ground taper or ball joints with waxed or greased seals
- Compression seals (e.g. glass tube to metal tube)

Another approach is to use so-called beaded end glass tubing (also known as process pipe) along with the associated connectors and gaskets to fabricate complex glass shapes from standard components. For example, on several occasions I have seen amateur projects constructed using process pipe. Examples of these various methods are shown in Figure 1.

For the amateur, each of these has limitations with regard to cost, flexibility, and ease of use. An alternative method of construction which I have found to be very useful is based on the so-called *Ace-Thred* equipped laboratory glassware which is supplied by ACE Glass Corporation, P.O. Box 688, 1430 Northwest Blvd., Vineland, NJ 08360. Not specifically designed for vacuum use, ACE has a variety of components which are designed for use in liquid chromatography, photochemistry, and air sampling applications. However, the materials and designs are compatible with any vacuum application where O-ring seals may be tolerated.

II. THE *ACE-Thred*

Figure 2 depicts the construction of the *Ace-Thred* connection. The basic elements are an internal thread which is molded into the glassware, a plastic bushing (either Teflon or Nylon), and an O-ring. The item to be joined to the glassware (in this example another section of glass tubing) is inserted into the bushing with the O-ring placed over the tube last. This is then inserted into the threaded member. When the bushing is tightened, the O-ring forms a seal between the the two pieces of tubing. The plastic bushing is external to the sealed off area.

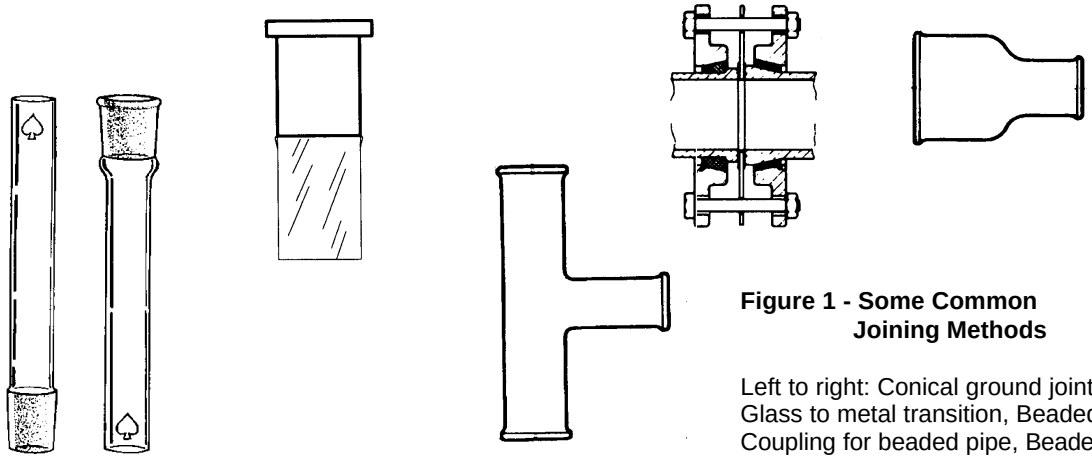


Figure 1 - Some Common Joining Methods

Left to right: Conical ground joints, Glass to metal transition, Beaded tee, Coupling for beaded pipe, Beaded reducer. Illustrations courtesy of ACE Glass and Kurt J. Lesker.

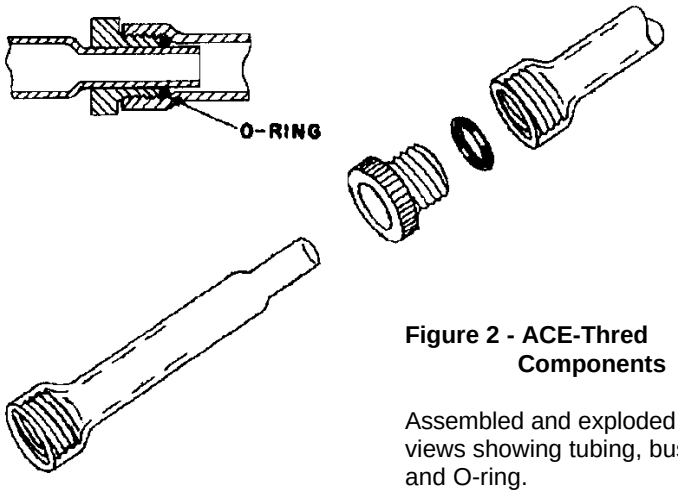


Figure 2 - ACE-Thred Components

Assembled and exploded views showing tubing, bushing, and O-ring.

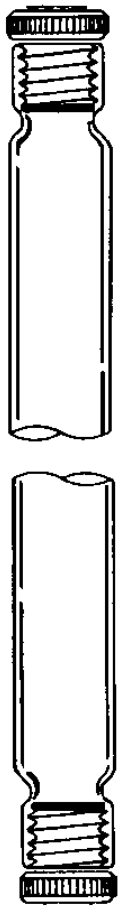


Figure 3 - ACE Chromatographic Column with Bushings

III. STANDARD *ACE-Thred* SIZES

Ace-Threds are sized according to the range of tubing diameters which each bushing and its corresponding threaded glass connector will accomodate. While a number of sizes are available, the ones of most interest are as follows:

- #7 6.0 - 7.0 mm tubing
- #11 9.0 - 10.5 mm tubing
- #15 12.5 - 14 mm tubing
- #25 24.0 - 25.0 mm tubing

For reference, the largest common size is #50 (47 to 48 mm tubing) and the series does extend to #80 (75 mm). The glassware and fittings associated with these larger sizes are quite expensive.

IV. SOME USEFUL GLASSWARE SHAPES

The simplest piece of *ACE-Thred* glassware is the type of tubing used for chromatographic analysis. These are called columns and a typical example is depicted in Figure 3. ACE has a wide variety of these in different diameters and lengths. In the smaller sizes (#11 through #25), prices range from $20.00 to $40.00 (at this writing) in lengths from 12 to 48 inches. The inside diameter of the column corresponds to the thread size, however, ACE does not specify the outside diameter of the column in their catalog. Knowing this is useful if, for example, you want to cut a column into two pieces and fit the plain end into, for example, a metal compression fitting. For reference, Table 1 provides those dimensions.

Table 1 - Outside Diameters of ACE Chromatographic Columns

#11	5/8 inch
#15	7/8 inch

Another useful shape is the preparative funnel, another chromatographic item. This is a basically a bulb with a pair of diametrically opposed threaded fittings. The smallest bulb, the #11, has an outside diameter of 82 mm and a capacity of 250 ml. The largest, the #50, has a bulb diameter of 180 mm and a capacity of 3000 ml. In my view, the most useful size is the #25 which has a capacity of 2000 ml and an outside diameter 160 mm. The current cost for the 25 mm size is about $60.00. Such bulbs are useful for experimental x-ray and Crookes tubes. They should also be useful for "plasma sphere" development and for replications of

Tesla's "button lamp". The preparative funnel is shown in Figure 4.

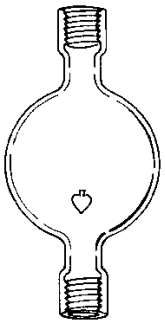


Figure 4 - ACE Preparative Funnel

Tees are essential components for a variety of purposes and ACE offers these in threaded form as well. As with the other components, several sizes are offered, all with equal diameter ends. Figure 5 shows the ACE Tee (called in the catalog a Connecting Adapter). The price of the 25 mm size is on the order of $46.00.

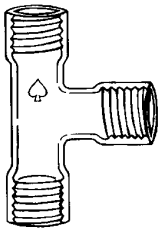


Figure 5 - ACE Connecting Adapter (Tee)

With regard to bushings, there is about a 3:1 price differential between the Teflon and Nylon bushings. At the 25 mm size, the respective prices are on the order of $11.00 and $28.00. As noted above, since the bushing is not exposed to vacuum, the cheaper material will suffice for most normal applications. A considerable number of other related fittings are available such as plugs, pipe thread adapters, and so forth. One type worth mentioning here is the coupling. This fitting allows threaded glass components to be directly connected together. The plastic of the coupling does get directly exposed to vacuum, so material selection is more important. In high vacuum work or where the coupling will be exposed to high temperatures, the Teflon hardware is preferable. Also, while couplings are available which will connect together various sizes of threaded glassware, in my view it is preferable to try to standardize on one size for most applications. My preference for all round use is the 25 mm (#25) size. Figure 6 shows the use of a coupling to join a column to a bulb funnel.

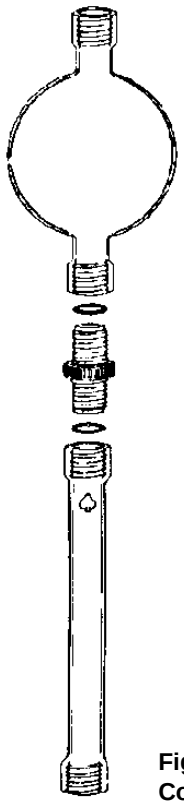


Figure 6 - Use of Coupling to Join Two Items

V. CONNECTING TO THE VACUUM SYSTEM

Now that we have seen a variety of threaded glassware and how the various types of threaded ACE Glass glassware can be fitted to each other, the next issue is how to affect a simple coupling to the remainder of the vacuum system. Typically, this means connecting to metal tubing.

If one has standardized on 25 mm fittings and components, then the adapter approach shown in Figure 7 will be useful. Here the main connecting tube to the vacuum system is 3/4" copper water tube (which is 7/8" OD). Since the #25 ACE bushing has a 25 mm inside diameter, the copper tube must be built up to the required diameter. This may be done by forcing a short length (1.5 to 2 inches long) of .062 inch wall, one inch OD stainless steel tubing (Shapiro Supply Co. is a good mail order source, see the *Sources* section elsewhere in this issue) over the end of the copper tube with a small amount of the stainless tube extending beyond the end of the copper tube. This area should be soldered or

brazed. Next, as the tube may get sucked into the glassware when under vacuum, some sort of shoulder should be provided as a stop. A simple metal hose clamp will work just fine. A more elegant solution is to take a copper 1 x 3/4 inch reducing bushing, file out the lip so that it will fit over the 3/4 inch tube, and then solder this in place with one end against the stainless steel tube section.

If you are using the #15 size components, a similar problem exists. The #15 bushing works best with a tube of about 9/16 inch diameter. The closest size in copper water tube is 3/8 inch tube which has a half inch outside diameter. Here the tube may be built up using two successive sizes of hobby shop brass tubing (K&S Engineering). Simply cut 2 inch lengths of the tubing, slide them over the copper tube, and solder at the vacuum end of the fitting. In this small size there is not enough pressure on the connection when under vacuum to require the shoulder bushing

VI. BUILDING UP THE "ERECTOR SET"

What follows is a recommended starter set of ACE Thred components. All of these components have #25 fittings.

- One 25 mm ID chromatographic column, 24 inches long.
- One preparative funnel (bulb), 160 mm OD.
- One connecting adapter (tee).
- Four nylon bushings
- One teflon coupling
- Spare O-rings

The above will set you back about $240. If you are comfortable with smaller components, the same list in the #15 size will cost about $170. All in all, I have found the ACE components to be of tremendous value in setting up apparatus for a wide variety of experiments ranging from simple discharge tubes to more complex x-ray and particle acceleration devices.

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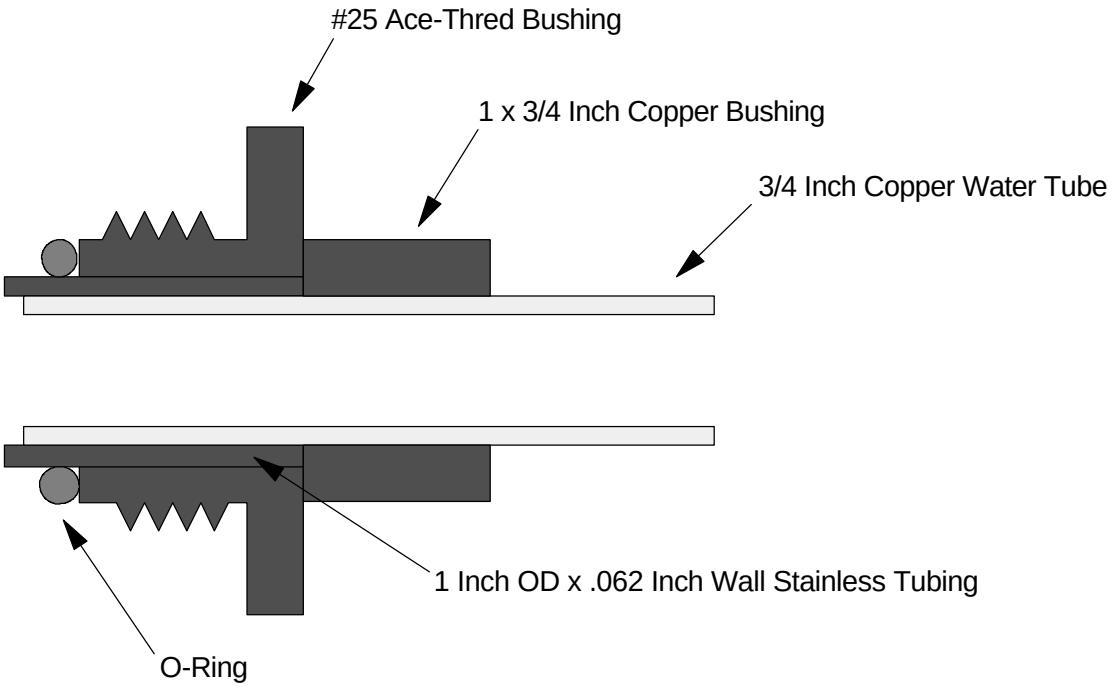


Figure 7 - Copper Tube to ACE-Thred Bushing Adapter

A Simple, Flexible Vacuum Chamber for Beam Experiments

Steve Hansen

I. INTRODUCTION

The purpose of this article is threefold. The main intent is to describe a vacuum chamber assembly which will be used as the basis for a series of articles on ion and electron beam sources and acceleration columns. Within this context, the article will show how a commercially available piece of glassware, in this case a chromotographic funnel, may be used as a vacuum vessel. Finally, two types of simple low cost high vacuum flange will be described.

The integrated assembly is shown in Figure 4. On the topside of the aluminum baseplate is the adapted funnel. Sealing is effected by a Viton gasket with pressure applied by means of an adapted PVC pipe flange and four bolts. Under the baseplate is a Tee manifold. This is connected to the baseplate using KF components. The vacuum system is connected to the side arm by means of an O-ring sealed KF flange. Beam sources, targets, or diagnostic probes may be attached to the CF flange at the bottom. Likewise, similar devices may be attached to the O-ring sealed port at the top of the funnel (not shown in this figure). A straight path is provided through the entire assembly for beam experiments.

II. THE CHAMBER

The chamber is a standard addition funnel which is normally used for liquid chromotography. The particular funnel used for this project is one which is manufactured by ACE Glass Co. (1430 Northwest Blvd., Vineland, NJ 08360). As depicted in Figure 1 this particular funnel, ACE catalog number 5822-15, has a 90 mm inside diameter with an od of 95 mm. The height from the beaded large opening to the neck is 310 mm. At the top is a 25 mm diameter port. This is an *ACE-Thred*® O-ring sealed port (see the previous article). This latter feature is of particular interest for the beam devices which we will be talking about in future articles. The total volume of the funnel is 1.5 liters. Current cost is about $52 (1993) which seems like a lot for a funnel but not too bad for a small borosilicate glass bell jar with a top port.

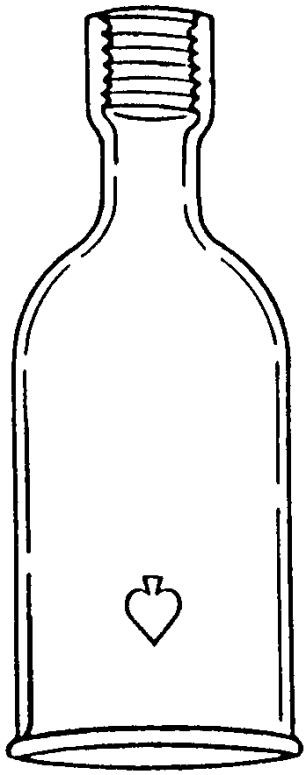


Figure 1 - ACE Addition Funnel. (Not to scale) Illustration courtesy of ACE Glass Co.

III. KF & CF FLANGE SYSTEMS

A very versatile commercial flange for amateur use is the KF system (also known as QF or NW flanges). KF assemblies are relatively inexpensive and, depending upon the materials selected (brass or stainless steel), they are compatible with soldering to standard inch dimensioned copper tubing (e.g. common water tube) and welding to common sizes of stainless steel tubes.

The principle of the KF flange is shown in Figure 2. There are four components: two similar flanges (each of which may accomodate a slightly different diameter tube), a metal center ring which supports the O-ring and keeps the flanges in alignment, and a clamp which, by means of a thumb screw, compresses and holds the entire assembly together.

Five sizes of KF flange are available which can accomodate tubes from ½" through 2-1/8" diameter. Center rings are available which have screens to prevent loose debris from passing through the flange. Also, adaptive rings are available which permit the coupling of some adjacent sizes of KF flange.

I've found the KF25 brass flange, sized for 1 inch copper water tube (1-1/8" OD), and the KF40, sized for 1-½ inch water

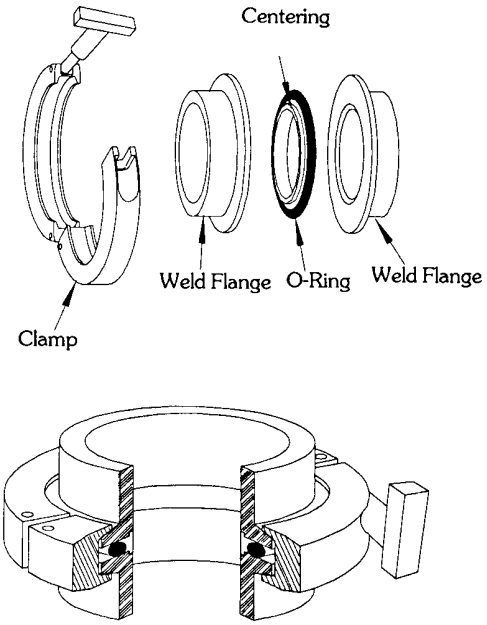


Figure 2 - KF Flange System.
Illustration courtesy of Kurt J. Lesker Company.

tube (1-5/8" OD) to be the most versatile in small vacuum apparatus. A complete brass KF25 assembly with Buna-N O-ring will cost about thirty dollars. As each additional KF25 flange is only about $9, the KF system is an inexpensive route to take when multiple flanged devices are to be attached, one at a time, to a system. A complete KF40 assembly is about forty dollars. Viton O-rings may be substituted at additional cost.

CF (Varian ConFlat® type) flanges use a knife edge seal and, when used with copper gaskets, are fully compatible with ultra high vacuum (UHV) practice. A widely accepted standard flange, they are produced in high quantities and are, particularly in the smaller sizes, quite economical. For less stringent applications, an elastomeric gasket (typically Viton) may be substituted.

Figure 3 shows a typical CF assembly. Flanges are available in stainless steel and aluminum and welded joints are standard practice. When the flanges are tightened with the gasket between the knife edges, the resulting pressure creates a very high quality seal. CF flanges are available in a variety of diameters from 1 1/3 inch up through 10 inches. Variations include bore diameter, tapped or untapped bolt holes, and fixed or rotatable configurations.

For the amateur, the 2-3/4 inch diameter flange is quite useful. Standard bore diameters range from about a quarter inch up through 1-5/8 inch. For special applications, blank flanges may be obtained. Non-rotatable flanges in this size cost about $14. The price is $18 if supplied tapped for the six ¼-28 bolts which are used to hold the assembly together. Copper gaskets are pretty cheap at under $2 but they can't be reused. Viton gaskets are reusable and cost about $6.

While welding is the standard method for joining tubing to a CF flange, there is no reason why soft or hard silver solders can't be used in high vacuum applications. (Solders, of course, would not be advisable in UHV systems or applications where the mechanical characteristics of solders would not be adequate.)

A wide selection of both KF and CF hardware is available from a number of vendors including Duniway Stockroom Corp. (1305 Space Park Way, Mountain View, CA 94043), Kurt J. Lesker Co. (1515 Worthington Ave., Clairton, PA 15025) and MKS Instruments (6 Shattuck Rd., Andover, MA 01810). These companies are excellent to deal with and have catalogs which detail all of the many options offered for these flange systems.

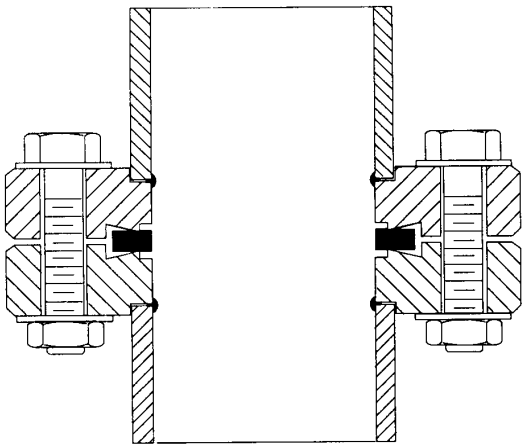


Figure 3 - CF Flange System
Illustration courtesy of Kurt J. Lesker Company.

CONSTRUCTION DETAILS

Start with a piece of ½ inch thick aluminum plate at least six inches square. A better dimension is eight inches on a side. Plate may be bought new from suppliers such as Shapiro Supply Co. (1259 Delaware

Ave., St. Louis, MO 63133) or surplus from a scrap yard. If you have to cut a piece to size, use a band saw or, as I have done, use a metal cutting abrasive disk in a circular saw and true up the edges with a good file.

Additionally, you will have to procure the following:

One 2 inch PVC pipe flange (plumbing store)
One 1 inch copper tee
Short lengths of 3/4 and 1 inch copper tubing
Short piece of 1 inch OD x 1/16" wall stainless steel tubing (brass or copper will do)
6 inch diameter x 1/16 inch thick Viton gasket (see text)
One brass KF25 center ring
One brass KF25 flange for 1-1/8 inch tube
One 2-3/4 inch CF flange, 1 inch bore, stainless, untapped
One ACE addition funnel catalog #5822-15
One steel washer for 1 inch bolt
Misc. hardware available locally

The hold down for the funnel is made from the 2 inch PVC flange (6" OD). This must be modified by enlarging the opening to allow a tight sliding fit over the funnel. I did this by cutting a hole in a piece of wood just large enough for the lip on the flange to fit and then, by clamping this in my drill press and rotating the flange by hand, cutting the hole with a ¼" end mill. This required several passes, the last leaving a bit of a web in place to keep the center core and the outer (saved) part of the flange together and in alignment. The core was then removed by cutting the web with a small file.

With a compass, square and scribe mark the plate for the 1 inch center hole, the PVC flange bolt circle (4-3/4 inch diameter), and the KF hold down screw circle (2" diameter). The first two are drilled through. I used a hole saw for the 1" hole, drilling a pilot hole first and then drilling half way through from each side with the hole saw. Be careful not to drill the KF hold down screw holes all the way through!

You may also want to drill other holes around the outside of the plate for mounting the plate to your system. As it is important to be able to have easy access to both the topside and the underside of the plate, I made provisions to hold the plate about 18 inches above the top surface of my vacuum system using two pieces of 5/8 inch threaded rod.

Start the plumbing with the copper tee. First attach the CF flange as shown. Begin by driving the 3/4" copper tube through the collar made from a ½" long piece of stainless steel tube as shown. Having this collar provides more strength to the tube/flange joint. The copper tube should extend through the collar just enough to come flush with the inner surface of the flange. Solder with tin/silver solder making sure that there is a good fillet on the inside of the flange. Complete this arm of the tee by coupling the other end of the tube to the tee using a copper bushing and then solder.

Find a steel washer that will go over the stub of the KF flange. A 1 or 1-¼ inch washer modified with a bit of filing will do fine. Mark and drill the washer to match the four holes you drilled in the base plate for the KF hold down. These should clear a 10-24 bolt. Then solder a short length of 1 inch copper tube in the upper arm of the tee. Place the washer over this tube and then solder a KF25 flange in place.

Finally, solder a length of 1 inch copper tube on to the side arm of the tee. The length will be dictated by how you couple this into your vacuum system. Although not shown, a KF25 flange would be advisable here.

Clean up the manifold by scrubbing in soapy water with a mild abrasive. 3M's abrasive cloth is excellent for this. Do not use steel wool as steel particles will get entrapped in the copper and brass fittings. Dry everything out in the kitchen oven at around 250° F. From this point, avoid getting fingerprints on the metalwork.

Place the center ring in the KF flange and affix the manifold to the base plate with the four 10-24 bolts. The glass chamber will require a gasket. I obtained a six inch disk of Viton from Kurt J. Lesker Co. (not a catalog item but supplied at nominal cost on special order) and cut a 3-3/4 inch hole with a gasket cutter. The four bolt holes were made with a gasket punch.

V. PROJECTS

As noted at the outset, this is only the foundation for a series of future articles on beam devices. The first article will deal with a multiplate chamber electron & ion beam source, the principles of which are discussed elsewhere in this publication. That device will be designed to be coupled to the CF flange with the glass chamber serving as an experiment chamber. Also in the works are a "saddle field" ion source (a relatively new, and simple, source; sort of like a Philips source but without the need for a magnet) made from plumbing fittings, and a demountable 100 - 200 keV particle accelerator. Also, we'll have some simple ideas for cold cathode as well as thermionic electron emitters.

*Originally published in Volume 2, Number 4. Some of the devices mentioned in Section V are covered in this compilation. Others are in later issues of t*BJ. *And, some are still in the works.*

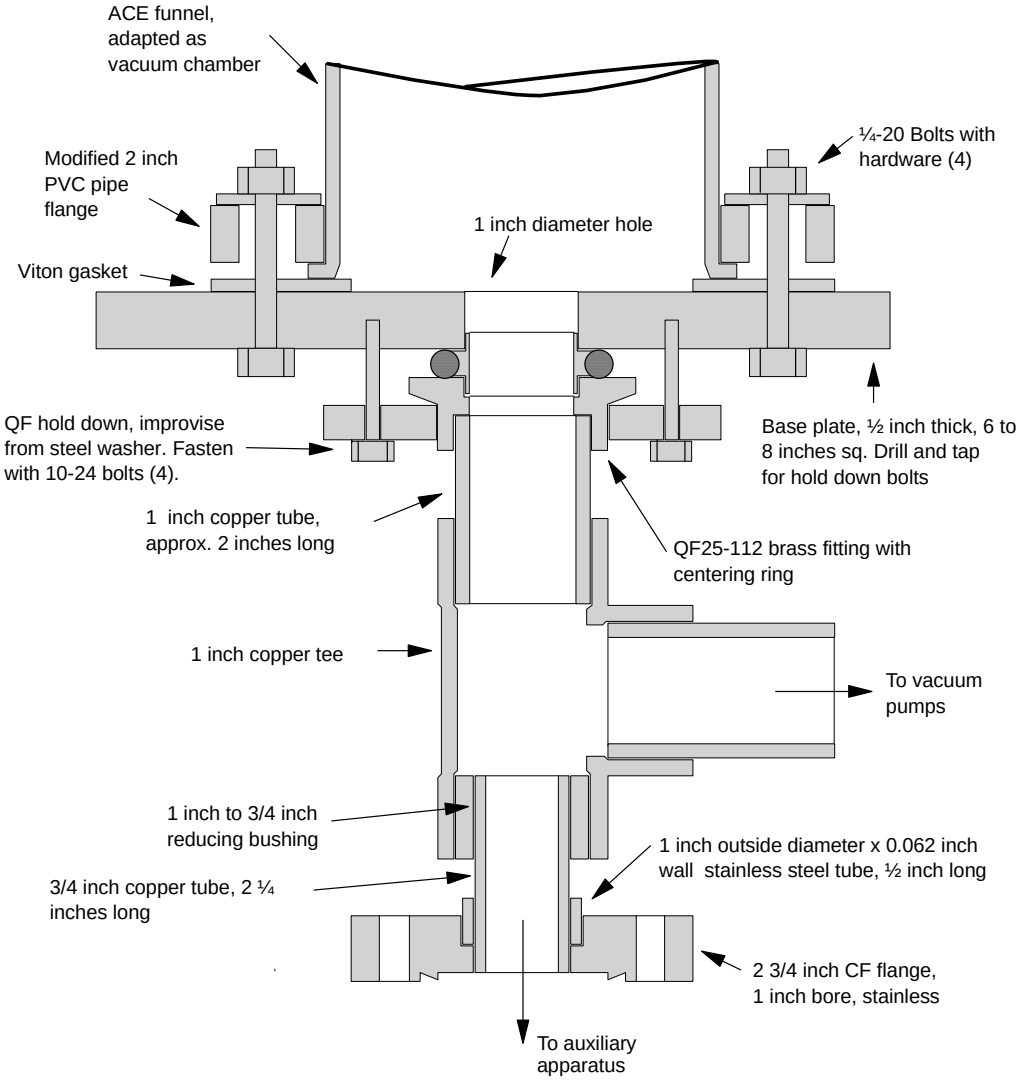


Figure 4 - Assembly Detail of Beam Experiment Apparatus

A Kit of Components for Conducting Gaseous Discharge and Electron Beam Experiments

Steve Hansen

I. INTRODUCTION

For the amateur, the gaseous discharge tube (e.g. the Geissler type tube and its derivatives) provides an interesting area for experimentation. This is also usually the first area that the amateur explores before proceeding on to more complex experimental apparatus. While ready-made tubes are available, they are frequently only for sale to educational institutions, generally expensive, and usually permit little in the way of 'tinkering' with the design. (For example, Electro-Technic Products in Chicago produces a variety of Geissler and Crookes tubes for lecture purposes.) The amateur usually begins by piecing together crude replicas of such tubes with bits of standard glassware, rubber stoppers, and metal rods.

The purpose of this article is to present a simple apparatus which can be easily reconfigured to replicate many of the early gaseous discharge and electron beam devices ranging from the simple discharge tube to the Crookes type tube and on to the Braun tube (the precursor of the oscilloscope cathode ray tube), cold and thermionic cathode x-ray tubes, and even a simple triode vacuum tube.

At the core of this apparatus is a simple tee shaped manifold which is made mostly from copper plumbing fittings. To make the various configurations, a variety of glass tubes and bulbs can be fitted to two O-ring compression fittings which are arranged in-line with each other. (The third arm of the tee connects to the vacuum pump.) A 5/8 inch compression fitting is typically used as the holder for the cathode. The other fitting, 1-1/2 inches in diameter, holds the various discharge tubes and bulbs. As an aid to fitting electrodes to the tubes and bulbs, appropriately placed threaded O-ring fittings are installed in the glassware (these are 11 mm *ACE-Threds*, described elsewhere). Armed with the manifold, a 5/8 inch od glass electrode holder, a bulb or two, and some miscellaneous hardware, many interesting devices may be created.

II. THE MANIFOLD

The metal manifold is shown in Figure 1. The central component is a 1½ x 1½ x 1 inch copper plumbing tee. At one end is a 1½ inch copper slip joint trap fitting (like what goes under your sink). I'll note that this particular type is somewhat hard to obtain given the broader use of threaded brass and plastic fittings, neither of which is acceptable. The figure shows a female fitting. A male fitting may also be used, just eliminate the intermediate length of tubing.

As these fittings are designed to use plastic ferrules in applications where there is almost no pressure differential, the fitting has to be modified a bit. First, throw the ferrule out. Then fabricate a 3/16 to 1/8 inch thick metal (aluminum or brass) or hard plastic bushing with an outside diameter that makes a loose fit in the fitting's compression nut. Finally, bore a concentric 1½ inch hole in the bushing. The bushing, when placed over an O-ring (use a Buna ring with a 3/32 inch cross section), will provide an even pressure to the O-ring producing a vacuum tight seal. This whole fitting can be made for under $12, considerably less than the $50 or so that a commercial brass 1½ inch vacuum compression fitting would cost.

The first step in assembly should be to solder (use 4% silver - tin 'hobby' solder) the trap fitting to the tee. As the sleeve on the fitting is short, I would recommend that a short spacer made from 1½ inch 'type L' copper water tube be placed below the trap fitting to keep the threaded portion above the end of the tee. You might also want to leave the burr from the tubing cutter on the lower end of the sleeve. This will help to center the glass tube which is sized a few thousandths under the sleeve ID. (The O-ring centers the tube at the outer end.)

At the other end of the tee, solder a 1-½ inch to ¾ inch copper reducer. Then insert and solder a standard brass vacuum compression coupling sized for 5/8 inch tubing. (This *could* be adapted from a 5/8 inch hardware store brass compression fitting. However, the standard component isn't that expensive and the machining that would have to be done to the hardware store item might negate any savings.)

To complete the manifold, attach a short (2 to 3 inches) side-arm of 1 inch copper water tube to the remaining leg of the tee. Any of a variety of strategies can be used to connect to the vacuum system but I would recommend a brass QF25 (KF25) fitting for low cost and the ability to rotate the joint.

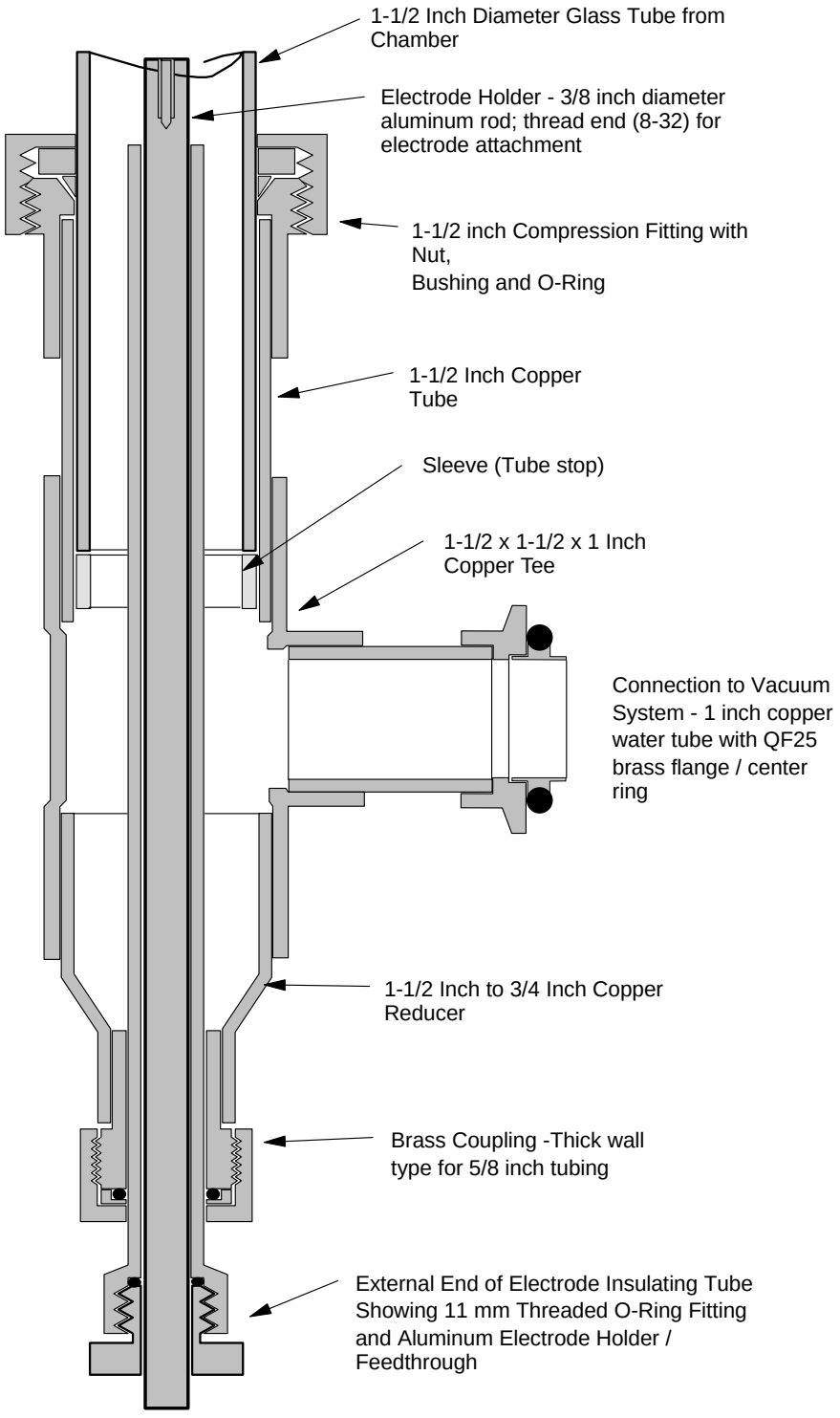


Figure 1 - Manifold Assembly Details

III. OTHER COMPONENTS

In Figure 2 are shown several components. One piece of glassware that is common to all of the configurations is a length of 5/8 inch borosilicate tubing, open on one end and fitted with an 11 mm *ACE-Thread* at the other. This is used to shield the 3/8 inch diameter rod electrode from the manifold in applications where that is necessary (e.g. in a Braun tube where the rod, the cathode, would be negative with respect to the manifold, which would serve as the accelerating anode).

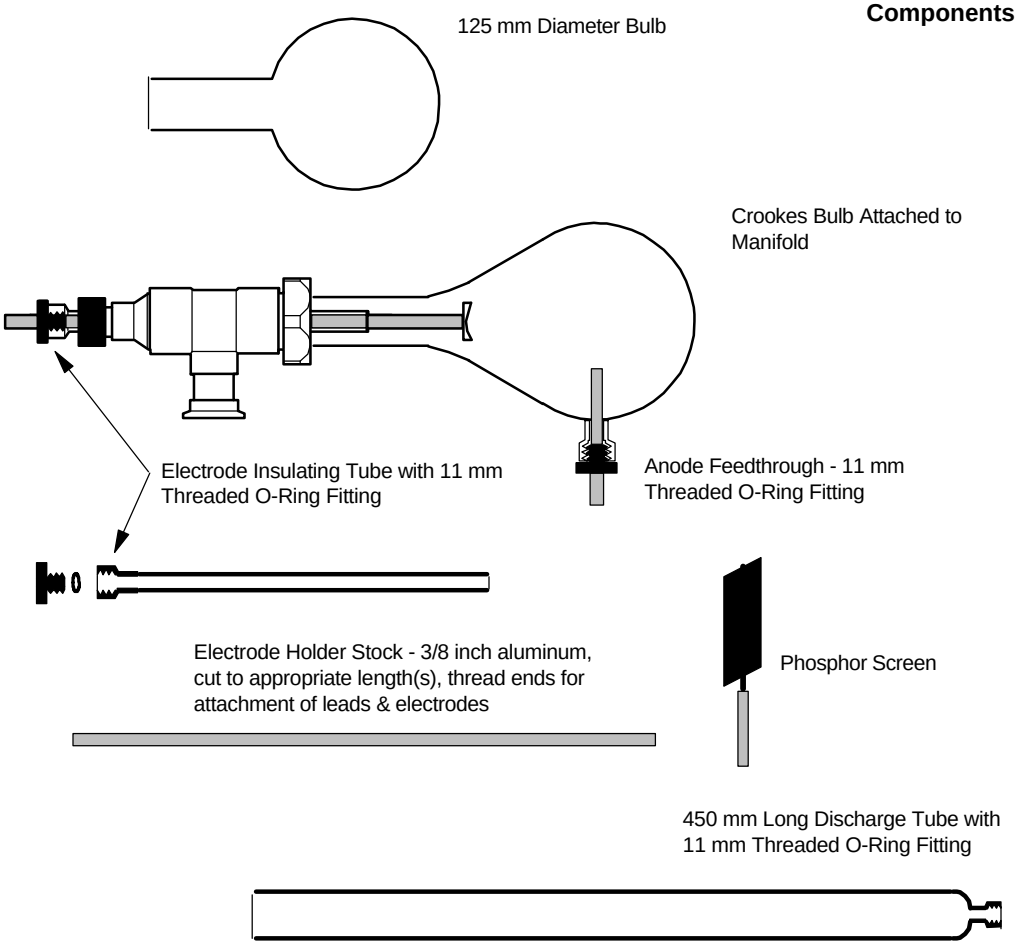
Three other pieces of borosilicate glassware are shown. One is a 125 mm diameter bulb which can be used for 'plasma sphere' and perhaps for Tesla 'button lamp' experiments. A straight length of tubing is shown for discharge tube experiments. Perhaps the most useful piece is an egg shaped bulb with a side feedthrough. This can be used for the construction of a Crookes tube (as shown), a cold cathode x-ray tube, or (with a pair of metal apertures installed in the manifold, external deflection electromagnets, and an internal phosphor screen) as a replica of Braun's cathode ray tube.

By replacing the cold cathode with a thermionic emitter (i.e. a filament) it would be possible to make a Coolidge type (hard vacuum) x-ray tube. At much reduced voltage and with a wire mesh grid inserted between the filament and the plate electrode it would also be possible to make a simple triode after the form of von Lieben's tube of 1910. Finally, a number of other configurations may be easily implemented with these basic components including molecular beam devices and flash x-ray sources.

Originally published in Volume 3, Number 1.

For a while, the author was supplying kits of these components. These are no longer available but I can recommend sources for the glassware.

Figure 2 - Basic Kit Components



Part 3: Projects

How to Make Buckyballs

Greg Konesky

Abstract: How, with simple apparatus, an amateur has duplicated the technique developed by Huffman and Krätschmer to synthesize and isolate that soccer ball shaped molecule of carbon, C_{60} , otherwise known as Buckminsterfullerene or Buckyballs..

I. INTRODUCTION

Buckyballs, also known by the more proper term Buckminsterfullerine, are hollow, cage-like carbon molecules. The buckyball family may have as few as 32 carbon atoms but the upper limit continues to be extended with such forms as nanotubes and bucky-onions (concentric spheres of buckyballs). The family of these peculiar molecules are known as fullerenes.

Buckyballs have been made to enclose virtually every atom on the periodic table and the characteristics of these modified fullerenes range from insulator to semiconductor to superconductor. They can also act as "soft" organic ferromagnets, act as frequency doublers and optical limiters, be compressed to form diamond, or act as a foundation for diamond thin films on a variety of substrates. Buckyballs are also being proposed as massive projectiles for molecular accelerators: the molecules have been accelerated to tens of MeV with their structure remaining intact. Best of all, they are relatively easy to make.

II. THE REACTOR

The simplest synthesis route uses a carbon arc within an inert atmosphere. The arc throws off a fine black soot which contains several different types of fullerenes. C_{60} predominates with a lesser amount of C_{70}. I've used both helium and argon atmospheres with equal success. The pressure that the arc operates in should be within a range of 100 to 300 Torr, although this does not appear to be too critical.

The electrical power supply for the arc does, on the other hand, have to be quite substantial. This must be able to provide 100 or more amps at 15 to 20 volts. AC or DC appears to work about the same. The former is

Various forms of carbon such as graphite and diamond have been known for millenia. Very recently a new form has been discovered. In 1985, a group at Rice University realized that a new molecule consisting of 60 atoms of carbon could be produced by the laser ablation of graphite in an atmosphere of helium. This group proposed that the shape of the molecule had a very special structure. Mathematically, the shape is a truncated icosahedron. In more common terms, this is the shape and layout of a soccer ball. The molecule was termed buckminsterfullerene in honor of the architect Buckminster Fuller , who became famous for his work with geodesic dome structures.

Until 1990 when Donald R. Huffman and Wolfgang Krätschmer developed a methods for both producing fullerenes from a carbon arc operated in an inert atmosphere and then being able to separate the fullerenes from the other forms of carbon in the residue (mostly graphite), only miniscule amounts of fullerines had ever been isolated. As a result of this breakthrough, research into the properties and potential uses of the fullerenes has proceeded at a very fast rate.

As this article shows, the methods are simple and amenable to use by amateur researchers. - *Ed.*

preferable as it avoids the use of large, expensive rectifiers, filters, etc. I used two paralleled transformers (obtained from C&H Sales Co., 2176 E. Colorado Blvd., Pasadena, CA 91107) with 240 volt primaries and secondaries rated at 24 VAC at 17 amps. During operation, the transformers are heavily overloaded but, as the entire synthesis only takes about 30 seconds, the transformers only warm slightly. The conductors are made from copper braid 1.0 inch wide and 0.1 inch thick. These get fairly warm in operation. The electrical feedthrough is made of ¼ inch solid copper rod. The insulator for the feedthrough should be made of a heat resistant material as this conductor gets quite hot. Only one feedthrough is needed as the other conductor is connected to the metal vacuum chamber baseplate.

Some sort of feeding mechanism is needed for the carbon arc electrodes to initially allow the arc to be struck and then to feed the carbon into the arc as the electrode becomes consumed. This is very fast: typically two inches of ¼ inch electrode in 30 seconds.

The first setup that I used had one carbon rod fixed. The other was clamped to a copper rod which could be rotated. A simple grommet, liberally coated with vacuum grease, acted as a rotary feedthrough as well as an electrical insulator. One connection was made through the metal baseplate. The other was through the copper rod. The copper rod was rotated until the carbon electrode tangentially made contact with the fixed electrode. As the electrodes were consumed, the copper rod was rotated slightly and continuously to maintain the arc. At some point the electrodes would erode to the point where contact would no longer be possible and the arc would extinguish.

Aluminum electrode holders were originally made from two pieces of 1 inch by ¼ inch bar stock. but the heat from the arc sometimes melted these. Copper bar stock ¾ inch by ¼ inch made a better choice.

This apparatus is depicted in Figure 1A. My latest version uses a linear feedthrough which allows several inches of carbon to be consumed in a single run. The electrical feedthrough for the "hot" lead is now fabricated using a ceramic insulator to eliminate the heat deterioration experienced with the rubber grommet. This new configuration is shown in Figure 1B.

Since considerable heat is given off during the synthesis, all materials in the reactor must be able to withstand a reasonably high temperature. I used BUNA-N for the bell jar gasket. The bell jar itself was a Kimax glass process pipe endcap about 6 inches in diameter and 6 ½ inch high. These were available from C&H Sales for $15 but they are no longer in the current catalog (*Ed. note - new these caps may be had from ACE Glass at 1430 Northwest Blvd., Vineland, NJ 08360, for about $75; other surplus alternatives or clever use of Pyrex bakeware would be usable.*)

Any air in the reactor will combine with the hot vaporized carbon, reducing fullerene yield. For pumping, I used a tandem pair of refrigerator pumps. Several cycles of evacuation and backfilling with inert gas were performed before operating the reactor. The initial pressure within the reactor should be under 300 Torr. Otherwise, the heating of the apparatus by the arc will raise the pressure, perhaps above atmospheric. This would break the seal on the bell jar and allow air into the reactor.

As the light from the arc will initially be intense and rich in ultraviolet, be sure to wear welder's goggles. However, a thick layer of soot will soon form on the chamber. This will reduce transparency to the point that the arc will no longer be visible. This also means that, depending on your method of feeding carbon rods into the arc, you may have trouble finding the other electrode once the soot gets too thick.

The carbon rod electrode feeding strategy is to apply just enough force to consume the electrodes as rapidly as possible but not so much as to fuse them together. If you try to feed the electrodes too fast, they will weld together, extinguishing the arc. When this happens, simply back off with sufficient force to break the weld and then restrike the arc. The whole process of feeding the rods does take some practice but mistakes are of little consequence. On the other hand, the feeding strategies which are published in the scientific literature typically take an hour to consume what I use in 30 seconds. The professionals do get about a 10% yield of fullerenes in the soot. My simpler method gets about a 3.5% yield. With the short thermal pulse in the quick feed approach I also don't have to contend with the need to water cool everything.

III. SEPARATION

Once the reactor has cooled the soot can be collected. I use a small paintbrush to sweep the soot into a large beaker. As the soot is very fine some ventilation should be provided to draw away airborne particles.

Most of the soot consists of unreacted graphite. To separate the fullerenes, we can take advantage of the fact that fullerenes are soluble in solvents such as toluene and benzene. When using these materials keep in mind that both are highly flammable and toxic. Perform the separation process in a well ventilated area. Having a fire extinguisher at hand is not a bad idea either.

For every gram of soot I use 100 ml of solvent even though the solubility of C_{60} is 5 milligrams per milliliter of toluene. The soluble materials will dissolve faster if

heat is applied. If you elect to do this, DO NOT use a direct flame or other source of heat. Acceptable heat sources include electrically heated steam or oil baths. Typically, I bring the black liquid up to boiling and then allow it to sit for a few minutes. This is followed by filtration. The undissolved soot will quickly clog fine filter paper so I first use coarse paper followed by a finer one.

The orange to brown colored solvent can then be evaporated slowly making sure that the vapors are well ventillated. If you have distilling apparatus that is compatible with the solvents you may want to reduce the volume of the of the solution first. The condensed liquid solvent can then be used for future extractions. The solid remaining is mostly C_{60} and C_{70} in a ratio of three to one with some smaller fullerenes in trace amounts. The sludge remaining in the filter paper is mostly unreacted graphite, but there are also insoluble, large fullerenes. These can include molecules to C_{240} and above plus some cylindrical nanotubes or "buckytubes", as well as other forms of fullerene not yet identified. The sludge will also have some C_{60} and C_{70} that did not go into solution. Subsequent extraction and filtration will increase the total yield.

These are the basics to get you started having fun with fullerenes.

Originally published in Volume 2, Number 2.

Some Published Properties of C_{60}

Density	1.7 g/cm^3 *
Crystal structure	Face-centered cubic
Interatomic distance within a molecule	1.40 & 1.45 Å **
Cage (ball) diameter	7.1 Å
Distance to nearest neighbor	10.04Å

Notes:

* In contrast, diamond is 3.5 g/cm^3 and graphite is 2.3 g/cm^3.

** Faces of C_{60} are comprised of both hexagons and pentagons. Thus the two interatomic distances.

Some Additional Notes and Suggestions for Further Reading

The first paper which describes fullerenes was the one produced by Richard Smalley's Rice University team (Heath, O'Brien, Curl, and Smalley in collaboration with Harry Kroto of the University of Sussex) and published that year in the journal *Nature* (**318**, 162). The process used was laser ablation of graphite in a helium atmosphere. Although a group at Exxon Laboratories had, the year before, observed similar large clusters of carbon atoms, Smalley's group was the first to suggest the special shape of the C_{60} molecule.

Krätschmer and Huffman began working with the the soot from a carbon arc in 1983 as part of a search for carbon particles which might explain some spectrographic properties of interstellar dust clouds. With the results of the Rice group's work, both began to realize that that their smoke also contained C_{60}. In May, 1990 Krätschmer suggested that C_{60} would be soluble in benzene. Huffman then demonstrated that this was the case and that reasonable quantities of the substance could be produced using this property as the basis for a method of separation. This work, which also involved graduate students Lowell Lamb and Konstantinos Fostiropoulosis, was described in a paper also published in *Nature* (**347**, 354, 1990).

An excellent review of Huffman's work on solid C_{60} is contained in an article written by Huffman and published in the November, 1991 issue of *Physics Today*. Included is a discussion of the crystal structure of C_{60} along with one of the first micrographs of crystals grown from a benzene solution. (Pure C_{60} forms needle shaped crystals about 20 microns long.) This article also includes a good bibliography of papers on fullerenes.

One area of great interest with fullerenes is the potential for new superconducting materials. By doping C_{60} with various alkali metals, a group at AT&T Bell Labs has discovered that normally insulating C_{60} can become superconducting. Potassium doping results in a superconducting transition (T_c) temperature of 18 K. Other alkali dopants have resulted in T_c's as high as 42.5 K. An overview of this work is contained in an article by Arthur Hebard in the November, 1992 issue of *Physics Today*. *- Ed.*

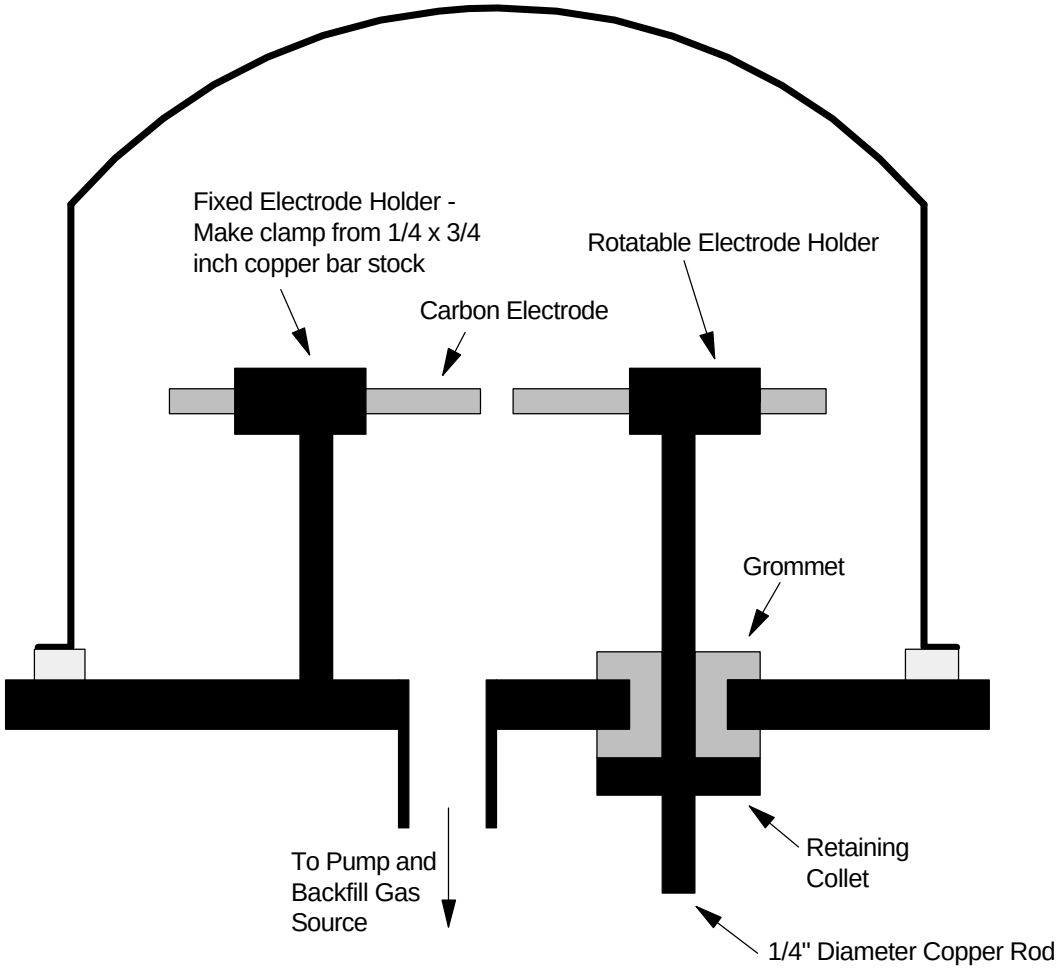


Figure 1A - Fullerene Synthesis Apparatus with Rotating Electrode Holder

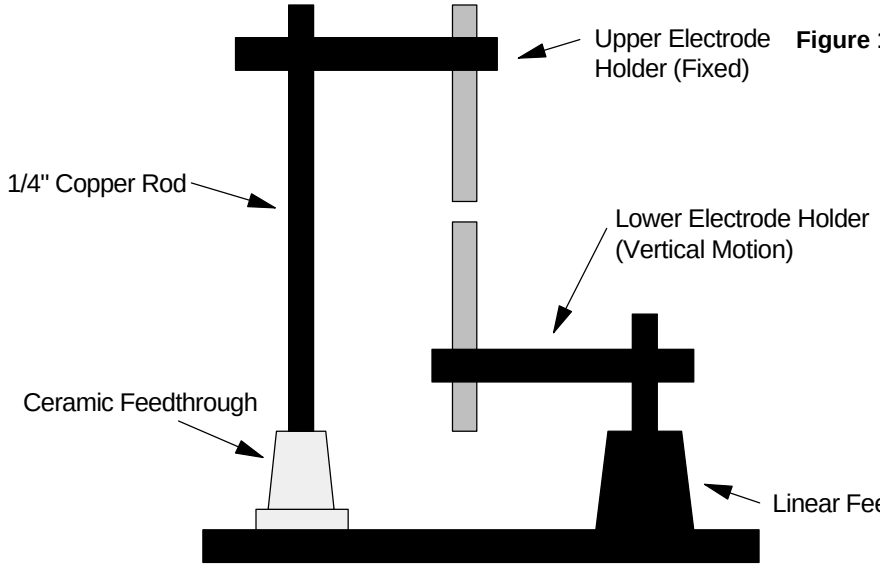


Figure 1B - Modified Electrode Holder Using Linear Motion Feedthrough

Build a Pair of Magdeburg Hemispheres

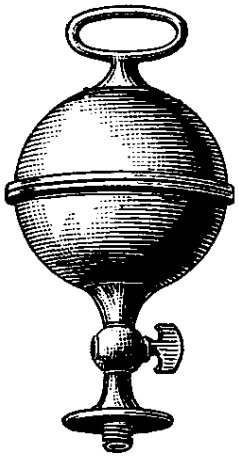
Steve Hansen

Otto von Guericke (1602-1686) was trained as a lawyer. Living up to his family's expectations, he spent his life in politics, serving in a variety of posts connected with his hometown of Magdeburg, Germany.

Guericke, being a man of several dimensions, developed a strong interest in natural science. One area that fascinated him was the issue of the definition of space. Specifically, there was a debate which had existed since the time of Aristotle. We all know the saying "nature abhors a vacuum" ... *horror vacui*. This phrase had been taken quite seriously for a couple of millenia. Were the classical *plenists* correct in their belief that space always had to be occupied by something? Or, were the radical *vacuists* correct in their belief that space could be occupied by nothing - a vacuum? Guericke decided to settle the debate once and for all.

Now, whenever a lawyer/politician undertakes to work on something like this, we tend to be suspicious of whatever might result. However, Guericke had also studied mathematics and engineering while attending Leiden and so he was fairly well equipped to undertake the task. (Nonetheless, I suppose that he would be classified as an amateur scientist.)

As is often the case with experiments, his first couple of attempts failed. Starting in 1647, he first tried to demonstrate that air could be removed from a container without an ensuing implosion. That container, a wooden keg, leaked. (Of course, the plenists felt that this proved that they were right ... the air rushed in to ensure that there could be no void.) Next he tried to seal the keg by immersing it in water contained in a second, larger keg. Water leaked in. (Again the plenists claimed victory.) Still persisting in his quest for the truth, he had a copper sphere constructed. It imploded. (More ammunition for the plenists!) Well, the fourth time never fails and the persevering Otto had a new vessel made, this time with stronger walls. Success at last! The vessel was evacuated with no leaking or implosion.



Magdeburg Hemispheres for Classroom Use

Several variations on this experiment were made. The most important involved placing the exhaust port at various points on the sphere. This showed that the air expanded to fill the volume as it was pumped (i.e. not sinking to the bottom as would happen if air behaved like a liquid). This showed the elastic nature of air.

Of course, the experiment for which Guericke is now most well known was the demonstration done in public at Magdeburg in 1657. Here he used two well fitting hemispheres which were pumped of air. Each hemisphere was attached to a team of horses and the force of the atmospheric pressure holding the hemispheres together was so great that the horses could not pull them apart. (Was this the beginning of the oxen pull event at county fairs?)

Subsequently, Guericke continued his experimental and philosophical studies in addition to serving as mayor of Magdeburg. In 1666 he was elevated to the German nobility (hence the *von* being added to his name).

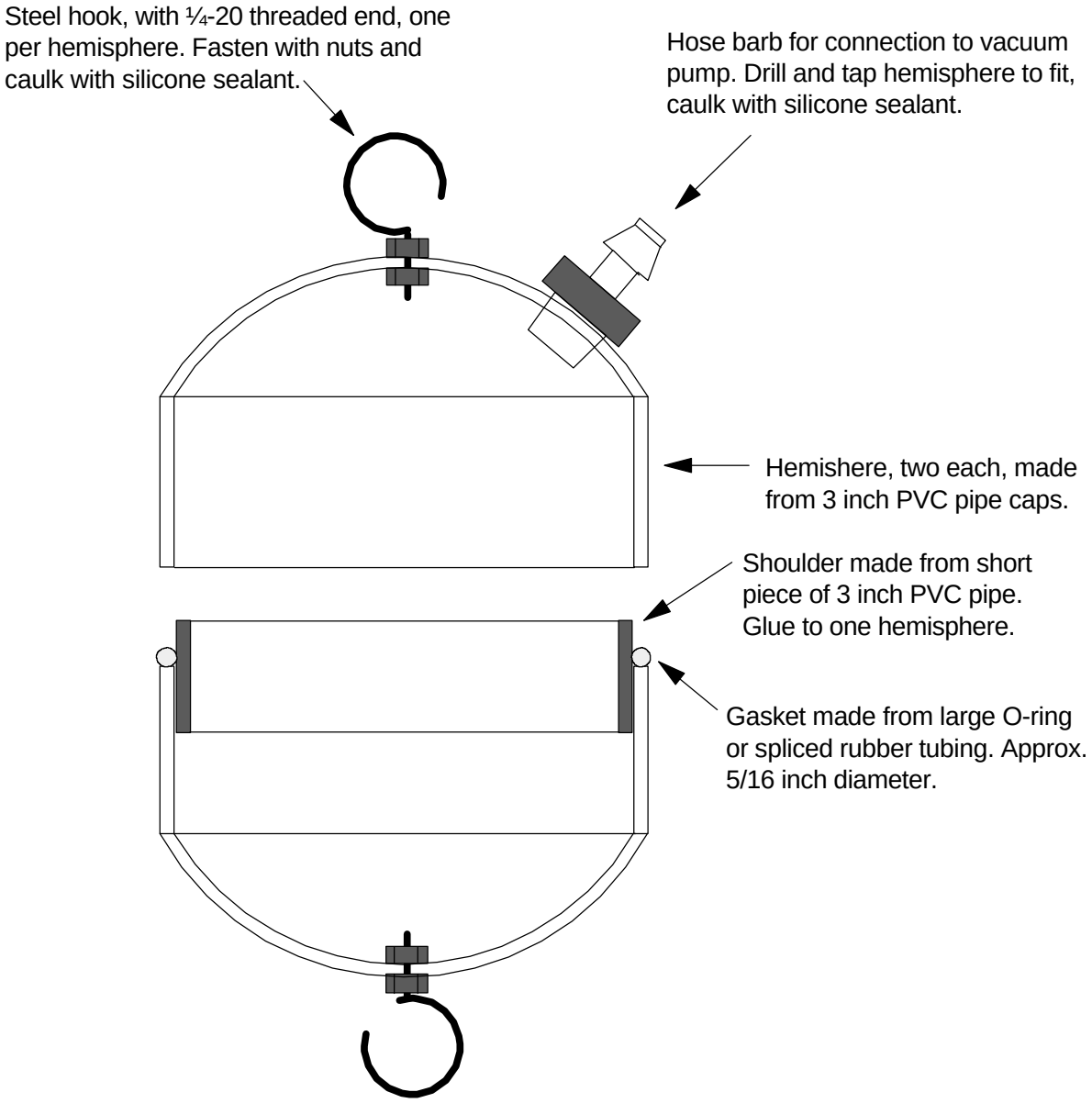
Magdeburg hemispheres have continued their existance as a physics demonstration tool and most everyone who has taken high school or first year college physics has been exposed to a miniature replication of von Guericke's famous experiment. A typical classroom set of hemispheres, ca. 1900, is shown at the left.

The figure on the next page shows a simple set of hemispheres which can be fabricated from a few dollars worth of PVC plastic pipe fittings and miscellaneous hardware. The hemispheres themselves are 3 inch end caps of the rounded variety. A short piece of 3 inch pipe is glued into one of the hemispheres to hold the two caps in alignment. The gasket is either a large O-ring, if one can be found, or it may be fabricated from a short length of small diameter soft rubber tube, ends cut on a bias and glued with rubber cement. A screw eye must be affixed to each hemisphere with the holes sealed with a suitable sealant. Silicone caulk is a satisfactory choice. Finally,

a plastic hose barb has to be installed in a threaded hole in one of the hemispheres. Seal the threads with caulk.

A useful set of accessories would include a simple bourdon type vacuum gauge which can be used to measure the degree of vacuum in the apparatus and a spring scale for measuring the force required to separate the halves at varying degrees of vacuum.

This article was originally presented in Volume 2, Number 4.



Simple Set of Magdeburg Hemispheres

Production of Very Low Temperatures

F. B. Lee

This article originally appeared in a collection of vacuum related projects which was published by the firm of Morris & Lee. My thanks to Frank Lee for permission to reprint this material. - Editor

I. INTRODUCTION

Temperatures within a few degrees of liquid air temperatures may be obtained by evaporating dry ice at low pressures. Dry ice (solid carbon dioxide) has a sublimation temperature of -78° C (-108° F) at atmospheric pressure. At a pressure of 1 mm Hg (1 Torr) it sublimes at -135° C. At a pressure of 1 micron, the temperature is -166° C. For comparison, liquid oxygen boils at -183° C. If liquid nitrogen is substituted, evaporation at 1 mm Hg will produce a temperature of -226° C (or 47 K).

Unfortunately the heat transfer from solid carbon dioxide to an experimental chamber is quite poor and it is necessary to immerse the test object well into the crushed dry ice and allow ample time for equilibrium.

The temperature-pressure relationship for carbon dioxide may be found in the *Handbook of Chemistry and Physics* or similar reference books. Detailed information on the subject of thermodynamics may be found in just about any physical chemistry textbook.

II. APPARATUS FOR LOW TEMPERATURE EXPERIMENTS

The following materials are needed to assemble the apparatus shown in Figure 1:

Two canning jars with lids
8" of 8 mm glass tubing, closed at one end
Rubber one-hole stopper to fit above tubing
Appropriately sized "tin" cans
Aluminum foil
Rubber vacuum hose
Copper tubing
Wire mesh screen
Flake sodium hydroxide (household lye)
Dry ice
Mechanical vacuum pump

Assemble the system as shown in the figure. The double walled container is made from two tin cans or paper. The two containers are held concentric by lightly crumpled aluminum foil which serves as a radiation shield. The assembly is then placed in a canning jar and supported by more aluminum foil on the sides and bottom. A double foil cover is placed over the dry ice in the interior. The lid for this has a copper tube connection for exhausting and a central hole for admitting the glass cold chamber. The cold chamber is a piece of glass tube closed at one end and inserted through a rubber stopper. A tight joint is obtained by greasing the stopper where it joins the lid.

In order to reduce the gas load on the pump, a trap may be installed as shown to capture the bulk of the carbon dioxide released. A pressure release is not necessary if the cold chamber is installed as shown. If the cold chamber is fastened to the lid, some sort of pressure release device should be employed.

III. RELATED APPLICATIONS

Low temperature chemical reactions: The reaction

$$NaHCO_3 = Na_2CO_3 + H_2O + CO_2$$

which normally takes place at 270° C will take place at room temperature in a vacuum of about 1 mm Hg. Other reactions which take place at much lower temperatures in a vacuum are the following:

$$Cr_2O_3 + 3C = 2Cr + 3CO$$
$$CaC_2 + 2\,NaCl = CaCl_2 + 2Na\ (vapor) + 2C$$
$$2MgO + CaO + Si + 2Mg\ (vapor) + CaSiO_3$$
$$FeO + C + Fe + CO$$

These reactions are of great commercial importance. Any reaction evolving gas will proceed at lower temperature in vacuum.

Distillation at low temperatures: Often a heat sensitive material may be safely distilled by using a low pressure and correspondingly low temperature. Water may be distilled at room temperature if the pressure is reduced to less than 30 mm Hg. Vitamins A and D, turpentine, tar, lubricating oil, naphthalene, and some vegetable shortenings are products using vacuum distillation.

This article was originally presented in Volume 2, Number 4.

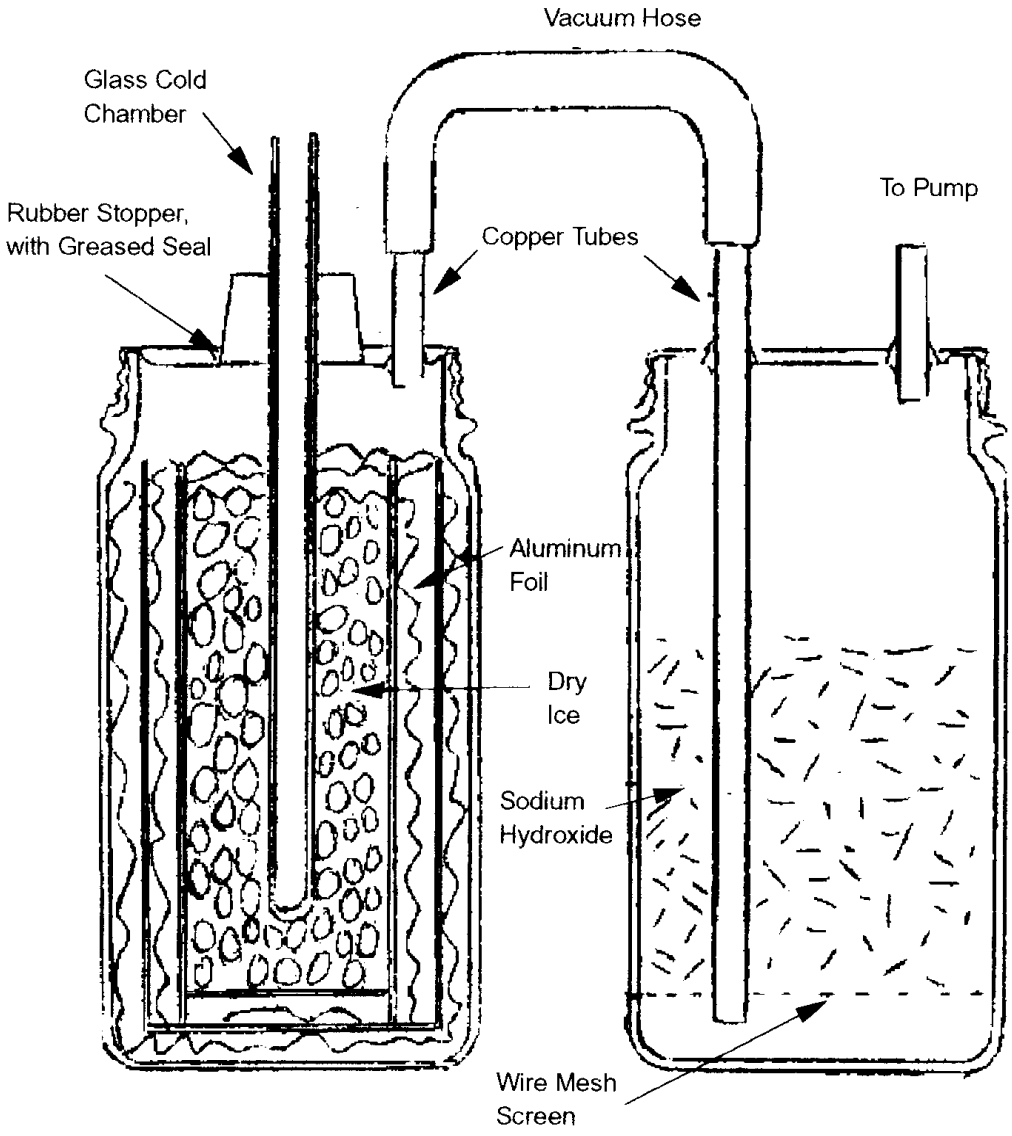


Figure 1 - Apparatus for Generating Very Low Temperatures

Introductory Vacuum Experiments

Franklin Lee

Here are several basic experiments that were designed to be used with a simple water aspirator that was supplied by the firm of Morris & Lee. Glass and metal water aspirators are available from a number of sources and they provide a simple way to produce a level of vacuum that enables a number of interesting experiment to be performed, as this article demonstrates. This material is reprinted courtesy of the author. -Ed.

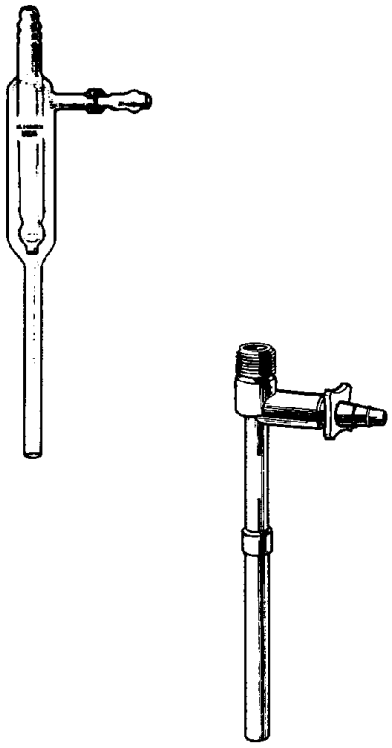
There are many interesting processes that can take place only in the absence of air and other gases. Examples of items that require processing in high vacuum for some key part include virtually all electronic devices, light bulbs, high tech alloys, many chemicals, mirrors, shiny plastic sheeting and even some wood products. It is difficult to find any industrial operation that does not use vacuum techniques somewhere.

There are two main fields of vacuum technique. Low vacuum depends upon the difference in pressure between our normal atmosphere and the pressure inside the vacuum system. This pressure difference has a function similar to any pressure difference. High vacuum is a term used to distinguish environments relatively free from any form of gas. We may wish to have an empty environment so that vapors of our choice can travel without interference (either chemical or physical) from other substances.

It is helpful to become familiar with some terms used to measure the degree of removal of gas from a certain volume. We can give a count of the number of molecules in a certain volume of space (say, one liter) or we can use the pressure that these molecules exert at room temperature. Under this system we can say that a liter of space at normal atmospheric pressure contains enough molecules to use a number containing 23 digits. That is a big number like the weight of the earth in lbs. or the number of bacteria in a large city. Usually we say that our atmosphere has a pressure of 760 millimeters of mercury or 760 Torr (after Torricelli and his barometer) or 760,000 microns or one atmosphere or any of an number of terms. Vacuum systems having pressures of one-millionth of an atmosphere are relatively common.

The device enclosed herewith is a small vacuum pump which depends on running water for its function. A small nozzle inside produces a small jet of water which sweeps gas out of a small chamber surrounding the nozzle. It is effective, inexpensive and will produce a vacuum of 10 to 30 Torr. The water vapor from the jet produces this pressure which is the smallest when the water is cold. This device is called an aspirator or a

Water Aspirator Vacuum Pumps



Water aspirator type pumps are routinely used in such laboratory applications as filtering. This illustration shows an all-glass pump at the upper left and a plastic pump at the lower right. The glass pump costs about $40 while the plastic pump, besides being unbreakable, costs about $6. Both types are available from labware suppliers such as Ace Glass, who supplied this illustration.

filter pump. It operates on any water pressure over 11 lbs. per square inch.

Tubing is needed to connect the water aspirator to the experiments. We recommend 1/4" od polyethylene tubing and a few lengths of 3/16" id vinyl tubing for connecting purposes. These are available in large hardware stores and plastics dealers. Modelling clay will come in handy for making air-tight connections. We also recommend getting a piece of Plexiglas tube 1" in diameter or larger.

Safety Precautions: Have you ever broken a light bulb? The atmosphere rushes inward violently and throws glass all over the place. Glass bottles should only be evacuated when enclosed in a protective screen or box. Pop and beer bottles were made for pressure and are quite safe under most circumstances and if free of cracks or scratches. Bottles larger than these are dangerous unless they have well rounded bottoms and even so should only be used in a protective container.

EXPERIMENTS

Behavior of Liquids in a Container: Partly fill a transparent pop bottle with alcohol and connect it to your vacuum line as shown in Figure 1. Apply a vacuum and observe. The alcohol will begin to boil. It will also become cold and frost may form on the bottle. All substances tend to turn to vapor in a vacuum, even glass and iron, although very slowly. All substances have the property of vapor pressure. Vapor pressure depends on the nature of the material and the temperature. The vapor pressure of alcohol is higher than that of water and at a given pressure it will boil at a lower temperature.

Warm water will also boil but the boiling stops when the water becomes as cool as the tap water that is driving the aspirator. One of the problems in obtaining very high vacuums is that the metal, rubber and oil in the systems all have significant, though small, vapor pressures.

Properties of a Nearly Gas-Free Environment: Place the following items inside a clean, dry transparent pop bottle: a wisp of cotton, a feather, thin paper or other light object and a bead, button paper clip or other relatively heavy object. (See Figure 2.) Invert the bottle and you'll see that the heavy object falls faster than the light one. Now evacuate the bottle and look again. Heavy and light objects fall at the same speed. By removing the air we remove air friction because there is almost nothing to impede the objects' free fall. As another example, place a few spoonsful of flour in the clean, dry transparent bottle. (See Figure 3.) If the bottle is shaken, the dust from the flour is slow to settle. Roll the bottle from side to side - the flour flows smoothly. Now remove the air and observe again. The dust settles quickly and the flour no longer flows smoothly.

Sound travels readily through solids, liquids and gases providing the gas is relatively abundant. This can be demonstrated by means of a small bell. If you are unable to find a bell small enough to put in a pop bottle, use a bottle with a wider mouth, such as a peanut butter jar. Punch a hole through the jar cover with a nail and pry this hole open enough to admit the tubing from your aspirator. Modeling clay placed around the hole will make a good seal. Attach the bell to the jar cover or to a wire frame inside the jar. (See Figure 4.) Put the cover on the jar and shake the jar enough to ring the bell. Apply vacuum and observe the sound as the pressure falls. In a good vacuum there is no sound transmission at all.

Low Vacuum Applications: There are devices that use vacuum not as a means of avoiding gas molecules but which use the pressure difference between the vacuum and the atmosphere. A suction cup is an example, a household vacuum cleaner is another. Since atmospheric pressure is a little over 14 lbs. per sq. inch, it doesn't take a large suction cup to lift a ton. If you visit a metal warehouse you might see large plates of aluminum weighing a ton or more being lifted by what looks like an electromagnet. Actually it is a large suction cup. To make a working model of this device from a metal jar cover, see Figure 5.

Aurora Experiment: Gases become electrically conducting at moderately low pressures. At room conditions, an electric spark of 5000 volts will occur in a gap of 3/16" or less. At lower pressures, the gap may be increased greatly. At about 5 to 10 Torr, the gap may be several feet. Neon sign tubes operate in this range.

A plastic tube 1" to 1-1/2" in diameter and a foot long is fitted with two electrodes (Figure 6) and is connected to a vacuum system. The transformer of a bug killer will produce 5000 volts at about 5 to 10 milliamperes. While the possible electric shock is usually just uncomfortable, use care anyway. Connect the tube to a vacuum system and proceed to pump out the air. A high voltage discharge through the air produces a reddish-purple glow. As the air is removed, water vapor will predominate. Water vapor produces a blue discharge, characteristic of hydrogen.

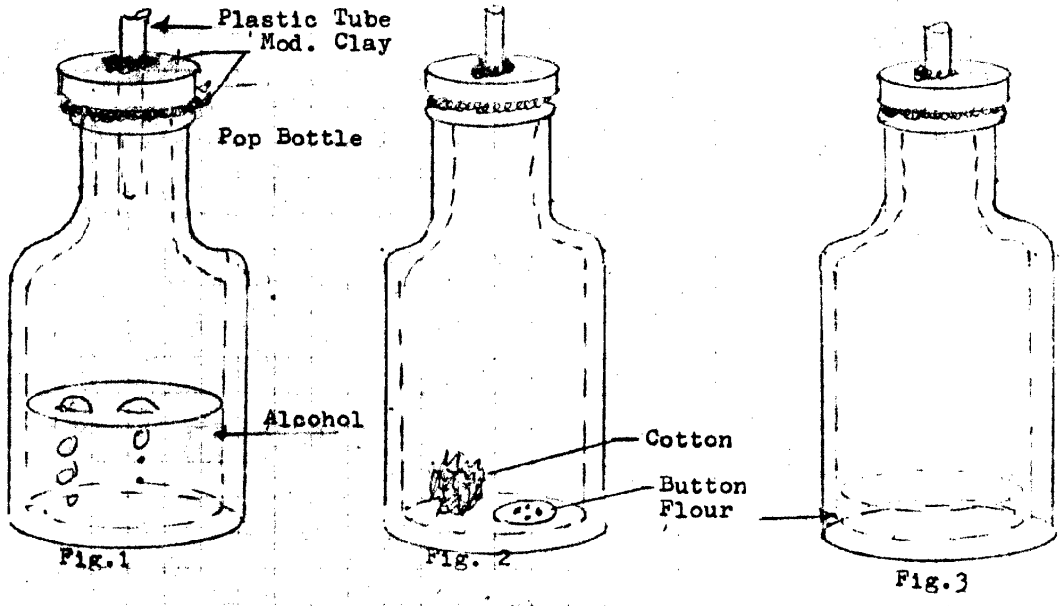
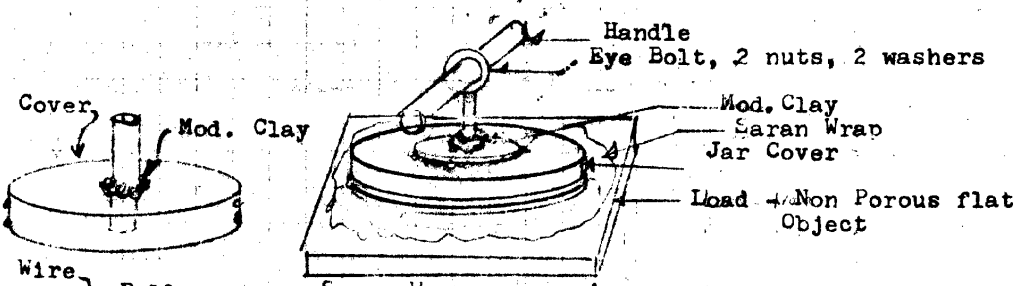


Fig.1 Fig. 2 Fig.3



Saran Wrap covers jar cover and load to seal leaks.
Use rubber gasket between cover and load if available

Fig 5

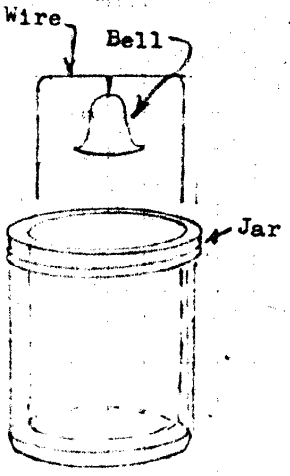


Fig 4

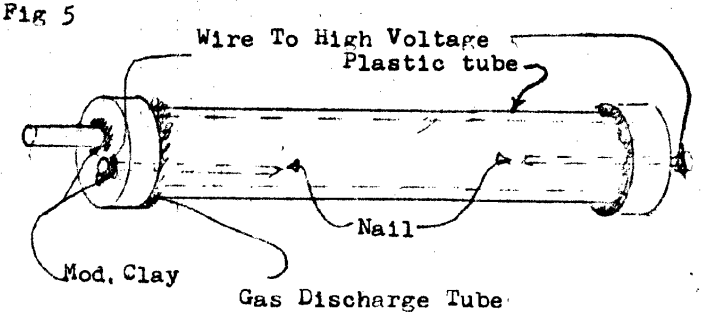


Fig 6

Constructing a Radiometer

F. B. Lee

This material originally appeared in a collection of vacuum related projects that was published by the firm of Morris & Lee. My thanks to Frank Lee for his permission to reprint this material. - Editor

A radiometer is a set of vanes, shiny on one side and blackened on the other, which is mounted in an evacuated vessel. When exposed to light or other radiation, the vanes revolve.

The first radiometer was constructed to settle the controversy regarding whether or not light exerted a force. Theoretically, a reflecting surface would experience a greater force than an absorbing one. The instrument was therefore made in the now familiar form. Unexpectedly, the opposite effect was observed. The blackened vane retreated from the light source. We now know that the black surface is warmer than the shiny one and that gas molecules and a pumping action takes place (thermal creep) that causes gas to flow from the cool side to the warmer side. This flow creates the "thrust" that moves the vanes. Later experiments in a much better vacuum have confirmed the pressure of light. It is very small.

A simple radiometer may be made as shown in Figure 1. Place two strips of aluminum foil together and skewer with a needle. Fold out the strips into a paddle wheel and glue to the needle if necessary. Blacken one side of the vanes heavily with a smoky flame so that the right hand vane is bright, the left black. Make a frame from the sheet metal of a tin can. Stamp a dimple to receive the needle point and a hole to guide the other end. Place the assembly in a bell jar of suitable size and evacuate to 60 microns Hg. A source of light will cause the vanes to spin.

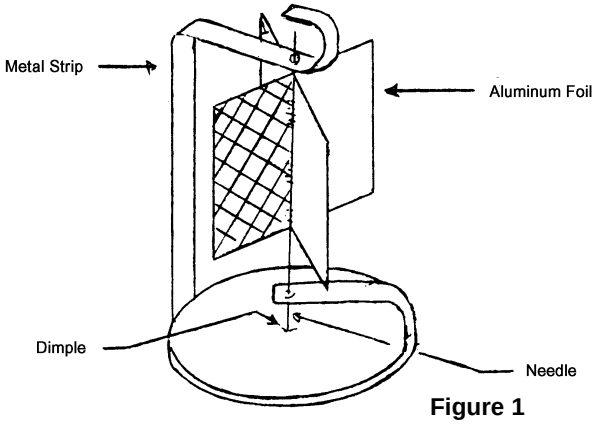


Figure 1

The rate of spin is influenced by the temperature difference between the light and dark sides and the

Some Further Commentary

The action of the radiometer is commonly thought to be as a result of molecules recoiling from the surface of the darker (warmer) side of the vane. As noted in the main text it is due to an effect called *thermal creep* or *thermal transpiration*. This effect is associated with molecular flow and begins to appear as the gas pressure gets well into the transition flow regime. A future issue of tBJ will have a more detailed discussion of the radiometer and the thermal transpiration effect.

Since what goes on within the radiometer involve a variety of phenomena other than thermal transpiration, Saul Dushman (see *Further Reading* at the end) noted the use of the radiometer to determine when the vacuum in an incandescent lamp had reached the required level. At the proper pressure, the vanes would cease to rotate, even in very bright illumination. He also noted that the level of vacuum could be quantitatively determined by shaking the bulb to set the vanes in motion and then noting the rate at which the spinning ceases..

Ready-made radiometers are available from science supply houses. Also they are increasingly popular as window ornaments and can often be obtained for about $10 from local craft shops. I've also seen radiometers in the windows of "New Age" boutiques, leading me to wonder what strange powers people might be attributing to them.

The disadvantage of the ready-made radiometers is that they are sealed off. Fortunately, the glass pump-out tube is readily accessible. A suggested conversion is shown in Figure 2. With a file, nick the end of the pump-out and break the tip off. (I'd suggest placing a piece of rubber or vinyl tubing over the glass to prevent cuts). Using epoxy cement, seal a length of 5/16" OD brass tubing (K&S Engineering) to the bulb. Be careful not to get epoxy into the original evacuation ports in the stem. The hanging style of bulb is shown in the figure. These are available from Edmund Scientific as Catalog #G38,510 at $10.95 (1994). - *Editor*

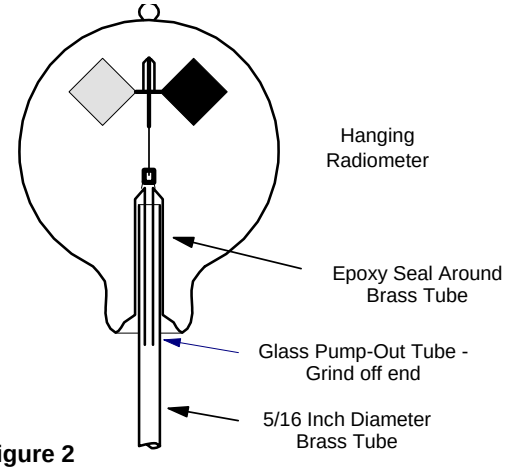


Figure 2

on gas pressure. The optimum condition is found at about 60 microns Hg.

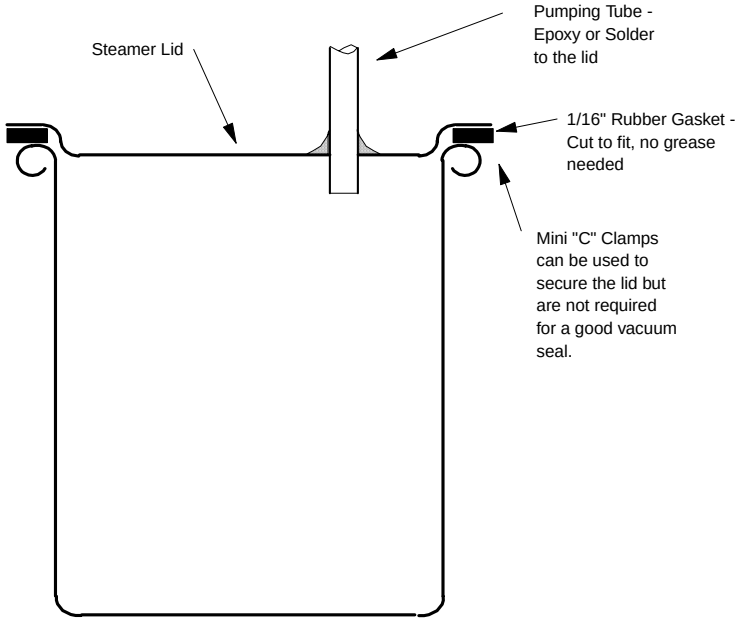
A more delicate suspension may be made by suspending the needle point from one pole of a magnet. A thick walled glass vessel absorbs heat energy readily and it may not be possible to operate the radiometer with an incandescent lamp light.

A Cheap Metal Vacuum Chamber

Contributed by Prof. Robert Jones, Emporia State Univ., Emporia, KS

This chamber is made from a 5 quart stainless steel vertical steamer that may be obtained from Progressive International Corp., Seattle, WA 98109 as catalog number SSAC-50. The price (perhaps not current) was $18.99. The chamber is 6 inches in diameter by 9 inches tall. With a forepump, the chamber is easily evacuated to a couple tens of microns Hg.

The chamber may be easily modified by the addition of various improvised feedthroughs. Clear windows may also be affixed, preferably to the flat bottom surface. Drilling should be done with a sharp drill.



A Simple Vacuum Stand

Steve Hansen

Here is a little table-top system that can be used with the manifold and other accessories described in an earlier article. The manifold was assembled on top of a small laminated hardwood breadboard which also holds the thermocouple gauge indicator. With the exception of the KF (or QF) flange, all of the plumbing is right from the hardware store. All solder joints are made with tin-2% silver soft solder and the threaded connections that are not intended to be taken apart are sealed with household epoxy. A 1/4-20 brass bolt is soldered to the tubing cap at the lower end of the system. This bolt is used to mount the manifold to the wooden base. A 5/8 inch hose barb serves as the connection to the mechanical pump via a short length of reinforced PVC tubing.

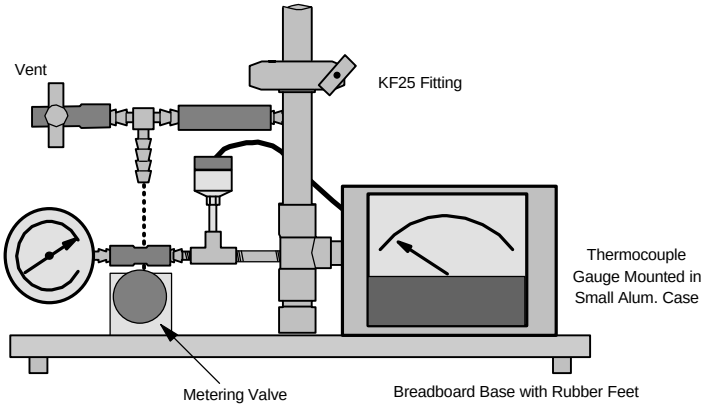
There are two side-arms that are fabricated from 1/8 inch threaded brass fittings. The first connects to a pair of gauges: a Bourdon vacuum gauge of the type used for automotive engine testing, and a thermocouple gauge tube. The second arm is connected to a simple needle valve that is used as an air leak. This serves as a pressure control. I got this valve from American Science and Surplus for about $5.00. It is all stainless with a Teflon packing. Also connected to this arm is the system vent, a short length of rubber tubing with a pinchcock.

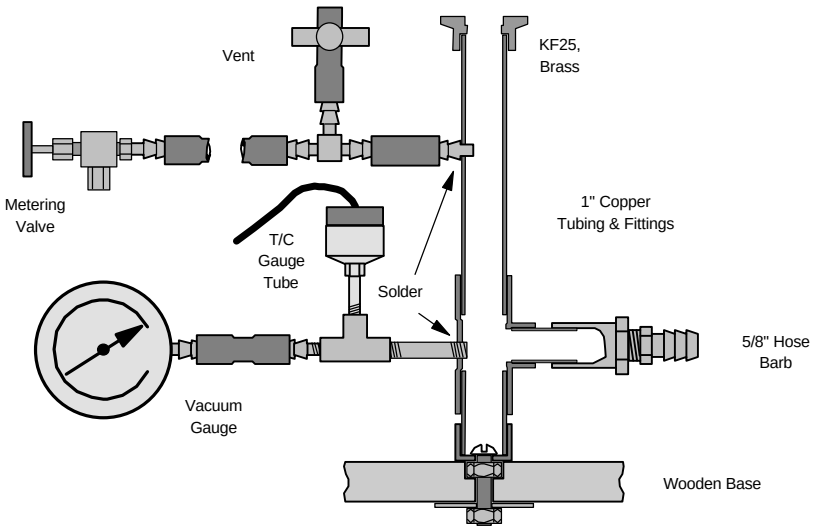
Having the two gauges is, of course, necessary if experiments are to be conducted across the entire rough and medium vacuum range. Additionally, if the system is to be used for classroom demonstrations, the dual gauges help to illustrate that there's still quite a bit to vacuum below where the Bourdon gauge bottoms out.

The pump used with this could be a 2-stage refrigeration service pump as previously described. Suitable pumps include models from Robinair (4 cfm) and J/B Industries (3 cfm).

Having some sort of pressure control on a system such as this is important for showing the different effects that occur at various pressures. With the metering valve and with some adjusting of the pump's isolation valve, it is a simple matter to adjust and maintain the pressure with coarse precision at any value between 30 milliTorr and atmosphere.

This article was originally presented in Volume 4, Number 4.





A Homebuilt Transmission Electron Microscope

A status report on Chris Frye's work-in-progress

Chris Frye of Silver Spring, MD began working on his TEM over 20 years ago. Apparently this journal has helped to revive Chris' interest and work has recently been progressing in earnest.
The microscope is based on the RCA EMU series of instrument. In trying to replicate some of the specifications and features of the commercial instruments, Chris is somewhat fearful of needless overdesign and complexity in the machine - features which an amateur machine need not possess. The final instrument may include some further simplifications.
This article represents Chris' current status and outlook. Suggestions and guidance are sought from anyone with familiarity with this type of instrument or its subsystems. - Ed.

For much of this discussion, the reader should refer to the two figures. Figure 1 shows a general view of the microscope. Figure 2 shows in more detail the construction of the column.

Starting from the top there is a simple electron gun consisting of a neoprene stopper and a couple of 6 mm ID glass tubes holding 5 mm steel electrodes. A thoriated tungsten filament is connected to the electrodes by screws. This structure is encased in a brass cylinder which holds the brass cathode. I am looking at some more elegant (but still cheap) alternatives to this design. The outside wall of the electron gun column is made from a Pyrex cylinder (maybe a hot drink glass if I can find one), epoxy cemented to the brass plate holding the neoprene stopper at the top and to the brass anode at the bottom.

During operation, 5 to 6 volts will be applied across the filament and 50 kV to the cathode cylinder. (I guess that will be a good part not to brush against). After the electrons pass through the anode (which is maintained at ground potential) they pass through a copper bellows and into the condenser lens aperture holder. The copper bellows is used so I can align and tilt the electron gun with respect to the condenser lens aperture. (Does anyone know where I can get brass bellows?) The apertures in all of the lenses are necessary to keep beam divergence angles down to about 10^{-2} to 10^{-3} radians. This has the effect of increasing image contrast, depth of field, and reducing lens aberrations. Now the electrons pass through the condenser lens coil where the magnetic field of the coil focuses them to a spot at the specimen holder. The brass specimen holder is seated in a mechanical stage which I have milled from 1/2" aluminum stock.

The diversity of metals is due to what I had on hand at the time. When I required a non-ferrous metal I used brass or aluminum interchangeably. Of course, the main column, lens coil enclosures and pole pieces are made from soft 99.92% pure iron. The mechanical stage movement in the X and Y directions is accomplished by advancing a 2 mm metal rod through an O-ring via a gear and screw arrangement. After the electrons pass through the specimen (which must be less than 1000 Å thick), they enter the pole pieces of the objective lens coil. They pass through the pole piece aperture and are focused by the coil current to a vicinity somewhere above the permanent magnet intermediate lens. (This lens is just below the brass bellows underneath the objective lens coil.) The permanent magnet is an intermediate lens which focuses the electrons to the vicinity of the projector lens (the lowest in the diagram) where they then pass through the projector lens pole pieces and then are brought to a final focus on the tiltable phosphorescent screen. The final image is viewed through the glass viewing port which is located just above the screen.

At this time I have constructed or obtained many of the items in the diagrams. The following discussion is mainly concerned with the missing pieces.

To make the lens windings I have to find a source of enamel coated AWG 24 magnet wire. As noted above, I need a source of brass or copper bellows as well as of thoriated tungsten filament and a new piece of lead impregnated glass for the viewing port. (The lead glass is to shield the operator from x-rays, which I assume will be produced when 50 keV electrons strike the metal backed phosphor screen. I will have to find out how many mR/hr are acceptable. At the moment I know very little about radiation dosage/retention health factors.)

With all of the constrictions and 30 or so O-rings, my gut feeling on the size of the vacuum system is the bigger the better. At first I thought a 16.7 cfm fore pump sounded right but various salesmen convinced me that pumps of that size are only necessary if I am admitting gas to the system. Supposedly, for the volume and for backing a 3 to 4 inch diffusion pump, I should only need a 1 or 2 cfm mechanical pump. In the end, I figured that something of at least 5 to 6 cfm should be acceptable.

After a bit of searching, I have just acquired a used, complete vacuum system from E. McGrath Co. It is comprised of a Varian EVAC200 7 cfm direct drive mechanical pump, an NRC H-4-SP four inch diffusion pump, a handwheel actuated manifold valve, an NRC cold trap, and a Hastings VT-6 dual T/C and cold cathode gauges with controller. All of this is mounted on a heavy Unistrut frame. At 250 lbs., it was a real chore to move it to my upstairs converted bedroom lab.

To get the pump into full operation I needed a bunch of KF25 fittings and some vacuum hose. These came from Duniway Stockroom. The cooling lines are now connected to the upstairs bathroom sink. (*My wife would never let me get away with this sort of stuff!* - Ed.)

After experimenting with various ways to use the system, I found a procedure to follow which I thought was correct. However, when I researched this subject in O'Hanlon's "A User's Guide to Vacuum Technology" I found out that my procedure was causing a great deal of backstreaming of mechanical pump oil into the chamber and the diffusion pump. Basically I was bringing both the chamber and backing line down as far as I could before turning on the diffusion pump water & heater. Then I would close the roughing valve and open the gate valve (after a 15 minute warm-up for the diffusion pump heater). Well according to O'Hanlon you should -- NEVER -- NEVER -- EVER -- pump any chamber below 15 Pa (112 mTorr) or else you will get gross backstreaming of oil into the chamber. (I guess some sort of foreline trap would solve this problem, at least until it clogs up, but I don't have one).

The last phase of this project involves the high voltage (50 kV at 300 microamps) and lens (120 volts, adjustable constant current to about 2.5 milliamps) power supplies. I am not too concerned about the 120

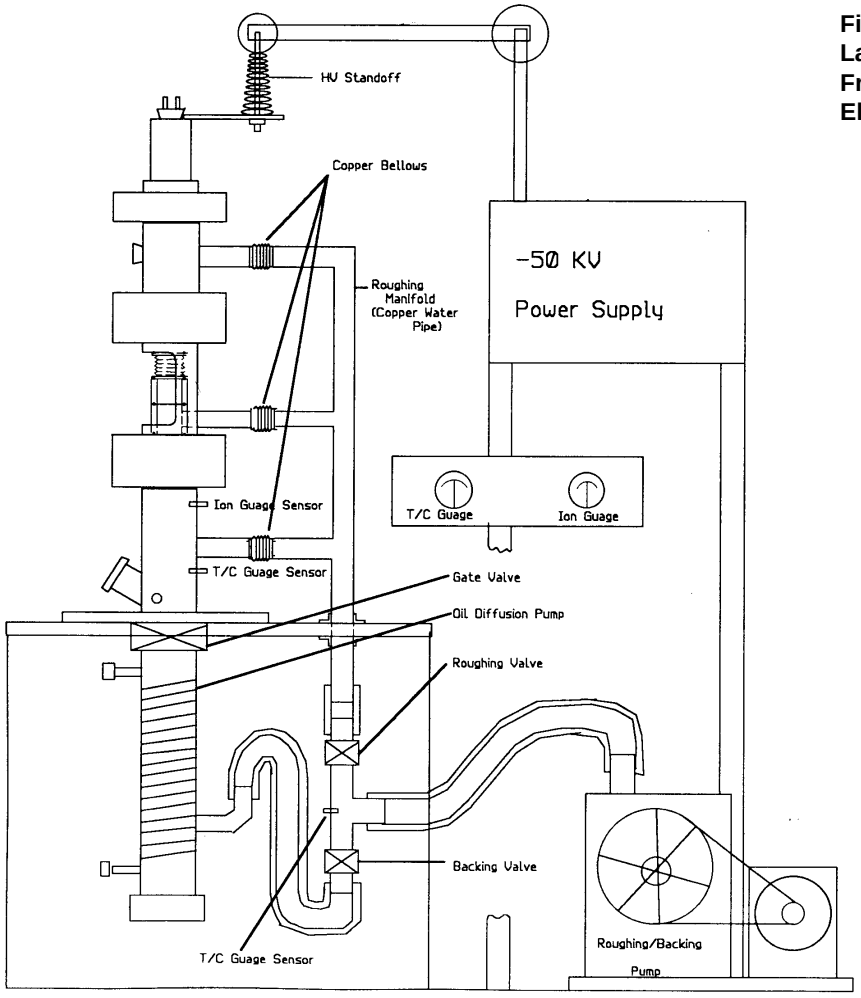


Figure 1 - General Layout of Chris Frye's Transmission Electron Microscope

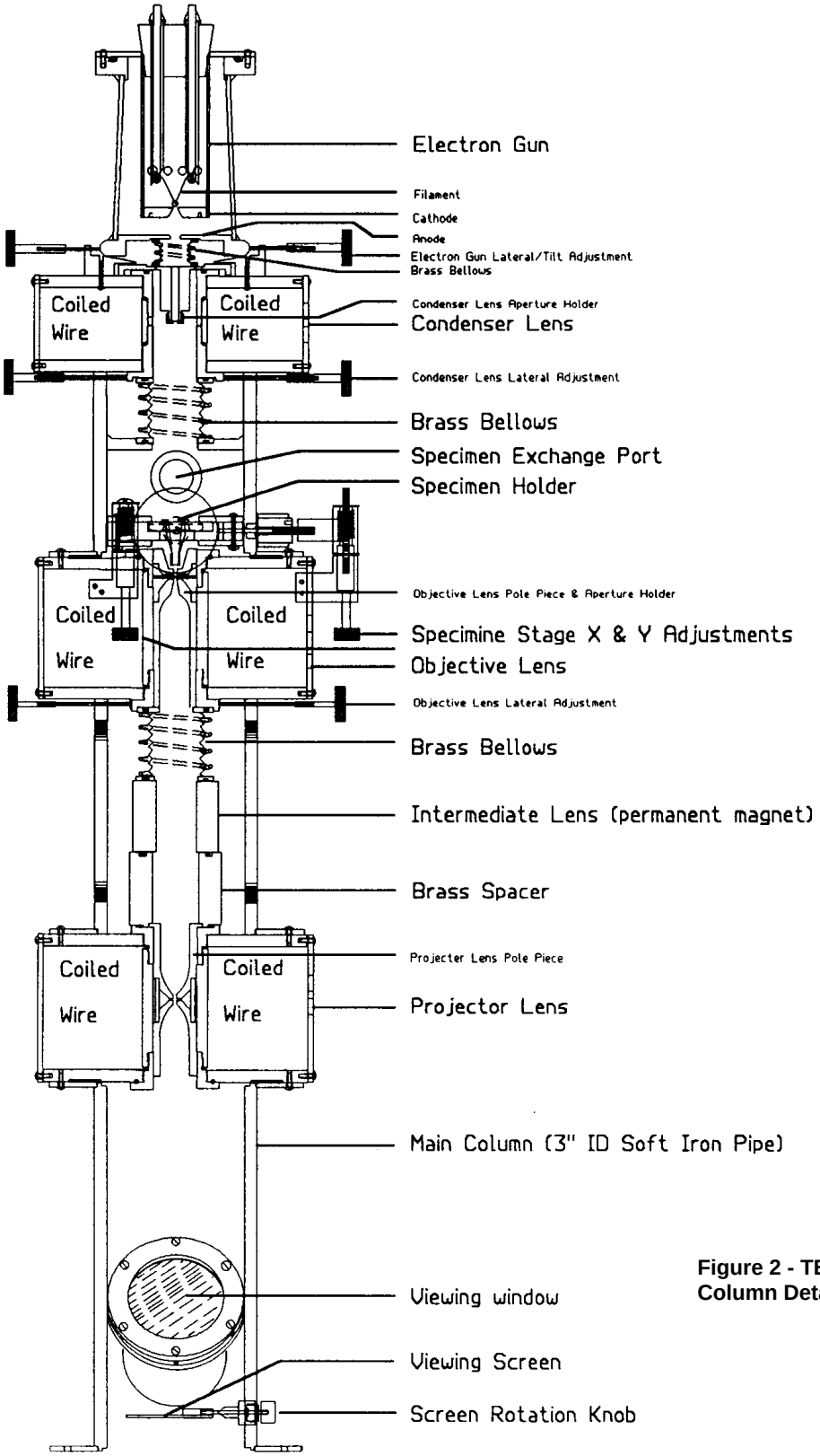


Figure 2 - TEM Column Detail

volt supplies as constant current supplies to the specifications I require are not too mysterious.

The high voltage supply is another issue. While the absolute voltage is not that critical, any drift from the nominal value is extremely important to prevent aberrations of the image. A drift rate not exceeding 1 volt per minute is my design goal.

I have obtained the basic supply, a modular unit which came from OE Technologies. Now I need to add some sort of regulator. Just placing a big capacitor across the output might work but I would like to pursue some sort of active regulation. When asking various people whom I thought would have some knowledge of high voltage regulation the responses I usually got were like "Are you kidding" or "No such stability exists." However, in looking at John Strong's "Procedures in Experimental Physics" (reprinted by Lindsay Publications), a Geiger counter regulator circuit shown on page 296 claims a drift of less than 0.1 volts per hour with an output of 1500 volts. Obviously, this would have to be scaled a bit and a modern higher voltage replacement for the obsolete type 57 tubes would have to be found. Perhaps the type of high voltage beam regulator tube used in TV sets would work.

Originally published in Volume 3, Number 2. Below is an update that was printed in Volume 4, Number 3.

AN UPDATE FROM CHRIS FRYE ON HIS TRANSMISSION ELECTRON MICROSCOPE

"It's been a while since I provided an update on my progress in making my TEM. Sorry for the delay but the progress has been slow and methodical. As you know, making a precision part on a miniature lathe can take many man-hours and I had to make several dozen. I really appreciate the suggestion about buying the lathe (a Sherline) as well as the recommendation that I design the TEM in a modular fashion. Without these suggestions I would have been in trouble. As it is, everything is coming along fine.

"As you can see by the photo, I have completed the column and the manifold. After completing all the components of the column, which included winding many miles of hair-fine wire on the four lenses, I attached the viewing chamber to my vacuum system base plate. I tapped four holes into the baseplate (#12-32). The viewing chamber has a flange at the bottom with a 5 inch O-ring recessed into a groove that I cut with the Sherline. The phosphor screen is set at a 45 degree angle to the viewing window. After attaching the viewing chamber I started the long process of vacuum testing the chamber.

"In testing the column components for vacuum integrity I started from the bottom with the viewing chamber and proceeded up the column as I added each section until I had the electron gun in place (seen at the top). As I worked my way up, I blanked off the top of each component with a rubber stopper or O-ring and metal plate as appropriate.

"Recalling the diagram that I submitted in my last article, you will remember that the column consisted of quite a number of vacuum seals and feedthroughs. You can imagine the nightmare I had in making all of these vacuum-tight. The two things that saved me a great deal of time were *Duxseal*, a poor man's leak detector that is a clay-like putty for temporarily sealing suspected leaks, and *Epoxy Patch*, and equivalent to Varian's *Torr Seal*. This is used to seal the leaks found by the *Duxseal*. (Both of these products are available from Duniway Stockroom Corp.)

"The mechanical pump is an EVAC-200, a 200 liter/minute (7 cfm) pump. For vacuum testing the column I didn't need to use the oil diffusion pump because I knew that my mechanical pump would pull a 20 liter (12" x 12") down to 10 microns of Hg in about 1 minute. I figured that if I could get about the same performance on the column I would be okay. It's lucky that I have to use the diffusion pump for testing as this would have caused a great inconvenience in warm-up and cool-down time. As it is, the testing took me over 5 months working 6 hours a week, or 130 working hours, to leak test and fix the system. One reason for this long period is that it takes 72 hours to cure the *Epoxy Patch*. In total, I found and corrected 72 leaks. Most of the leaks were the result of poor weld, solder or braze joints. Obviously I need to acquire some better skills in this area.

"The result of this testing is that I now have a column capable of holding a vacuum of 30 microns Hg after a 5 minute pumpdown with the mechanical pump. This is not quite the 10 microns I was striving for but I think it will be sufficient to bring the column down to $4x10^{-5}$ Torr after I've kicked in the 300 liter/sec. diffusion pump. I will test this theory later.

"During the course of these procedures I became curious about the effective pumping speed of my mechanical pump. Referring to "A User's Guide to Vacuum Technology" by John F. O'Hanlon, I found the following equation:

$$\frac{\text{Leak Rate (Torr-liters/sec)}}{\text{Pumping Speed (liters/sec)}} = \text{Ultimate Pressure (Torr)}$$

The volume of my column plus all the pump plumbing is 6 liters. After achieving a pressure of $3x10^{-2}$ Torr, I

turned off the pump and the pressure rose to $4x10^{-2}$ Torr in 8 seconds. From this I concluded that I had a leak rate of:

$[(4x10^{-2} - 3x10^{-2}) / 8] \times 6$ or $7.5x10^{-3}$ Torr-liters/sec.

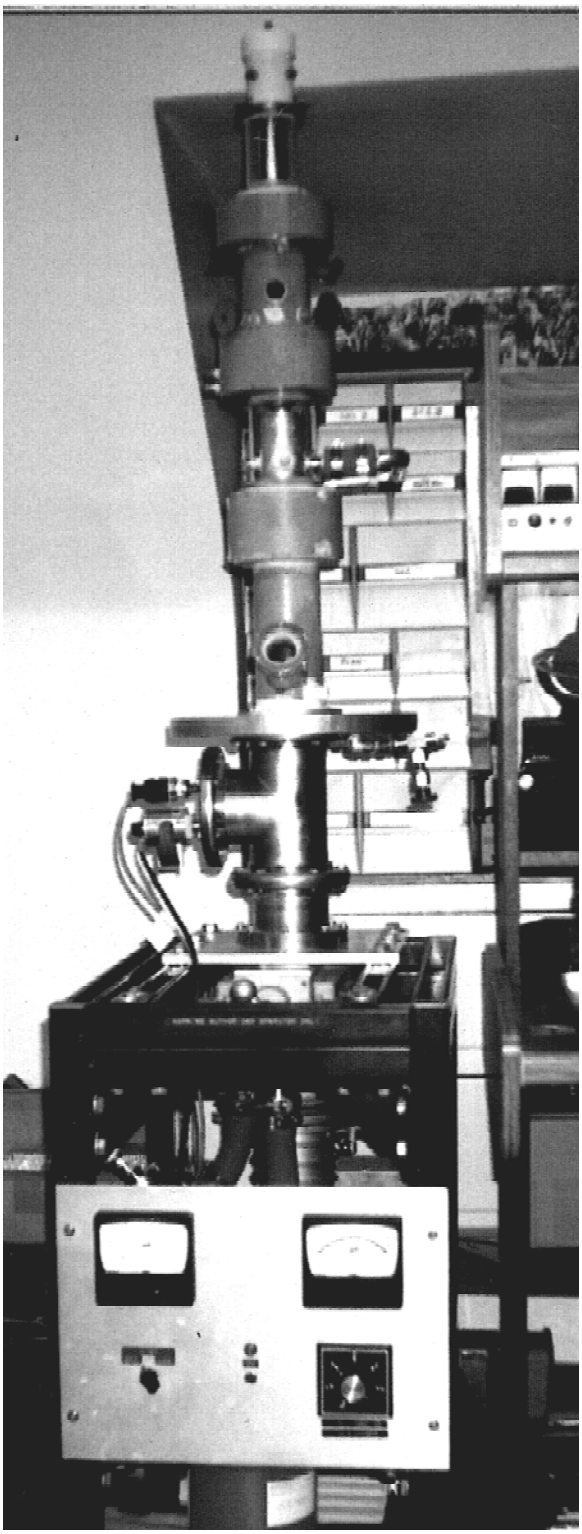
"The ultimate pressure I can obtain is 30 microns Hg. Substituting into the above equation we get:

$7.5x10^{-3}$ / Pumping Speed = $30x10^{-3}$.

Transposing and solving gives a pumping speed of 0.25 liters/sec. Since the pump is rated at 200 liters/min or 3.33 liters/sec, I am only getting an effective speed of 7.5% of the rated value. A little disappointing but interesting."

Anyone with any advice concerning this project is invited to contact me at 2116 Linden Lane, Silver Spring, MD 20910-1705.....Chris Frye.

Readers who are interested in TEMs should also check out the two simple microscopes described in Scientific American's Amateur Scientist *column, September, 1973.*



The Production of Phosphors: An Introduction

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I. INTRODUCTION

The production of phosphors suitable for use on x-ray screens or for visualizing electron beams is easily within the realm of the amateur scientist. In industry and in laboratory phosphor research, great pains are taken to insure that the highest purity, cleanest environment, and most pristine manufacturing equipment are employed in the production of phosphors. Often, special atmospheres and very high temperatures are required for creating the phosphors . Sometimes electrical and magnetic fields are needed. Also, state-of-the-art analytical instruments are used to monitor chemical composition and crystalline state of the phosphors at various stages during production.

Fortunately for our purposes in this article, we are not concerned with most of the restrictions listed, nor do we need to monitor the process. However, for repeatability, as well as to obtain a phosphor with satisfactory properties, we will need to use AR (analytical reagent grade) purity chemicals wherever possible. The area used for phosphor production should be well-ventilated and clean. ***Also, familiarity with the proper handling and storage of potentially toxic chemicals should be a basic requirement before launching your career with phosphors.***

In addition to the chemicals, a high-temperature furnace, some quartz crucibles and covers, distilled water, access to a laboratory balance, various pieces of lab glassware and a mortar & pestle are required.

The high-temperature oven I use is an L&L kiln. This can maintain a maximum temperature of 1250° C. A Honeywell UDC 2000 controller is used to maintain the chosen temperature to within 3° C.

Ideally, an oven which can reach 1600° C and which can operate under various atmospheres is desirable. However, those ovens are very pricey! I limit myself to phosphors which can be produced below 1250° C. I do use a tube furnace along with quartz tubes when I must have a controlled atmosphere (e.g., flowing hydrogen, argon, nitrogen, etc.), but here again I have a limiting temperature maximum: below 1100° C in this case.

II. PHOSPHOR SELECTION AND NOMENCLATURE

There are as many different phosphors as there are applications which require them. The phosphor to use is dependent on the following requirements:

1. The source of excitation: alpha particles, electrons, x-rays, heat, ultraviolet light, high-frequency electrical power, etc.
2. The preferred wavelength of light emission: what "color" should the phosphor emit when excited?
3. Stability under the conditions of the application: Can it withstand the imposed environmental conditions such as humidity, atmospheric oxygen, light exposure and so forth during handling? And, can it withstand the source of excitation? An electron beam at 50 kV has quite a bit of energy and it can "burn" the phosphor if it bombards the same area for a prolonged period of time. This gets worse at higher beam currents/ (Hence, a booming market for computer screen savers!)

One of the easiest phosphors to prepare is Cub. ZnS:[Zn]. The nomenclature for this phosphor is translated as follows: Cubic zinc sulfide with zinc atoms as the presumed activator. In "phosphorese," an activator is an atom or ion which is added to a compound (usually in trace quantities) to promote the emission of light. The activator ions find their way to surface sites on the host phosphor crystals, inside the host crystals in interstitial sites, as well as inside the host crystal in substitutional sites whereby the activator atom replaces the atom of the host crystal. Thus, solid solutions of the activator within the host crystalline compound can be formed. The colon is followed by the chemical abbreviation of the activator and indicates that the activator is present in variable (nonstoichiometric) proportions.

In our first phosphor example, brackets ([Zn]) surround the free atom indicating that the presence of the atom or ion is conjectured and not actually proven. Generally, we add a small quantity of a compound (e.g., manganous oxide) to our host in which case there is no question about the presence of the activator. In zinc sulfide which is exposed to a high temperature, some of

the compound breaks down releasing sulfur (which burns or evaporates away), leaving small quantities of zinc in its atomic state. It is this atomic zinc which is believed to self-activate the phosphor in our first case. Enough said, let's produce some phosphors!

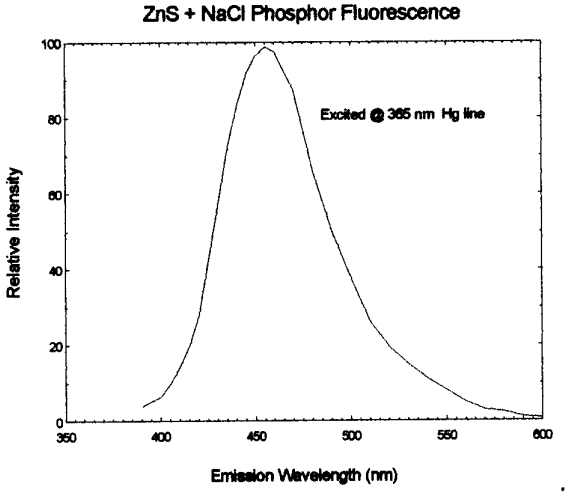
III. PREPARATION OF Cub. ZnS:[Zn]

In a small mortar, mix together 5 grams of 99.99% pure zinc sulfide with 0.1 grams sodium chloride (AR grade). Thoroughly mix the compounds, grinding as necessary to form a fine powder. **(CAUTION: zinc sulfide is toxic, as is the hydrogen sulfide given off when the compound is exposed to high humidity or when heated.)** Place the mixture into a quartz crucible (35ml capacity) and cover with a quartz crucible cover. Place the crucible in the high-temperature oven and set the temperature to 940° C. Typically, the oven may take one hour to reach the working temperature. Heat the mixture for an additional one full hour with the oven at 940° C. Turn off the oven and allow the crucible and its contents to cool slowly to room temperature.

It should be noted that the sodium chloride serves as a flux which aids in melting the zinc sulfide. It does not otherwise enter into this particular phosphor reaction unless there are contaminants within the sodium chloride. In general, fluxes serve to provide a fluid phase to aid in transporting and dissolving the ingredients used, as well as increasing surface reactivity and promoting good crystal growth which is very important for successful phosphors. However, there are fluxes used which are an integral part of the chemical reaction as they add oxygen to the phosphor. In fact, without those particular fluxes (which some consider activators when so used), the phosphors may not be successful.

At this point, the purist will wash the powder with distilled water saturated with hydrogen sulfide gas **(DEADLY GAS!!!)**. This washing does a good job of removing contaminants and it produces a homogeneous phosphor. However, for our purposes in this initial experiment, we will bypass the washing and just examine the powder under short and long wave ultraviolet light. In doing this, you may notice a fluorescent yellow layer of phosphor lying atop a fluorescent white layer with some areas of blue fluorescence. The fluorescent homogeneity of your phosphor will depend strongly on how well you blended the ingredients. The yellow layer on top of the white layer most probably represents some oxidation which occurred at the interface between the air and the phosphor. It is the white material which we want. I examined the phosphor (excited with long wave UV at 365 nm) using a Perkin-Elmer model 203 fluorescence spectrophotometer and recorded an emission peak at 455 nanometers. The spectrum is shown in the figure below.

In his book "An Introduction to Luminescence of Solids," H.W. Leverenz recorded a peak at 470



nanometers for this phosphor. The difference can easily be attributed to the presence of extremely small quantities of contaminants from the chemicals used, in the containers employed, or "something in the air," or all of these. And that is the way of things in the world of phosphors.

IV. PREPARATION OF Cub. ZnS:Mn

If we modify the above reaction a little, we will come up with another phosphor, Cub. ZnS:Mn, which has a peak emission at 572 nanometers (orange-red).

In the mortar, combine 7 grams zinc sulfide, 0.14 grams sodium chloride and 0.315 grams manganous oxide (AR grade MnO). Grind and mix well. Place the mixture in a quartz crucible and cover it. Place this in the oven and heat to 1000° C. Keep it at this temperature for 90 minutes. Cool as before and examine under short and long wave UV.

The above phosphors are nice, but due to the sulfide content they not too stable if the phosphor is to be repeatedly exposed to the air (and humidity). My most recent requirement was for a stable phosphor which I would use to coat the inside of a Crookes bulb (obtained from *the* Bell Jar). I wanted a bright yellow-green emission under electron bombardment. In short, I wanted a zinc silicate phosphor.

V. PREPARATION OF A ZINC SILICATE PHOSPHOR

Start by mixing 16.28 grams zinc oxide, 6.08 grams silicon dioxide and 0.085 grams manganous oxide (MnO) in a mortar. I find that adding small amounts of pure alcohol to form a slurry or paste helps produce a homogeneous mixture when you are using milligram quantities of activators. Evaporate the alcohol thoroughly before placing the mixture in a covered quartz crucible. (Leverenz recommends a platinum crucible, but if I could afford that I could afford an oven that reaches 1600° C!) Place the crucible in the oven and heat at 1250° C for one hour. (That's one full hour at that temperature. Don't take into account the hour or so it takes to get there.) Cool and examine under UV light. You have produced Rbhdl.Zn_2SiO_4:Mn (artificial willemite) with a peak emission at 525 nm. Also, examine the quartz crucible under short-wave uv. Notice that the walls of the crucible fluoresce just like the powdered phosphor. Generally, you can only get one or two uses out of a quartz crucible, especially when heated to 1250 degrees. If you do reuse the crucible, only use it to produce additional quantities of the same phosphor.

I have not gone into the details for coating inert surfaces (i.e., glass) with powdered phosphors. I am still trying to master the technique and will pass on suggestions in a future article. Furthermore, there are many other phosphors which can be produced by the amateur: those that employ rare earth compounds, tungstates, organic chemicals, etc., etc. The field is quite interesting...*illuminating* one might say. But that's another subject.

Originally published in Volume 5, Number 2.

A Brief Note on Solder Glass for Sealing

Daniel Koller

Daniel Koller of East Brunswick, NJ has a few tidbits based on his practical experiences while a grad student at SUNY Stony Brook.

First of all I have a whole bunch of notes on solder glass techniques, mainly references to articles in the American Journal of Physics, for building tubes and other things for student demos. I looked into this stuff as one possible experimental approach to my thesis. Solder glass is supplied in the form of a paste consisting of the glass 'frit' and the volatile vehicle. It is "painted" on the components to be assembled and is then fired in a small oven. A common use of solder glass is to seal the leads in ceramic integrated circuit packages. The articles that I found useful were:

New High Vacuum Technique: Kits to be used by students to make vacuum tubes (J.H.O. Harries, American Journal of Physics, Vol. 28, pg. 698 (1960). One of the first, if not the first, article on this topic. Has charts of vacuum *vs.* time after firing getters in homemade tubes.

Sealing with Solder Glass (R.H. Dalton, American Journal of Physics, Vol. 32, pg. 479, 1964). This article provides general information and considerations.

Experiences with Solder Glass and Students (J.G. King, American Journal of Physics, Vol. 32, pg. 473, 1964). This has some examples of things built by students and their operating characteristics.

Homemade Frank-Hertz Tube (Tyler Rosenfeld, American Journal of Physics, Vol. 33, pg. 849, 1965). Covers fabrication making use of a cut-up commercial tube.

Homemade High Vacuum Techniques (Multiple authors, American Journal of Physics, Vol. 32, pg. 483, 1964). This article describes a homemade Penning gauge. There are some illustrations of the sealing ovens.

Corning Glass still sells solder glass and I have a whole bunch of samples from them. Easy to get if you are trying something out. Otherwise, you can get the materials safety data sheets, and use them to make your own formulations and frits.

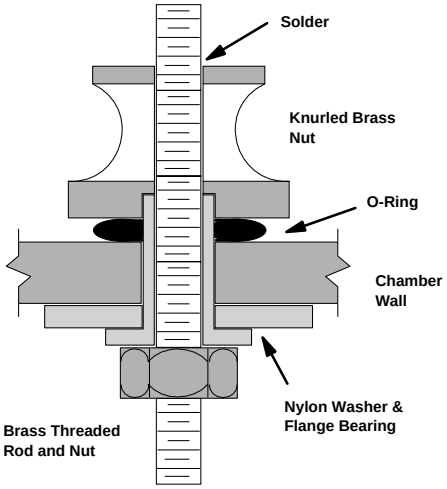
Originally published in Volume 4, Number 3.

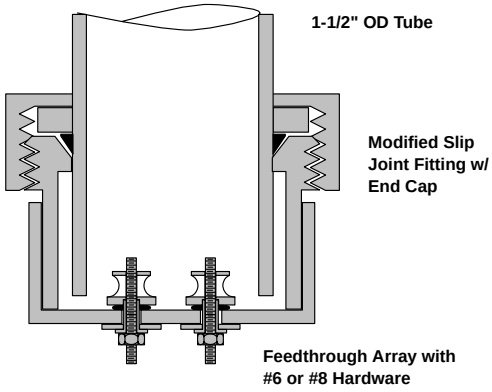
Some Ideas for Electrical Feedthroughs

Steve Hansen

Here are a couple of approaches for electrical feedthroughs.

The first design is for lower voltages. The key components are a knurled brass nut, a length of threaded brass rod (which can be obtained by cutting





Above: Low Voltage Feedthrough
Right: Array of Feedthroughs in 1-1/2" Copper End Cap

the head off of a brass machine screw), a brass hex nut, a nylon flange bearing, a nylon washer and an O-ring.

The left hand figure above shows the general arrangement. The nylon components are manufactured by Jandorf and are available at most hardware stores. These items are typically used in toys, lawn furniture, and the like. Jandorf makes a number of flange bearings ranging in size from #6 through ¼". Available lengths go from about ¼" to ¾" in the larger diameters. The washers are listed under *thick nylon washers* and are manufactured in a wide variety of hole sizes and outside dimensions.

As the outsides of the flange bearings are not standard screw sizes, it may be necessary to buy a slightly undersized washer and drill it out to clear the flange bearing.

Assembly is straightforward. Precise dimensions will be dependent upon such factors as rod size and chamber wall thickness. First enlarge the threaded hole at the wider end of the knurled nut to the diameter of the flange bearing. Then screw the threaded rod into the knurled nut allowing it to protrude an adequate distance at the smaller end for the attachment of leads, etc. inside the vacuum chamber. Solder the nut to the rod at the small end of the nut.

The length of the flange bearing should be such that the sleeve passes through the O-ring and into the recess in the nut but not so long as to prevent the O-ring from squashing out.

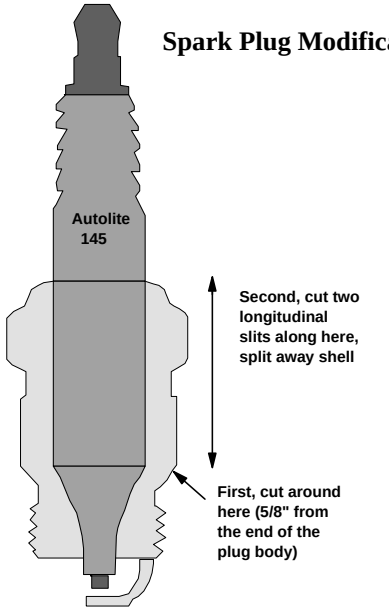
A complete set of parts for this feedthough should cost on the order of $2.00 to $5.00 depending mainly upon the type of O-ring used.

The right hand figure above shows how the smaller sizes of hardware may be used to provide multiple feedthroughs into a small glass chamber. The example shows a small array of feedthroughs mounted on a modified copper slip joint fitting with a 1½ inch end cap. This would be compatible with the VacuKits.

The figure on the next page shows how a common spark plug can be modified for use as a high voltage feedthrough. Rather than trying to fashion a screw mounting for an as-is spark plug, this approach uses only the ceramic insulator (with center conductor, of course). A vacuum seal is effected by mounting the insulator in an O-ring sealed compression fitting.

The particular plug that I took apart was an Autolite #145. The insulator has a ½" diameter straight section nearly ¾" long. This is compatible with either standard vacuum compression fittings or adapted half inch brass compression plumbing fittings.

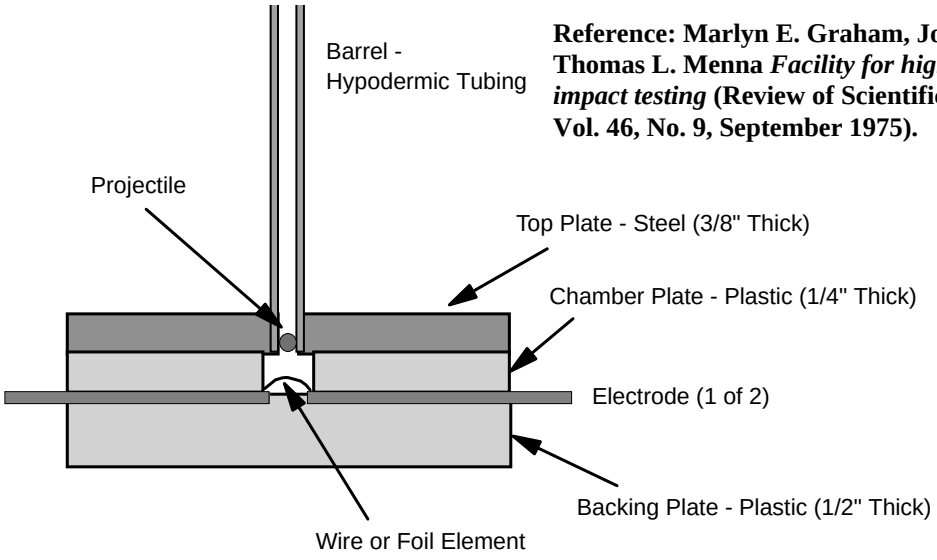
The only trick is getting the insulator away from the metal shell. This may be easily done in a couple of steps. First, measure about 5/8" from the end of the plug (the end of the threaded portion) and make a cut all the



Spark Plug Modification

way around the plug. This will separate the lower part of the shell from the insulator. Then make a couple of longitudinal cuts diametrically opposite each other through the remaining part of the shell. With some gentle prying, the metal should separate cleanly. The plug is now ready to install in a compression fitting.

Tidbit - Gun for High Speed Particle Impact Testing



Reference: Marlyn E. Graham, John D. Carlisle, Thomas L. Menna *Facility for high-speed particle impact testing* (Review of Scientific Instruments, Vol. 46, No. 9, September 1975).

This type of device has been used to accelerate small projectiles (4 to over 40 mils in diameter) to speeds in the range of 2000 to 6000 meters/sec (i.e. from about 4,500 to 13,500 mph). Particles driven to such speeds can be used to simulate micrometeorite impacts on spacecraft. The device consists of a sandwich of 3 plates, securely bolted together; a plastic backing plate into which is imbedded a pair of thick copper or brass electrodes; another plastic plate which has a chamber in which a wire or foil element is exploded; a metal plate to which is mounted a barrel. The discharge of a capacitor bank (a few kilojoules at several kV) explodes the wire or foil, driving the projectile out of the barrel. The barrel should be coupled to a vacuum chamber which would also contain the target. Suitable hypodermic tubes and projectiles (e.g. 1/64" dia. chrome steel balls) may be obtained from Small Parts, Inc. of Miami Lakes, Florida. Needless to say, the device needs to be securely bolted together, a blast shield must be placed between the device and the operator, and strong precautions must be take to keep the operator from becoming part of the electrical circuit. Since the gun is a muzzle loader, it should be exempt from BATF laws. In any event, I doubt if it would be useful in a hold-up or for self defense.

The Evolution of the X-Ray Tube

Dan Smith
8904 Cypress, Cotati, CA 94931

The First

Roentgen's first experiments were with an ordinary Crookes tube with a bit harder vacuum than was customary - on the order of one-millionth of an atmosphere or about 1 micron Hg. The vacuum was produced by a mercury displacement pump, most likely a Sprengel pump. It was necessary to have the right amount of residual gas: too much would result in a normal glow discharge and the tube would be too conductive. Too little and an excessively large induction coil would be required for the tube to conduct [1].

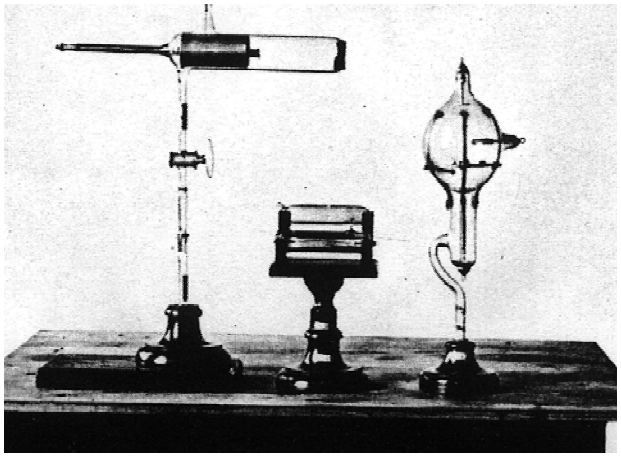


Figure 1 - Roentgen's Apparatus. Lenard tube to the left, Crookes tube at right.

A flat aluminum disk cathode produced the cathode ray stream that would be accelerated by a wide anode. The stream merely impinged on the glass walls of the tube with the x-rays originating from the interaction of the cathode ray stream with the glass wall. The radiographs that resulted were murky but none-the-less magical. The lack of clarity was due to the lack of focus of this crude x-ray source [2]. Heat dissipation was soon found to be a problem and the glass itself was a poor generator of x-rays due to the low atomic number of the elements in the glass (e.g. silicon) [3].

In order produce substantial quantities of x-rays the speed of the electrons needs to be very high. To achieve this, a tube needs to sustain a voltage drop of at least 40 kV. This translates to an induction coil with a spark of about 2 inches. A tube with a 75 kV potential accelerates the electrons to about half of the velocity of light. Roentgen was using a coil that would produce a 5 inch spark. Some of Roentgen's apparatus is shown in Figure 1.

The Focus Tube

Some improvements were necessary and, as early as 1896 the first focus-type tubes (see Figure 2) were introduced. These had a concave aluminum cathode. With the radius of the curve in the cathode approximately twice that of the cathode-anode spacing, the cathode rays would converge to a sharp spot on the anode. The anode was angled at 45 degrees and was of a high melting point, high atomic weight metal such as platinum [1]. The focused beam of cathode rays combined with the high atomic weight of the anode material provided a point-source of "harder" x-rays that would produce a more well-defined image with a shorter exposure. The glass envelope was blown into a round bulb-shape around the anode in order to reduce electrostatic stress across the surface of the tube [3]. This allowed the use of higher potentials.

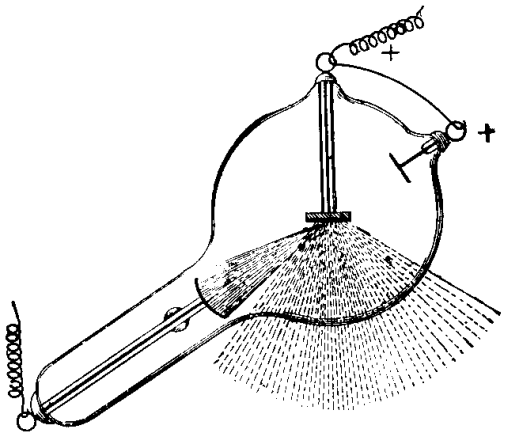


Figure 2 - Focus Tube.

Self-Regulating Tubes

Good as the focus tube was, it was soon discovered that the residual gas within the tube tended to be absorbed into the relatively porous metal of the anode and cathode. Platinum also had the unfortunate characteristic of adsorbing gas when hot. This tended to reduce the vacuum over time with the result that the tube would become "harder" over time. Exposures became guesswork although it was possible for the operator to roughly gauge the "strength" of the tube by observing the intensity of the greenish glow of the glass envelope of the tube or the brightness of a fluoroscope screen. Many operators suffered terrible radiation burns from repeated exposure tests that they performed on their own hands.

A variety of pressure regulation schemes were introduced to address this problem. An early attempt involved the use of a palladium wire that was sealed into the bulb near the cathode. This wire was covered with a glass cap. Working on the principle that palladium is relatively porous to hydrogen when hot, a flame would be allowed to play against the wire which would then allow some hydrogen to pass into the tube.

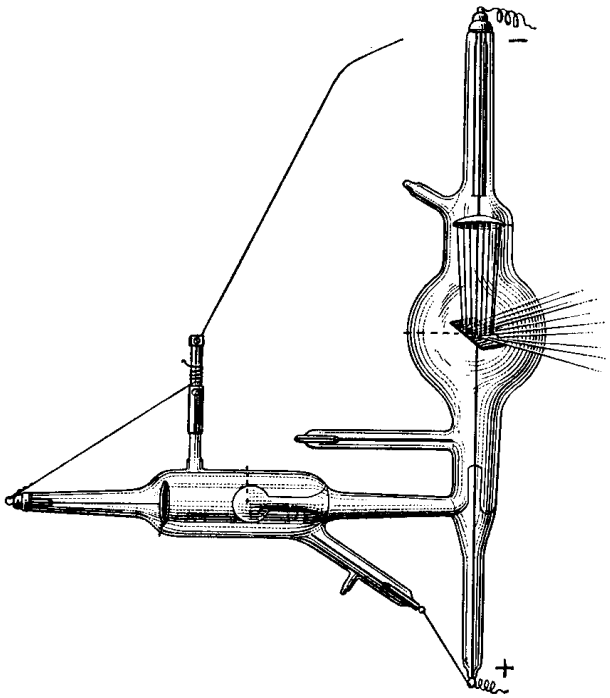


Figure 3 - One Form of Self RegulatingTube. The horizontal appendage contains a bulb with a moisture containing material. When the vacuum is too high for the main tube to conduct, the side appendage is connected by means of the gap at the top and conduction occurs within that appendage. Warming of the bulb releases gas which raises the pressure in the tube and normal operation continues

Next came the self-regulator. This configuration used a side tubulation near the cathode, inside of which was a bit of asbestos impregnated with potassium chlorate, manganese dioxide or other suitable chemical that would release gas when mildly heated. Sealed into the tube was a long wire that dipped into the asbestos. The theory of operation was thus: with the wire end about 1 inch from the cathode, the tube would function normally until the vacuum became too "hard" for the current to flow. The current would then take the path of least resistance and jump to the wire. With the discharge now going through the asbestos filled bulb, the asbestos/chemical mix would liberate some gas. This would lower the degree of vacuum in the tube whereupon the discharge would revert back to passing between the x-ray tube's cathode and anode. Thus, it was no longer necessary to vary exposures according to subjective judgments.

The degree of vacuum could be varied by changing the gap to the wire: a harder tube could be created by increasing the gap, a softer tube by shortening the gap [1]. A tube of this type is shown in Figure 3.

This system worked extremely well, the author once having a tube from 1915 whose regulator still functioned despite the tube's heavy use as judged from the purpled glass.

These early gas tubes produced a beautiful glow when activated. A greenish hemisphere of light would emanate from the glass opposite the anode due to the radiation (UV and x-rays) striking the German-made glass used to make the bulbs. Should a tube be connected backwards, green rings appear. Very few x-rays are produced as the cathode stream is dissipated as opposed to being focused [4].

Self-Rectifying Tubes

Another obstacle was the power source. The old induction coil, although a good dc source and well suited to an era when ac mains weren't widely available, was expensive to manufacture and difficult to insulate at higher voltages [5].

A variety of static generators were also used, including the Wimshurst influence machine. These had the advantage of requiring no electrical power to run. However, they suffered from electrical leakage, a situation made worse in humid weather.

The new Tesla coil had many advantages but the ac output either wouldn't operate some tubes or wasted energy by dissipating power in the tube during the

wrong part of the cycle. Some tubes were water cooled to permit the use of the Tesla coil but with the advent of high-tension line frequency transformers, some sort of rectification became necessary [3].

Elihu Thompson developed a tube with two sets of cathodes and anodes. Although rectification wasn't necessary and the full wave of the unrectified ac was used, the tube produced two distinct images on the radiograph.

The next step was to incorporate an "assistant" anode. This was another tubulation, this time near the anode. Within it was a concave aluminum disk, much like the cathode, and oriented at a 45° angle to the anode. When connected to the anode, the positive half-wave operated the tube normally but, in the negative half-wave, the beam would be defocused and dissipated harmlessly on the cathode. Variations on this included a design with a second platinum target located in-line with the assistant anode and another version with a necked-down area in the glass envelope around the assistant anode to channel the cathode rays [1]. Finally, a corkscrew-shaped anode was also devised, the effect of which was to retard the electron flow during the reverse half-cycle.

Thus, the x-ray tube had become quite an interesting arrangement of tubulations, electrodes and spark gaps. The author has seen one tube with several spark gaps and a cluster of tubulations at the Perham Museum. (This tube is now in storage.)

Beyond these various internal features, a number of external devices became employed. Cold-cathode rectifiers, called valve tubes, came into use as did synchronous rectifiers. Similar to the rotary spark gaps used in wireless apparatus, the poles of the driving motors were aligned with the gaps such that the correct polarities were always delivered to respective electrodes of the x-ray tube [4]. Figure 4 shows a system with a synchronous rectifier.

The Coolidge Tube

When W.D. Coolidge of General Electric's Victor X-Ray Division made the first tungsten wire filament tube in 1913, he circumvented many of the problems that were inherent in the gas-type tube.

In this tube, as shown in Figure 5, a filament was spiraled within a the cathode cup. Heating the filament would cause electrons to "boil" off through a process called thermionic emission (originally called the Edison effect as he was the first to observe the phenomenon in a lamp that was provided with an extra electrode), whereupon the electrons would be accelerated by the potential across the tube. Since electron emission was now controlled by the current applied to the filament as opposed to originating in a low (but not too low) pressure gas discharge, the pressure in the tube could be reduced to a hard vacuum i.e. well below 10^{-5} Torr. This required some improvements in the manufacturing process, e.g. vacuum firing of the components prior to final evacuation and sealing, to minimize any subsequent outgassing of the components. Tungsten, besides its use as the filament, also became the standard material for the anode.

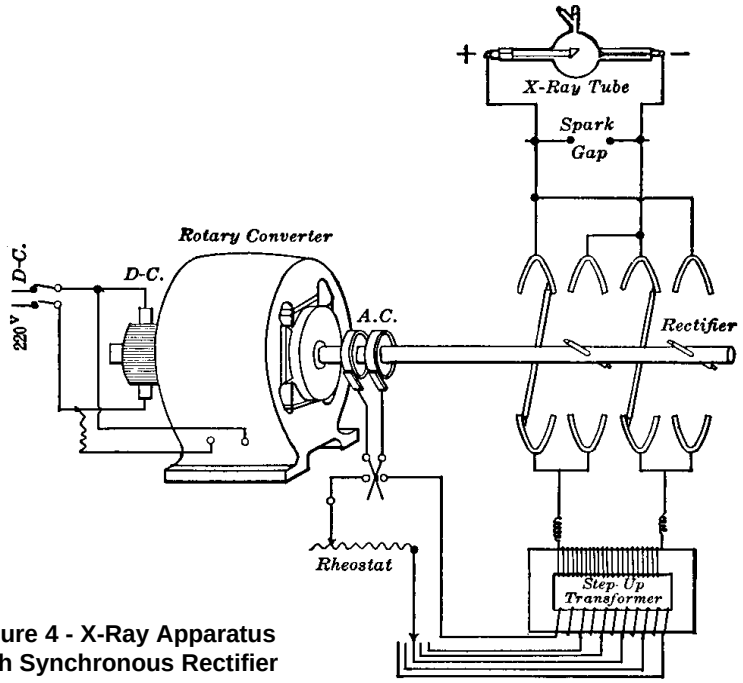


Figure 4 - X-Ray Apparatus with Synchronous Rectifier

By this time the standard power source had become the rectified output of a high tension ac transformer. With the hard vacuum in the tube, the Coolidge tube could sustain a much higher potential across the tube.

There is an obvious similarity between the Coolidge x-ray tube and a thermionic rectifier tube. Both have two elements, one of which is a filament. The difference is that the rectifier is operated in a region where there is a copious emission of electrons which results in a low voltage drop across the tube. With the x-ray tube, the filament is

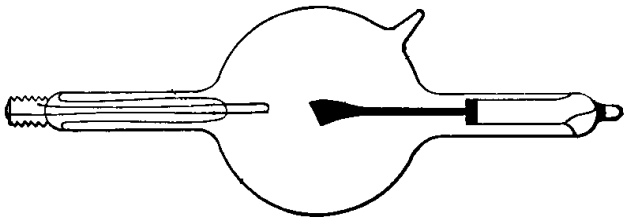


Figure 5 - Coolidge X-Ray Tube with Improvements

operated such that there is a lower emission of electrons with the result that a high potential can be sustained across the tube. Varying the electron emission by changing the heating current to the filament will alter the characteristics of the tube, allowing it to be fine tuned to its best area of operation [2].

With the transformer-type power supply and the Coolidge tube, high speed radiographs were now possible. Some exposures could be made in a fraction of a second [1].

Improvements Beyond Coolidge

The next improvement was the self-rectifying hot cathode tube. Since the Coolidge-type tube has the elements of a rectifier, this combining of two functions into one would seem to be a simple way to reduce the complexity of the x-ray apparatus.

The danger in feeding ac to a tube is that, if operated long enough for the target area on the tungsten anode to heat to incandescence, it too will begin to emit electrons and a reverse current will be set up in the tube. This will then lead to the destruction of the cathode.

Because of this, tubes designed for self-rectification have a very large and heavy copper rod attached behind the tungsten target disk. Leading out of the tube, this would be either attached to a finned radiator for air cooling or it could be cooled with a fluid such as water or oil. Complete immersion in oil also provided electrical insulation and tubes designed for operation in oil could be made more compact.

To reduce the fragility of the tube, metal shrouded "shockproof" tubes were introduced. The metal case would be connected to either the anode or cathode, whichever was to be held at ground potential.

Around 1925 the 45° angle of the anode was changed to 22.5°. With this the cathode changed from a spiral within a concave cup to a straight coil placed inside a shallow groove. This was the Benson, or Goetze, "Line-Focus." This configuration projected a long cathode stream to the anode, as viewed from the cathode end, but resulted in a very shallow angle from the exit. This permitted a sharper focus without causing accelerated wear of the anode. Most of today's tubes employ this feature [3]. Figure 6 shows this configuration which is further detailed in Figure 5.

It is interesting to note that Coolidge had a patent on the hard-vacuum, hot-cathode tube. Many attempts were made to circumvent the patent. The author once had a C.H.F. Mueller tube which had helium in it, at least initially. Far from being essential to the operation of the tube, after a few hours of operation the helium would permeate the materials of the tube rendering the device a hard-vacuum tube.

Cold cathode field emission tubes were developed in 1941. These relied on the development of a very high

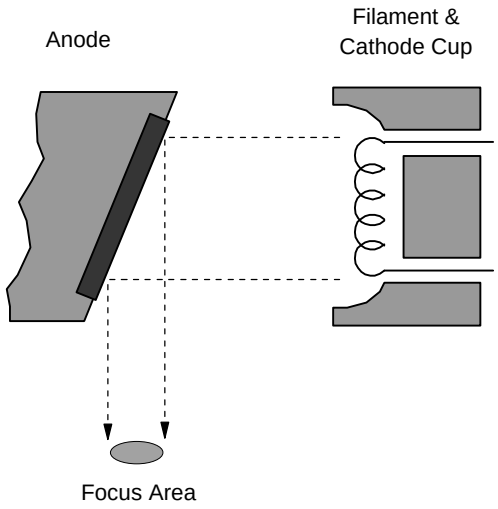


Figure 6 - Line-Focus Configuration

electric field at the tip of a pointed cathode which would then permit the passage of electrons across the high vacuum of the tube. Such tubes are not suitable for steady-state operation but they are excellent sources of high intensity x-ray flashes of short duration, rendering them ideal for the study of objects in motion, e.g. photos of bullets moving through gun barrels, the movement of organs within the body, etc. Power supplies for field emission tubes are usually some sort of capacitor discharge circuit such as high potential Marx generators, pulse forming networks, etc. Voltages from tens of kV to millions of volts are used.

In the 1940s the rotary anode tube was introduced. This provided another method of anode cooling by distributing the focal spot across a larger effective anode area. The mechanism is basically an induction motor with the armature located within the tube. The field windings are located outside. All modern medical (not dental) tubes are of this design.

Higher voltage tubes have been made to inspect steel and concrete fabrications over a foot thick. These use a very long tube, perhaps six feet or more in length, arranged like a potential drop particle accelerator. The filament, held at high potential, is located at one end and the target anode is a grounded disk at the opposite end. In between there may be several focusing electrodes: aperture plates that are held at intermediate potentials. The generated x-rays pass through the anode plate in much the same fashion as Roentgen's original tube.

High voltage tubes of this type may be operated at potentials of 1 million volts or more. The power sources generally operate at higher frequencies than 60 Hz in order to reduce weight and losses in the transformer core. Ac transformers may run at 180 Hz or high frequency resonant transformers may be used. Taps in the transformer secondary are connected to the intermediate electrodes.

Beyond the Roentgen and Coolidge tubes and their variants, there are now other sources of high energy x-rays including the Betatron accelerator, linear accelerators, and so forth. These devices provide incremental energy "boosts" to clumps of electrons which can ultimately provide energies of many millions of volts without the use of extremely high voltage power supplies. These devices can be used to inspect large castings in a fraction of the time that even a million volt tube could.

Detection and Miscellaneous Notes

Roentgen originally used a barium-platino-cyanide screen to detect x-rays. For regular use, this material proved to be too expensive. It was also found that the cyanide screens deteriorated with use.

Columbia University commissioned Thomas Edison to find a cheaper substitute. He eventually suggested calcium tungstate, a powder similar to the coating used in modern fluorescent tubes. Although it was more economical, its image was not nearly so bright and barium PtCN continued to be used. Quinine bi-sulfate was also tried but it too produced only a dim glow [1]. Other materials, or phosphors as they came to be called, suffered from long after-glows or had other disadvantages. Real success was eventually had with zinc-cadmium sulfide, to which was added a trace of silver. This glowed ten times as bright as calcium tungstate and did not deteriorate. The ZnCdS-Ag phosphor became known as Patterson Type B.

Radiographs that took a long time to make an exposure proved to be a problem. In order to shorten the exposure time, Prof. Michael Pupin of Columbia University sandwiched a photographic plate between two BaPtCN screens. The exposure time was reduced by a factor of 16 through the use of these "intensifier" screens. Today there are other enhancements including faster films and lead screens that improve detail by reducing x-ray scattering [3]. Intensifying phosphor screens are available in a wide variety and typically use rare-earth phosphors that are imbedded in flexible plastic sheets. Finally, image tubes are routinely used to intensify real-time images and reduce the image size for compatibility with conventional 35mm film and video camera formats.

Cited References

[1] F. Strong, *The Electrical Experimenter*, Sept. 1917. (Available through Lindsay Publications.)

[2] *National Geographic*, Jan. 1987.

[3] W.T. Sproull, *X-Rays in Practice*, McGraw-Hill (1946).

[4] *Hawkin's Electrical Guide #10*, Audel & Co. (1916).

[5] W.H. Meadowcroft, *Experimental Science* (1906). (Available through Lindsay Publications.)

Other References

J.D. McCoy, *Dental and Oral Radiography* (1922).

H.M. Muncheryan, *Industrial Radiology and Related Phenomena*, Aircraft X-Ray Laboratories (1943).

Roentgen's two communications that describe his discovery of the x-ray are included in *Great Experiments in Physics*, Morris H. Shamos, ed., Dover (1959). The translations include commentary.

Originally published in Volume 4, Number 4.

Generating X-Rays with Receiving Tubes

Old TV tubes are used as cold cathode x-ray emitters in a simple apparatus developed by Bob Templeman. With some beam tubes, the intensity is adequate to make x-ray photographs of objects using standard films.

The earliest x-ray tubes were of the cold cathode variety. These tubes, referred to as Crookes or Hittorf tubes, were of the general class of gas tubes since the pressure had to be in the 'soft' vacuum range (about 10^{-3} to 10^{-4} Torr) to permit the passage of electrons from the cathode to the x-ray producing target in a so-called 'dark' discharge. Higher pressures would result in a luminous discharge (as in a neon lamp) with only a small potential drop across the tube. Lower pressures (a 'hard' vacuum) would result in no current flow regardless of applied voltage. Figure 1 shows an early form of cold cathode x-ray tube.

The cold cathode tube went out of use shortly after 1910 when W. D. Coolidge introduced a tube with a hot cathode (thermionic) electron emitter. The Coolidge tube, which uses high vacuum (i.e. below 10^{-5} Torr), has a number of advantages over the gas tube.

With the gas tube, the electron current, at a given voltage, is dependent the voltage across the tube which, in turn, can vary depending upon the degree of vacuum. Furthermore, the degree of vacuum will change over time. This will affect the spectrum (hence the penetrating quality) of the x-ray output as well as the intensity. With a heated cathode in a high vacuum tube, the electron current may be controlled simply by varying the filament temperature. Then, by varying the voltage across the tube, the penetrating power of the x-rays (a function of the x-ray energy) may be varied. Thus, two important parameters may be controlled independently.

Bob Templeman has been able to use conventional vacuum tubes as cold cathode x-ray tubes. He has done most of his work with the 6BK4B, a beam triode used for voltage regulation of high voltage, low current dc power supplies in color and black-and-white television sets. The tube has an octal base and a plate cap. Bob has also tried several other tubes including the 6EN4 (which is very similar to the 6BK4B), 3AT2, 3CZ3A, and 3BW4. He has found that all will emit measurable amounts of x-radiation but only the beam tubes appear to provide sufficient radiation to expose standard films. Selected specifications for the 6BK4B tube are provided in Table 1.

Since the tube is operated in a cold cathode mode, the tube's degree of vacuum is quite important. Bob found that about one in eight tubes is able to produce enough radiation to expose his film. One might ask "why not just heat the filament to get an assured, controlled emission of x-rays?" The answer lies in the basic characteristics of a high vacuum diode. Referring to Figure 2, a 'normal' vacuum diode, such as a rectifier tube, operates in a region where the tube current varies nearly linearly with the voltage drop. Thus, substantial increases in current would be required to produce a voltage drop across the tube significant enough to produce useful levels of x-rays. For normal tubes, the current would be well in excess of the tube's power rating. Normal operation for a rectifier tube is moderate to high current with a low voltage drop.

What is good for rectifiers is not good for x-ray tubes. In the case of the x-ray tube, the tube is operated in the upper part of the characteristic curve, the *saturation* region. This is also shown in Figure 2. In this mode, the voltage can be increased with little increase of electron current. Getting the right balance between current and voltage is part of each tube's

Table 1: Tube Specification - 6BK4B

Tube Function	Beam Triode
Base	Octal (8)
Pin Assignments	
Heater	2, 7
Cathode	1
Grid	5
Plate	Cap
Voltage/Current* (V/A)	6.3 @ 0.2
Maximum Ratings*	
DC Plate Voltage (kV)	27.0
Ave. Plate Current (mA)	1.6
Plate Dissipation (W)	40.0

* In normal use

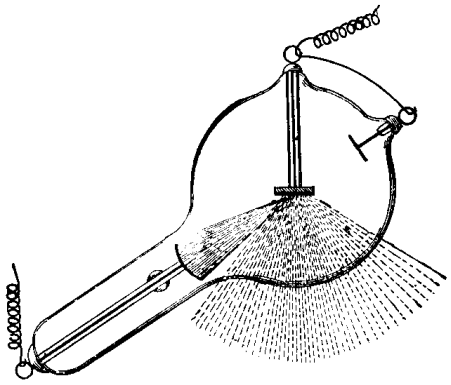


Figure 1 - Early X-ray Tube

design. Also, as noted before, varying the filament temperature (e.g. by means of varying the filament voltage) allows the intensity of the tube's output to be adjusted. For each filament temperature, there is a different current vs. voltage characteristic.

Bob uses a TV flyback driven voltage multiplier to power his tubes. Figure 3 shows the circuit. This is a fairly common implementation using a pair of general purpose NPN transistors to drive the 10 turn transformer primary which is added to the stock flyback. A four turn feedback coil is needed to sustain oscillation. This needs to be tightly coupled to the primary and it is best to wind it directly over the primary winding. 22 to 24 gauge wire is adequate for this winding.

Prepare and debug the flyback circuit before attaching the multiplier. Start with 6 volts applied and check for output from the flyback's high voltage lead. Do this by trying to draw an arc between the lead and a grounded piece of well insulated wire. (Be careful, the shock can be painful, or worse.) If the circuit is not working, try swapping the wires leading from the feedback coil. Proper phasing is required.

The multiplier is of the cascade (Cockroft-Walton) type. A modular tripler scavenged from a TV set can be used as these can usually be pushed to about 40 kV without failing. A better alternative is to make the multiplier from scratch using discrete diodes and ceramic disk capacitors. The diodes should be rated at 20 kV. A good value for the capacitors would be 0.001 μF at 15 kV.

As the flyback circuit will provide about 10 kV into the multiplier, six stages (note that there are two diodes and capacitors per stage) will be needed to boost the voltage to a maximum of 60 kV.

Assemble the multiplier on a piece of bare perf board with good separation between the components. To avoid excessive leakage or arcing, immerse the whole multiplier assembly in mineral oil. A rectangular plastic food storage dish makes a good container for this assembly.

A means of measuring the high voltage output is essential. A resistive divider is appropriate for this application. However, standard components are not suitable for high voltages, low current measurements. A good circuit for measuring the output voltage is shown in Figure 4. This uses a potted RCA focus divider which contains the necessary high voltage/high value resistors. The only additional components needed are one external resistor and a standard high impedance dc meter.

When testing the completed multiplier, avoid the temptation to draw sparks from the output. This will only stress the components and lead to premature failure.

When all is set with the high voltage circuitry and several candidate tubes are in hand, it is time to try generating some x-rays. First, make sure that you have an operating x-ray monitor. This will be needed for checking tubes for output as well as for checking the effectiveness of the shielding. Bob uses a simple Geiger counter circuit which is provided as a kit from Electronic Goldmine (P.O. Box 5408, Scottsdale, AZ 85261). This kit, #C6430, which has an audio output, currently lists for $59.95.

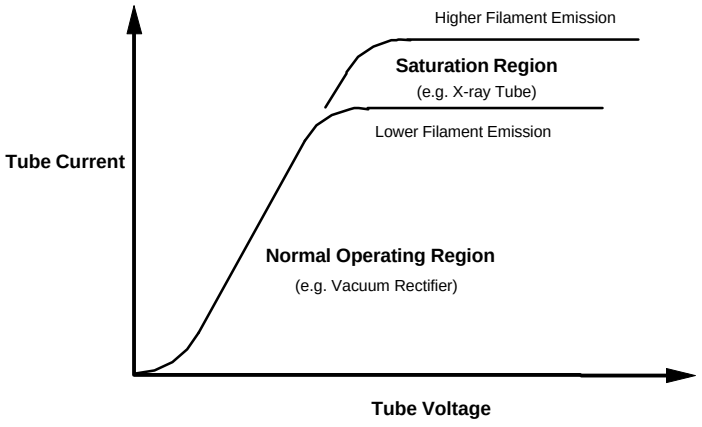


Figure 2 - Characteristic Curve of a Thermionic Vacuum Diode

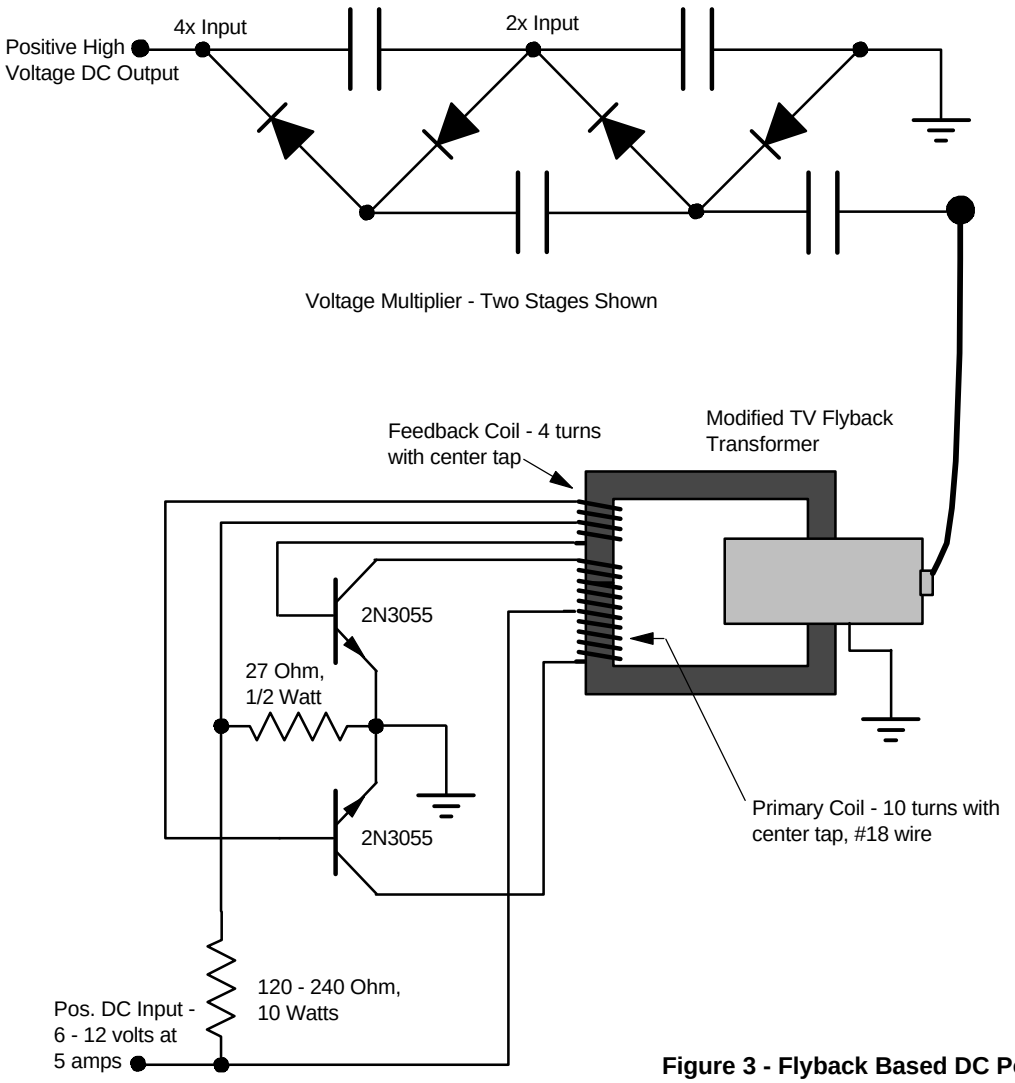


Figure 3 - Flyback Based DC Power Supply with Multiplier Circuit

Bob notes that the tubes tend to operate better when the cathode is positive, probably because of the slightly higher impedance in this configuration.

X-rays are nothing to be treated casually. Bob surrounds his tubes with 2 to 4 inches of lead. (At 60 kV, 1/16 inch of lead is the absolute minimum.) Maintaining a dosimetry program (see below) is advisable. Finally, the safest practice is to operate the tube from a remote location.

Arc-over is a problem at the voltages Bob has been using. Encasing the tubes in wax was tried but found to be only partially effective. Bob's prize 6EN4, the best emitter of x-rays, was destroyed in spite of this encapsulation. Immersing the tubes in mineral oil appears to be more effective. (Watch your druggist's face when you purchase the several bottles of mineral oil which will be needed for insulation of the tube and multiplier!)

Even operating in a cold cathode mode, the current through these tubes at 40 to 60 kV is enough to cause heating. Furthermore, as the tube elements warm up, the cathode begins to emit electrons thermionically. This leads to increasing dissipation, lowered potential, and a shift of the x-ray emission toward the soft, less penetrating, region of the spectrum.

To show that useful quantities of x-rays can be emitted from some standard vacuum tubes, Figure 5 shows the image of the internal circuitry of a small (about one inch square) plastic potted electronic module. The exposure was 40 minutes on Ilford

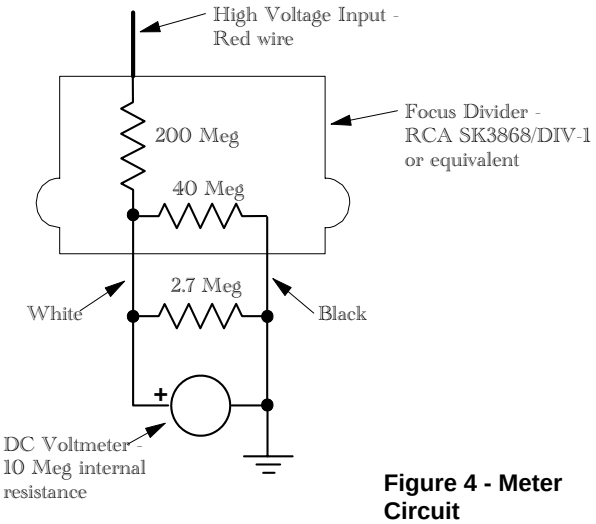


Figure 4 - Meter Circuit

Multigrade III photographic paper, ASA 25 (approximate). Tube current averaged 20 microamps. The image is somewhat fuzzy due to the fact that these vacuum tubes are diffuse emitters of x-rays.

FURTHER READING

Using standard vacuum tubes to produce x-rays is nothing new. C. L. Stong's *Scientific American Book of Projects for the Amateur Scientist* (Simon and Schuster, 1960) has a chapter describing Harry Simons' impressive experiments using antique 01 tubes driven by a homebuilt Oudin coil. Mr. Simons also fabricated a variety of his own tubes, simple bulbs with a sealed-in molybdenum cathode with a magnesium target. The latter was deposited on the inside of the bulb, opposite the cathode, and was capacitively coupled to the Oudin coil by means of a layer of aluminum foil which was wrapped on the outside of the bulb. Simons evacuated his tubes to 0.1 mTorr before sealing.

A large number of books are available which deal with the physics of x-rays, x-ray production, radiographic techniques, and safety. Before proceeding too far with experimentation with x-rays, please visit your local library to obtain further background information, particularly with regard to safety issues.

DOSIMETRY

Any person who regularly works with any combination of high voltage and vacuum should maintain a dosimetry program.

Landauer (2 Science Road, Glenwood, IL 60425-1586, (708) 755-7000) provides film and thermoluminescent (TLD) dosimeters as part of their service. These are provided as either wearable badges or as room monitors. To sign up for the service you select a monitoring frequency (weekly, monthly, quarterly) and pay a small set-up fee plus a year's payment in advance. Before the end of each monitoring period, you receive a new badge. At the end of each period you send in the current badge and within 5 days you receive a report giving dosage for the period plus cumulative dosage. For a TLD dosimeter sensitive to x-ray, gamma, and beta radiation with a quarterly schedule, the cost is under $100 for a year.

Another approach to dosimetry and one which gives a continuous record is a Geiger counter provided by Aware Electronics (P.O. Box 4299, Wilmington, DE 19807, (302) 655-3800). Their RM-60 is a small monitor which interfaces directly to an IBM compatible computer via a phone type cable to the serial or printer port. A dedicated PC is not required as the software gathers the data and stores it to disk even while the computer is running other applications. The software displays the data in a scrolling bar chart format with date and time for each bar. Also provided is the cumulative average dosage. Cost for the RM-60 package is about $150. Aware's catalog also describes several other radiation monitoring items.

Originally published in Volume 3, Number 1.

Figure 5 - X-ray Negative of Potted Module

Update *(from Volume 3, Number 2)*

Bob Templeman has continued to keep me informed with regard to his experiments with x-ray production from TV beam triode high voltage regulator tubes. Bob's work was featured in the last issue of this journal.

Bob has been investigating the sensitivities of various phosphors to the x-rays emitted from his tubes. He had no luck with the phosphor salvaged from a fluorescent light tube but he did get a faint fluorescence from the phosphor scraped from the face of a broken color picture tube. The brightness was about comparable to that from a piece of standard medical rare earth phosphor x-ray intensifier screen.

Bob also has sent along some more radiographs. These were made with Agfapan 400 sheet film and the results are much more impressive than what he had achieved before with the slower Ilford paper with a speed of about ASA 25.

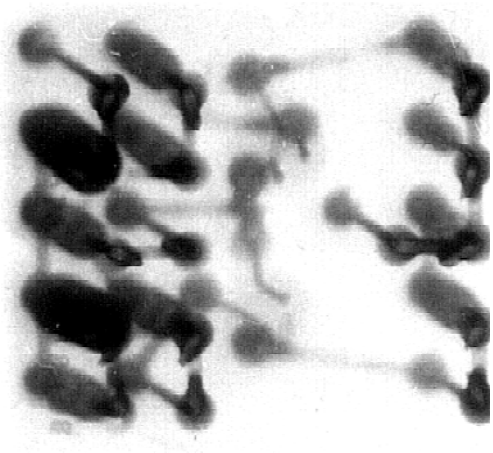
The accompanying pictures are a sampling of Bob's latest efforts. All exposures were 30 minutes with the tube about 8.5 inches from the film plane. Negative bias (40 kV) was applied to the 6BK4C plate cap. To avoid arcing, the tube was operated under mineral oil.

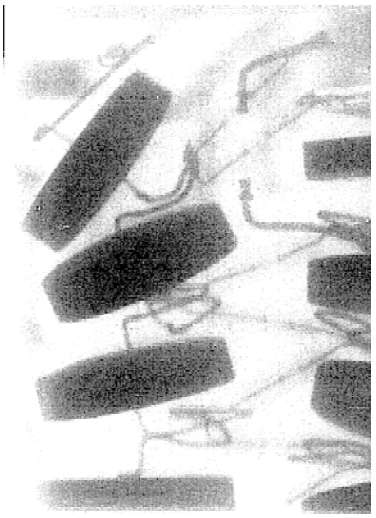
The first picture shows the same potted module that was included in the article in the Winter issue. Note the improved definition (much clearer on the original print, of course).

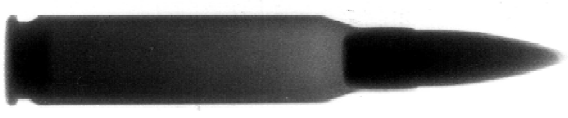
The image below is that of another potted module, this time a voltage multiplier circuit. Note the good definition showing clearly the components, their wire terminations, and the traces on the printed wiring board.

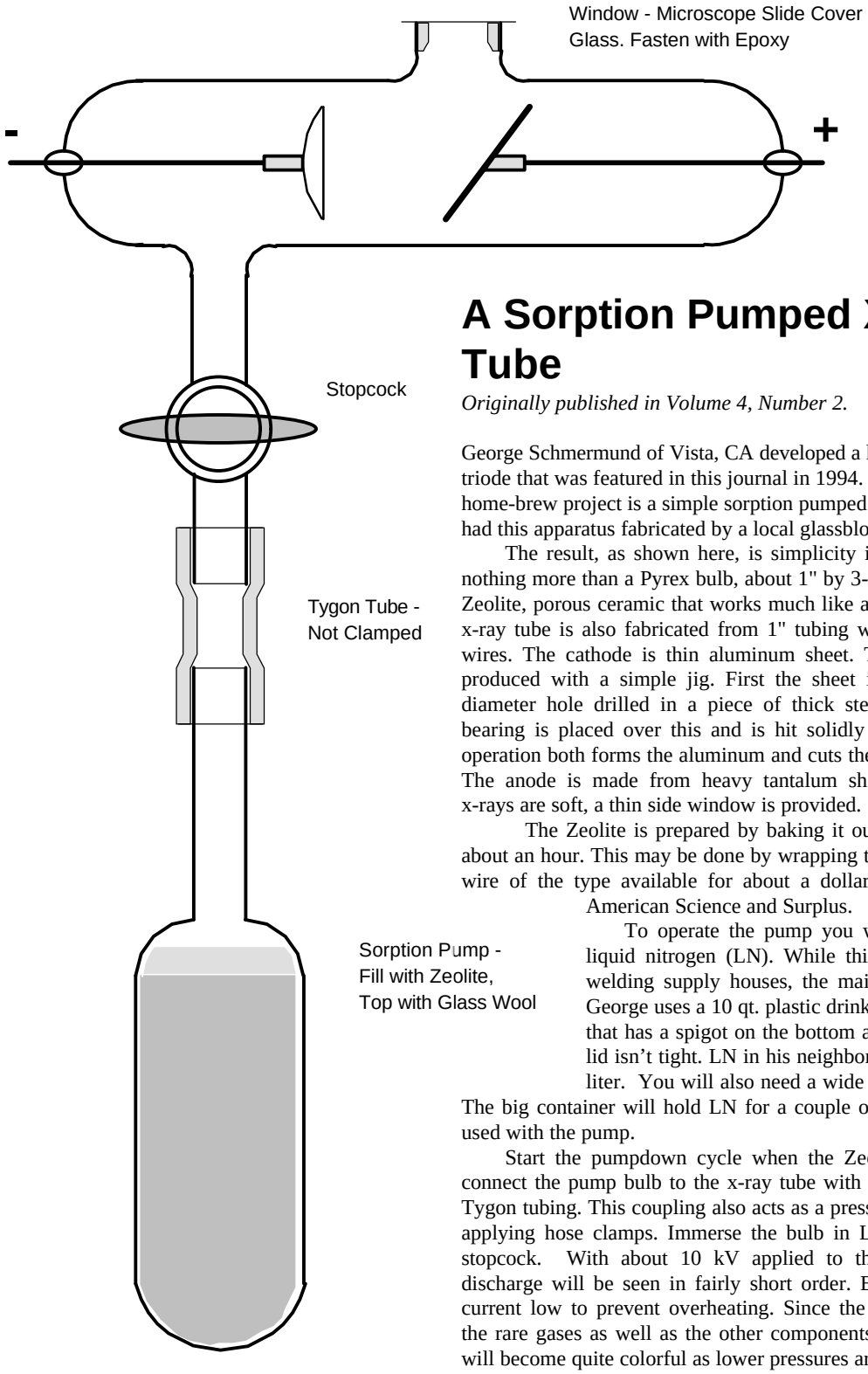
Last is a radiograph of a .308 Winchester cartridge. Here the bullet is shown seated in the neck of the shell. The primer is visible in the original print.

Overall, I am quite impressed with the results that Bob has achieved with his simple apparatus.









A Sorption Pumped X-Ray Tube

Originally published in Volume 4, Number 2.

George Schmermund of Vista, CA developed a homemade vacuum triode that was featured in this journal in 1994. His most recent home-brew project is a simple sorption pumped x-ray tube. George had this apparatus fabricated by a local glassblower to his design.

The result, as shown here, is simplicity in itself. The pump is nothing more than a Pyrex bulb, about 1" by 3-1/2", that is filled with Zeolite, porous ceramic that works much like activated charcoal. The x-ray tube is also fabricated from 1" tubing with sealed-in tungsten wires. The cathode is thin aluminum sheet. The concave figure is produced with a simple jig. First the sheet is placed over a 3/4" diameter hole drilled in a piece of thick steel. Then a large ball bearing is placed over this and is hit solidly with a hammer. This operation both forms the aluminum and cuts the circle from the sheet. The anode is made from heavy tantalum sheet. As the generated x-rays are soft, a thin side window is provided.

The Zeolite is prepared by baking it out at about 350 °C for about an hour. This may be done by wrapping the bulb with a heating wire of the type available for about a dollar from C&H Sales or American Science and Surplus.

To operate the pump you will need to get some liquid nitrogen (LN). While this is easy to get from welding supply houses, the main problem is storage. George uses a 10 qt. plastic drink cooler. Don't use one that has a spigot on the bottom and make sure that the lid isn't tight. LN in his neighborhood is about 60¢ per liter. You will also need a wide mouth thermos insert. The big container will hold LN for a couple of days, the thermos is used with the pump.

Start the pumpdown cycle when the Zeolite is still hot. First connect the pump bulb to the x-ray tube with a coupling made from Tygon tubing. This coupling also acts as a pressure relief so don't try applying hose clamps. Immerse the bulb in LN and then open the stopcock. With about 10 kV applied to the electrodes, a glow discharge will be seen in fairly short order. Be careful to keep the current low to prevent overheating. Since the Zeolite doesn't pump the rare gases as well as the other components of air, the discharge will become quite colorful as lower pressures are reached.

After a while, a dark discharge condition will be achieved. As the 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.

Geiger Counters and Power Supplies

David M. Raley, PE
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I. INTRODUCTION

By the time you finish this paragraph thousands of gamma rays, or particles, will have passed through your body, a number of beta particles will penetrate your skin to varying depths and your skin will repel some alpha particles. This is so even if you are not playing around with high voltage and vacuum. If you are so engaged, there is a good chance that your dose is higher.

The only alpha particles you have to worry about are the ones that are emitted by radon or other gases that you have breathed into your lungs. You can also forget about the gamma radiation that makes it straight through your body. That leaves the beta particles that penetrate and the gamma rays that interact. "Them's be the buggers to sweat." If you are a member of the majority of readers who already know these things, please be patient while the 10 percent who didn't get the word are brought up to speed.

Generally speaking a sub-atomic particle travels until it collides with the nucleus of an atom. It may then be absorbed by or bounce off the nucleus. If it is absorbed it will affect the nucleus in some way. From time to time that nucleus is in an atom in the DNA chain of one of your cells. A possible result is that the cell will mutate the next time it replicates. The vast majority of mutations are degenerate; most of them simply die, some of them become cancerous. If your immune system is working right the mutated cells are eliminated. Females face an extra hazard in that it sometimes happens that a ripe egg is hit and the immune system mistakes it for having been fertilized and allows it to attach as would a zygote. The result is almost always a tumor but parthenogenesis is a fact of life despite all the jokes about the doctor searching the horizon for the camels.

There has always been background radiation, more in the distant past than now. Some say that were it not for background radiation, life would not exist. I don't know about that; my belief is that we are stressed to tolerate a certain amount of the stuff and no more. What's the limit for long term exposure? Two percent above background? Five? Ten? Fifty? Two Hundred? I don't know that either. What's the goal? To keep our surroundings as close to the natural background as possible.

Before you can take precautions you have to be able to measure what you are trying to avoid. Low level radiation gives us a special problem. It is completely random in nature. While you would have no difficulty at all in detecting an increase of two percent if you were measuring fruit, flour or frequency. You could watch the meter of a Geiger counter from now on and never be able to say for sure if the background radiation was up or down by that amount.

The solution here is an accumulation Geiger counter. The classical device counts each interaction that takes place in the detector tube over a selected period. The longer the selected period the more the random nature of the interactions is smoothed out. By interfacing with a computer you can learn many fine things with a minimum of effort. One of the things that you can learn is whether or not there is an alpha emitting gas, say radon 220, in the air you breathe.

I have made a lot of closed tube readings using counts per hour, finding that the measurements vary for different locations within my house and that the safest place on my property is in my dog's house where the average is 3,033 counts per hour. The hound's house is wood, mine is masonry. Most likely the reduced risk of stray bullets from hunters or from woods fires makes the masonry safer overall. There is no doubt at all that masonry would give more protection from regional short term nuclear events.

How about your house? Would you be safer outdoors? Would you be better off without that super-insulation? Should you leave the room, or the county, when your grade-A, US Choice science project is running? If there were some sort of nuclear accident nearby would you be able to prove that your property had a safe background level before the accident? Would you even KNOW if there were such an accident if it happened one dark night and nobody talked? Is that rock your kid brought home a stray radioactive meteorite?

Stay tuned and learn how to build your own accumulation Geiger counter.

II. BACKGROUND

The Geiger counter is named for the Geiger tube. The Geiger tube was invented by Hans Geiger and improved by Wilhelm Müller. The proper name is Geiger-Müller tube or counter but it is almost always shortened to Geiger or G-M.

The tube consists of a gas filled outer cylinder with a fine wire running the length of the center. Brass is often used for the cylinder, tungsten for the wire and argon for the gas. I have used copper tubing for the cylinder and stainless steel guitar strings for the wire with good results. The cylinder can be structural or enclosed in glass. Almost any gas, even air will work with some degree of satisfaction. If you are looking for direct alpha from household radon you'll use air and like it, but the best gas for most purposes is argon with a little bit of impurity for quenching. Many tubes on the market use a mixture of neon and helium.

When the proper voltage is applied across the tube through a dropping resistor about one percent of the particles passing through the gas will cause it to ionize and conduct. (Figures 1 and 2) When the tube conducts, a voltage pulse can be detected across either the resistor or the tube. An especially energetic particle can cause more than one pulse. Multiple pulses (bounces?) are also produced when the tube is right near the glow discharge voltage. The conduction continues until the ionization is quenched. Some tubes are self quenching and some are externally quenched. Until the tube

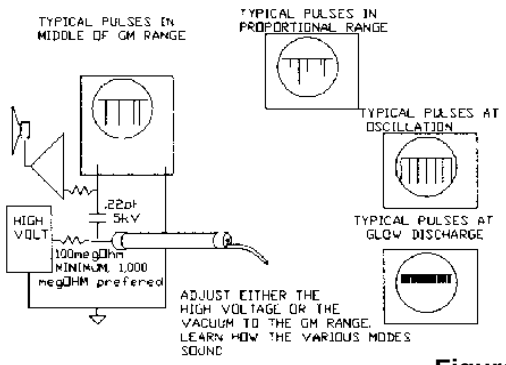


Figure 1

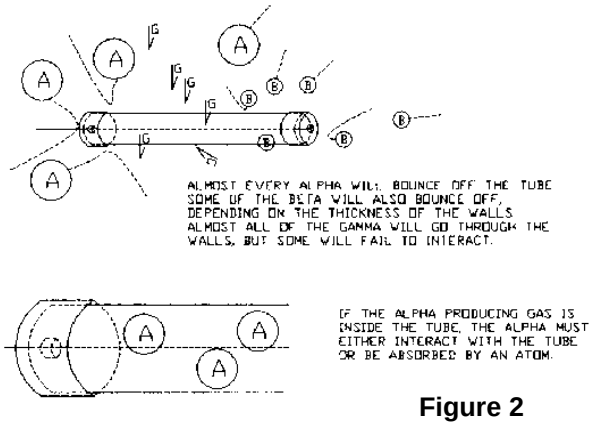
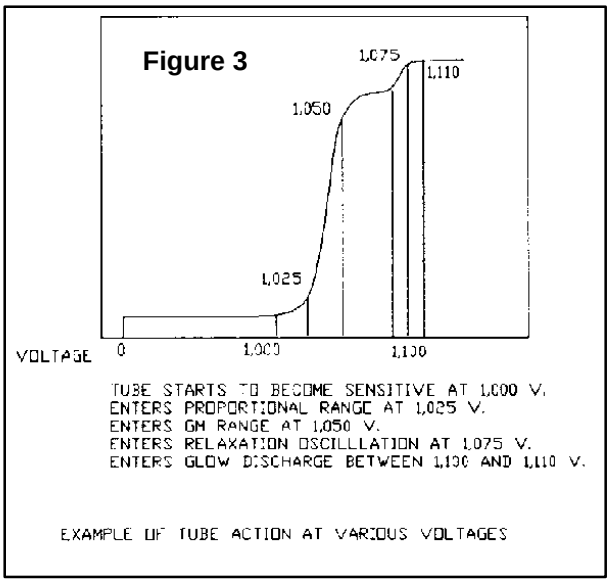


Figure 2

is quenched it cannot count another pulse. This places a limit on the number of pulses it can count before saturation. Saturation is not going to be a problem with background radiation. Tubes have been made in a wide range of sizes; larger to detect low levels of radiation, smaller for high levels. The smaller the size the fewer particles pass through it. The voltage on the tube is such that all pulses are of equal strength. This is known as the Geiger-Müller plateau (Figure 3). If the voltage is lower the tube will act as a proportional counter; if too high the tube will fall into glow discharge and not count. A proportional counter finds use in the laboratory where it is attached to a "kick-sorter" to reveal the relative energy content of the particles it counts.



III. COMPUTER INTERFACE DESCRIPTION

While space does not permit the presentation of the circuit schematic and components layout, the following sections describe in general detail the computer interface and the accompanying software program. As noted later in this article, detailed information may be obtained from the author for a nominal fee.

Figure 4 shows the general organization of the system. The raw pulses from the G-M tube go through an LM1458 operating as an Amplifier Clipper. The shaped pulses go:

1. To the base of a 2N3904 and thus to a small speaker.
2. To an "on board" binary counter.
3. To a pulse stretcher . The width is controlled by the dipswitch.

The output to the computer is from one section of a 4016 quad bi-lateral switch. That's right, this rascal feeds right into the joystick socket and will run on almost any computer. Eat your heart out RS-232.

PROGRAM DESCRIPTION

The program was written for the Radio Shack CoCo and shouldn't require a whole lot of hacking to adapt to whatever hardware you might use. (You can hack line 130 to match the half-life of the element you are measuring for). Here is what it does:

1. Asks if the joystick socket is getting a closed circuit. If so a pulse is counted.
2. Asks if the joystick socket circuit has opened up yet. If not, it keeps asking. If so, it asks if it has closed again.

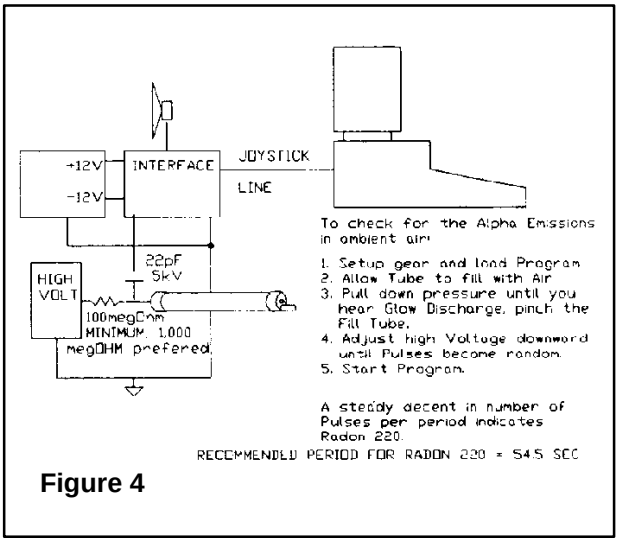


Figure 4

3. Keeps up with the time. This can be a standard time interval, one minute as written, or a half-life.
4. Keeps up with how many periods have been counted and stops at a predetermined number; 10 as written.

The pulse stretcher on the interface keeps a pulse from getting by during housekeeping.

STAND ALONE COUNTER

If you don't want to fool around with a computer you can attach a counter module such as the "CUB", available from Digi-Key, across the speaker. Alternately, you can feed the pulses into an integration circuit and use a meter. A meter won't be of any use for background and low level readings but it might tip you off if your project starts radiating. As a matter of fact, you can dispense with the active circuitry entirely and feed the pulses from the tube through a protection circuit and directly into the CUB.

IV. POWER SUPPLIES & THE G-M TUBE

Whether to buy or build your tube is best answered with "do both." Unless you are very handy with glassware, a store-bought tube is best and cheapest for building an instrument that will give consistent readings. To have some fun you should build your own.

Figure 1 has about as few parts as you can get and still have a circuit. The positive pole of the high voltage goes to the center wire of the G-M tube through a high value, 100 Meg to 1,000 Meg resistor, and the body of the tube goes to ground. This is backwards from the instincts of old-timers who tend to think of the outermost element as the "Plate." The pulses are carried off through a 33 pF 5,000 volt capacitor. Your friendly television technician can provide both the resistor and the capacitor.

POWER SUPPLIES

Battery Packs: You want a high voltage supply that is both stable and variable. One way is to buy yourself 150 9 volt batteries, a 24 position rotary switch and 24, 10 k resistors and assemble them into a pack as shown in Figure 5. The useful operating range of a 3/4" diameter air-filled G-M tube at one tenth of atmospheric pressure is only about 50 volts wide and starts in the neighborhood of 1,300 volts. If you intend to do all your experiments with a fast tube such as the 5979 you can get by with about half the batteries and without the switching system, even fewer for the LND712 or LND713. The operating life of the battery pack is about the same as the shelf life. I assembled a similar set three years ago and used it until recently. The only limitation on continuous operating time with that particular configuration was the life of the battery that operated the counting module.

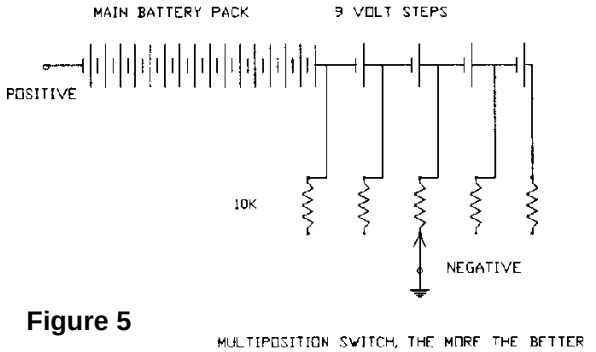


Figure 5

CAUTION CAUTION CAUTION: Be very careful when assembling this high voltage pack or it may last you your entire lifetime. Use plastic probes to attach the terminals. Don't "improve" the circuit by running the positive side through the on/off switch. Not only can the pack shock you, it can burn you. Assuming a nominal short circuit current of 250 milliAmps, the pack can deliver 349 watts. The bulk of the pack is just batteries strung together by their own terminals. The last 24 are connected, through 10 k resistors, to the rotary switch. The resistors keep the switch from putting a direct short circuit on the switched batteries. Put a little pinch of conductive foam at each snap connection. I didn't do that with mine and had to tighten up several connectors with pliers, getting the Peruna knocked out of me in the process. I have found that the best pain killer for an electrical burns is Americaine. It is about the same thing as Preparation H.

Medium Voltage Battery Operated Supply: This supply, (Figure 9) operates on the same theory as the high voltage supply which is described below. A rapidly falling pulse causes a rapidly collapsing field which generates a spike that is much higher than the voltage induced in normal operation. A six volt sine wave on the low side, for example, would produce only 60 volts in the secondary. The maximum output is limited by the insulation of the transformer and the maximum reverse voltage of the diode. If you want more than 750 volts you should use a doubler or a tripler. This circuit, as drawn, has a wide latitude. I use it with 5979 and LND712 tubes.

High Voltage Supply: This circuit (Figure 10) takes care of the top end of any Geiger need and nibbles at the lower edges of other vacuum projects. It uses either a flyback or an ignition coil. One advantage of using a flyback transformer is that the frequency of the drive current is higher. This allows you to use smaller filter capacitors. And, if you have a little age on you, you can't hear the coil sing. This same circuit, tuned lower by making C1 larger and used with an ignition coil and a single diode will give you all the voltage you will need for almost any Geiger project.

To convert the flyback from a tube type color TV:

1. Take it apart.
2. Dispose of all windings except the high voltage winding.
3. Re-assemble using eight to ten turns of #10 wire wound on the core inside the high voltage winding if possible. Less satisfactory results can be had by winding the new primary at the

other side of the core.

If first glance tells you that there are a lot of "unnecessary parts," I'll speculate that your other HV power supply runs hot and eats up a lot of components. This rascal will make 33 kV day and night and never get the heat sink hot enough to be uncomfortable to the touch. (74 degrees ambient, 92 degrees heatsink is typical.) The reason for running the output of the oscillator through a counter and AND gate is to create a fixed relationship between the drive pulse and the idle time. 1-16 is the best easy ratio to obtain. Every transformer has its best frequency and if you feed the output of the oscillator directly to the driver, you have the problem of adjusting both the frequency and the pulse width.

The level of the pulse is also important. If you drive it too hard, you will overheat the output transistor and produce LESS voltage. The excess drive is stored in the base spreading resistance, the transistor stays on too long and the cutoff is gradual rather than sharp. Of course, too low a drive level will also reduce the voltage. Two things control the level of the pulse. The VCC on the AND Gate and the setting of the drive pot. If you don't intend to use the regulator circuit, just tie VCC to the 12 volt bus.

The high voltage regulator is the most successful I have economically built. A sample of the high voltage is applied to the input of one of the OpAmps. As shown the gain is 1. More aggressive regulation could be had with higher gain. The second OpAmp has a reference voltage on the positive input and the output of the first OpAmp on the negative. If the high voltage sample is zero, then the second OpAmp applies maximum voltage to the AND Gate VCC. As the high voltage rises, this output to the VCC starts to drop. It will be ten times the difference between the voltages of the positive and negative inputs on the second OpAmp, when the gain pot is at maximum.

Regulation is a compromise between the most output and precision. As drawn, this circuit favors output. When warmed up it is stable to about two percent at 33 kV. The difference from hot shutdown to cold turn on is seven percent. Higher gain in the first stage will improve the regulation at the expense of the output. A much more complex circuit will give both, but be prepared for a LOT of "unnecessary" components.

Note that the buffer transistor is a Darlington and the output transistor has a built in damper. These transistors are designed for television high voltage supplies. If you substitute with audio grade transistors you run the risk of arcs in the secondary of the flyback reflecting into the primary and burning up both transistors and the drive circuit. When I was developing this design I used a B&K function generator for the drive. In due course a reflected arc found its way back to the generator. (Anyone wanting a more than slightly scorched B&K may contact me and hear of a bargain.)

To set up the circuit for operation:

1. Have everything built and connected.
2. Set the drive pot to minimum.
3. Set the gain pot to maximum.
4. Set the frequency pot to the middle of its range.
5. Set the feedback sample switch to off.
6. Turn on the exciter.
7. Adjust the reference pot to the point just below where the output of the second OpAmp stops rising.
8. Verify that the positive pulse is 1/16 the of the total time period between pulses.
9. Apply voltage to the driver/output transistors.
10. Slowly advance the drive pot to where the system is making four or five thousand volts.
11. Tune the frequency pot to maximize this voltage. If it goes over 20 thousand volts, adjust it down with the drive pot. Go back and forth from the frequency pot to the drive pot to get the most voltage with the least drive. Set the output to 20 thousand volts.
12. Turn the feedback switch on. The high voltage should droop about 25 %.
13. Advance the drive pot to the high voltage value that you want.

Raising the supply voltage to the output transistor will allow you to go even higher output voltages. The limitation then becomes the adequacy of the tripler. My experience is thus: Just above 100 kV the tripler threw in the towel, there was a flash, there was smoke and the meter momentarily read off scale, (200 kV range). This problem can be avoided by making a tripler out of single high voltage diodes and capacitors. When I need more than 40 kV I'll put one together and let you know.

MAKING THE G-M TUBE

Start with a five to seven inch length of 3/4" copper or brass tubing. Rigid is better because it will be straight when you buy it. Referring to Figure 6 you will also need:

1. A .007 stainless steel guitar string
2. Three to five inches of automotive rubber tubing
3. Two plastic end caps
4. Epoxy

Clean the inside more that you think it needs and don't leave any lint on the walls. I use Ivory Liquid soap and a soft toothbrush. If there is any roughness or corrosion on the inside, sand it out with fine sandpaper. Don't use steel wool. Sand the outside of the ends as far as the end caps will reach

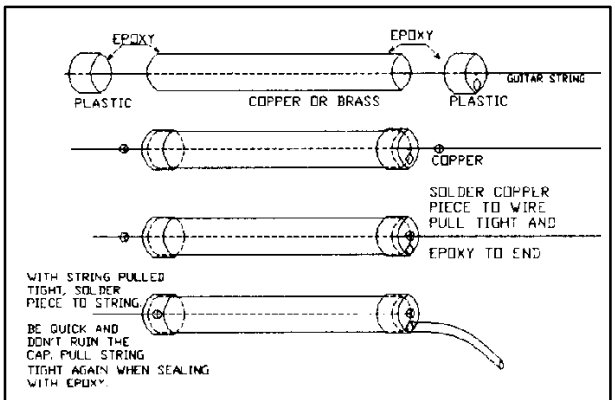
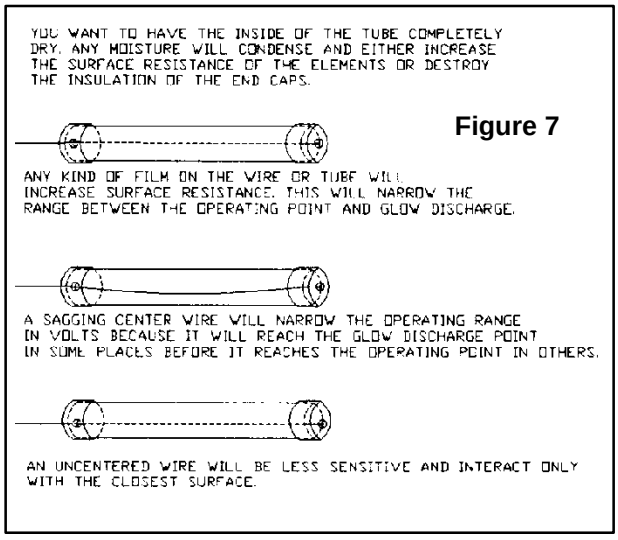


Figure 6

and sand the inside of the end caps. Drill a hole in the exact center of each end cap. This hole should be just large enough to let the guitar string pass through. Drill another hole near the edge of one of the caps that will just pass the automotive rubber tubing, the filling nipple. CLEAN UP! Never since the foundation of the Earth has a G-M tube been too clean or too thoroughly rinsed. Thread the guitar string through the center hole of one end cap, through the copper tube and out the other end cap. Don't cut the little piece of brass off the standing end of the string until the tube is finished. Tie a knot in the bitter end. This will keep the string from slipping out. With the tube held vertical by a pair of Vice Grip pliers, slather a right smart of well mixed epoxy onto the outside of the bottom end of the tube and into the inside wall of the end cap. Press the end cap onto the tube as far as it will go. Twist and work the joint so that the epoxy makes a solid seal. Stop messing with it while the epoxy is still very fluid. Let stand for half an hour, flip over and do the other end. (Flip the TUBE over, you understand.)

Crimp a small piece of 1/8" copper tubing onto one side or the other of the guitar string. You will have to untie or cut it, of course. Be careful not to lose the end into the tube. Fill with 60/40 solder. Attach a weight to the bottom end of the string. Make the end you have prepared fast to the end cap

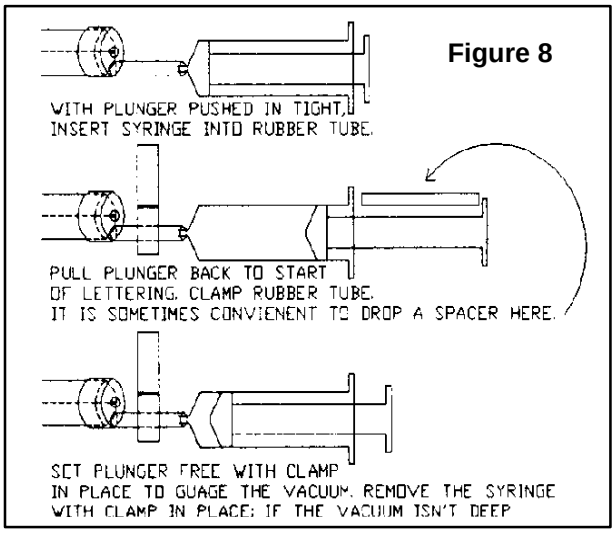


with epoxy. ALWAYS MAKE A FRESH BATCH OF EPOXY FOR EACH OPERATION. Do the same trick on the other end, this time attaching the weight to the tube and hanging it by the string. You want the string to be tight enough to prevent sagging as any sag will narrow the operating range of the tube, but don't make it so taunt as to cause it to pull the epoxy seals loose. (Figure 7) Coat one end of the rubber tubing with epoxy and work it in and out of the hole prepared for it. Remember to let each joint set hard before starting on the next one. With all joints made, test the tube to see if it will hold vacuum.

You don't need a laboratory vacuum pump or even a refrigeration compressor. A medical syringe or the vacuum from the intake manifold of a 1979 Ford LTD will do. Clamp the rubber tubing with a hemostat before disconnecting from the vacuum source and set the tube aside for a while to see if it holds. If you are using a medical syringe (Figure 8), you will have to work it several times to get down. Evacuate, clamp, disconnect syringe, press in plunger, reconnect, unclamp, evacuate, etc. If you let go of the plunger after you clamp but before you disconnect you can get a fair idea of the depth of the vacuum. The first few times you pull the vacuum the epoxy will outgas grossly. I once made an enclosed tube out of plastic pipe with a brass insert for the cylinder and equipped it with what looks like a tire valve but is in fact a fitting for pumping air into home water tanks when they become water-logged. Using argon, I alternately pumped it to 2 atmospheres and bled it off until I had 97% Argon. It worked fine the first day, but as days went by I had to keep raising the voltage, which was high enough to begin with because of the positive pressure. The plastic cement was outgassing and contaminating the argon. Note that you need some impurity when using argon or the tube won't quench. Air works pretty well for this. A whiff of ethyl alcohol works better.

To use argon in the tube you are building bring the vacuum down to the point where the syringe plunger, when turned loose after clamping, goes in all the way. Let in a syringe full of argon. Then pull the mixture back down to 1/10 atmospheric pressure. I pull the plunger out to 55 cc and call it 10 percent at 5. Most welders have a tank of sufficiently pure argon and will let you have a small amount. It is best captured in a dry condom although an ordinary balloon will do if you don't take too long getting the gas back home. Try to have as little air as possible in whatever you make the capture with. Drugstore clerks often feel obliged to point out the advantages of lubricated condoms and fail to take your meaning as you explain that the lubrication will outgas and contaminate the copper tubing.

We are talking about a slow G-M tube here. Making a fast tube is a little more involved and if your time is worth more than $3.21 an hour it is cheaper to buy one. If you make a fast tube and want it to stay fast you will have to make it of glass. Slow tubes are fast enough to monitor anything that you want to stay in the room with. I say again that you should make a slow tube to play and learn with. (There are lots of



things that you can't do with a store-bought tube.) Then buy a fast tube to make a portable, permanent, reliable instrument.

While all the parts needed for the project are available from electronics parts dealers or building supply stores it is conceivable that the reader might not want to buy, for instance, a ten foot section of copper pipe, (as I did from Z. V. Pate's General Store at the Laurel Hill Crossroads where there's plenty of free paved parking), and might wish instead to simply have a kit to put together. Therefore, in a spirit of greed and avarice I offer the following:

Basic Kit ($13.00) consisting of:
- 1- A set of full sized drawings
- 1- 7" length of 3/4" rigid copper pipe
- 3- Plastic caps to fit above
- 1- Guitar string
- 1- 24" length of rubber vacuum hose

Extended Kit ($21.00) consisting of:
- The Basic Kit plus
- 1- 60 cc syringe (minus the needle)
- 1- Hemostat

Alternate Extended Kit ($1,39.26) consisting of:
- Same as above except 1979 Ford LTD in place of 60 cc syringe. (Quantity limited!)

Shipping weight for the basic kit or extended kit is one pound. Add enough to cover your choice of shipping. If you want the alternate extended kit you'll have to come and get it. Bring plenty of motor oil.

There is no good reason for you to buy an etched circuit board for the computer interface or any of the other electronics. The only complicated circuit is the perfboard layout (not shown). I'll even throw in a piece if you send for one of the kits. Should there be an outcry for etched boards I will fill the need at some exorbitant price.

If you want to buy a fast tube you might consider a type 5979. I have used it myself with a high degree of satisfaction. This tube is available surplus for around $120.00. A smaller non-MilSpec alpha, beta, gamma substitute, the LND712 was recently selling for $58.00 and a beta/gamma only LND713 for $48.00. Another type tube on the surplus market in the hundred dollar range is the 2-01C. These tubes operate on various voltages and have a wide operating plateau.

OPERATION

Play with the tube with an oscilloscope for a while before you connect it to the interface (Figure 1). Once you have learned the quirks and "feel" of your tube you are ready to check for radon 220. Connect the equipment as shown in Figure 4, Hack the program to make the period equal to the half-life of radon 220 (54.5 seconds). Run the program a few times with aged air or outdoor air.

Now, pump the tube down as far as you can get it, then let it fill quickly with room air. Start pulling a vacuum as soon as possible and try to get it down in less than a half-life. As soon as you hear the tube go into glow discharge, stop pumping and pinch the filling tube. Back the high voltage down to a point just below relaxation. Start the program.

ANALYZING THE RESULTS

If the readings start off higher than your reference readings with aged air, and decline consistently, you can be sure something in your air is emitting alpha. UNLESS you have adjusted the high voltage too close to relaxation and picked up extra pulses at the first, or too close to the edge of the G-M range and lost some pulses on the later readings. There will be a sag in sensitivity because the air temperature drops as you pull the vacuum, and the air pressure increases as the air comes back to ambient temperature. You will get the hang of it.

If you are a real thrill seeker you may want to experiment with an open tube. The structure is approximately the same but instead of boring one hole for the evacuation line, bore four of them in each end cap. This will allow for natural circulation of ambient air. Alternately, bore a single hole in one cap and four in the other so you can force air through faster. You can also filter it. Try an aquarium pump. Any high voltage supply that is stable with a means of reliable adjustment will do. The operating range of the tube is still 50 volts wide, and at this voltage it is a much smaller percentage of the total. Start off at about 3,000 volts and go up slowly until it barely saturates. Then back off a little. You can take your time since fresh room air is circulating between the electrodes. If you filter the air you take a lot of the fun out of the game as gnats and fruit flies will not be able to get in. Operating the tube alternately with the holes open, then closed with thin foil and noting the difference in counts will tell you how much alpha activity there is. Operating a high voltage tube has another advantage. You can dispense with the counting circuitry and attach a piece of string to the point where the center electrode meets the dropping resistor. The string will stand out when voltage is applied and jump with every count. If your power supply is unregulated you will have to adjust it more often as it warms up. Even a regulated supply needs occasional adjustment at this voltage.

Sources and Further Reading

A wide variety (subject, of course, to the vagaries of the surplus market) of G-M tubes may be obtained from Don Orie at OE Technologies, P.O. Box 708, La Madera, NM 87539, (505) 583-2482. Request their latest list. A supplier of new tubes is LND, Inc., 3230 Lawson Blvd., Oceanside, NY 11572, (516) 678-6141.

The chapter on Geiger counters in John Strong's *Procedures in Experimental Physics* (1938, reprinted by Lindsay Publications, Bradley, IL 60915) provides much useful and practical information on G-M tubes and their construction. An excellent, up-to-date text covering all standard methods of radiation measurement is Glenn F. Knoll's *Radiation Detection and Measurement* (John Wiley & Sons, 1989). - *Editor*

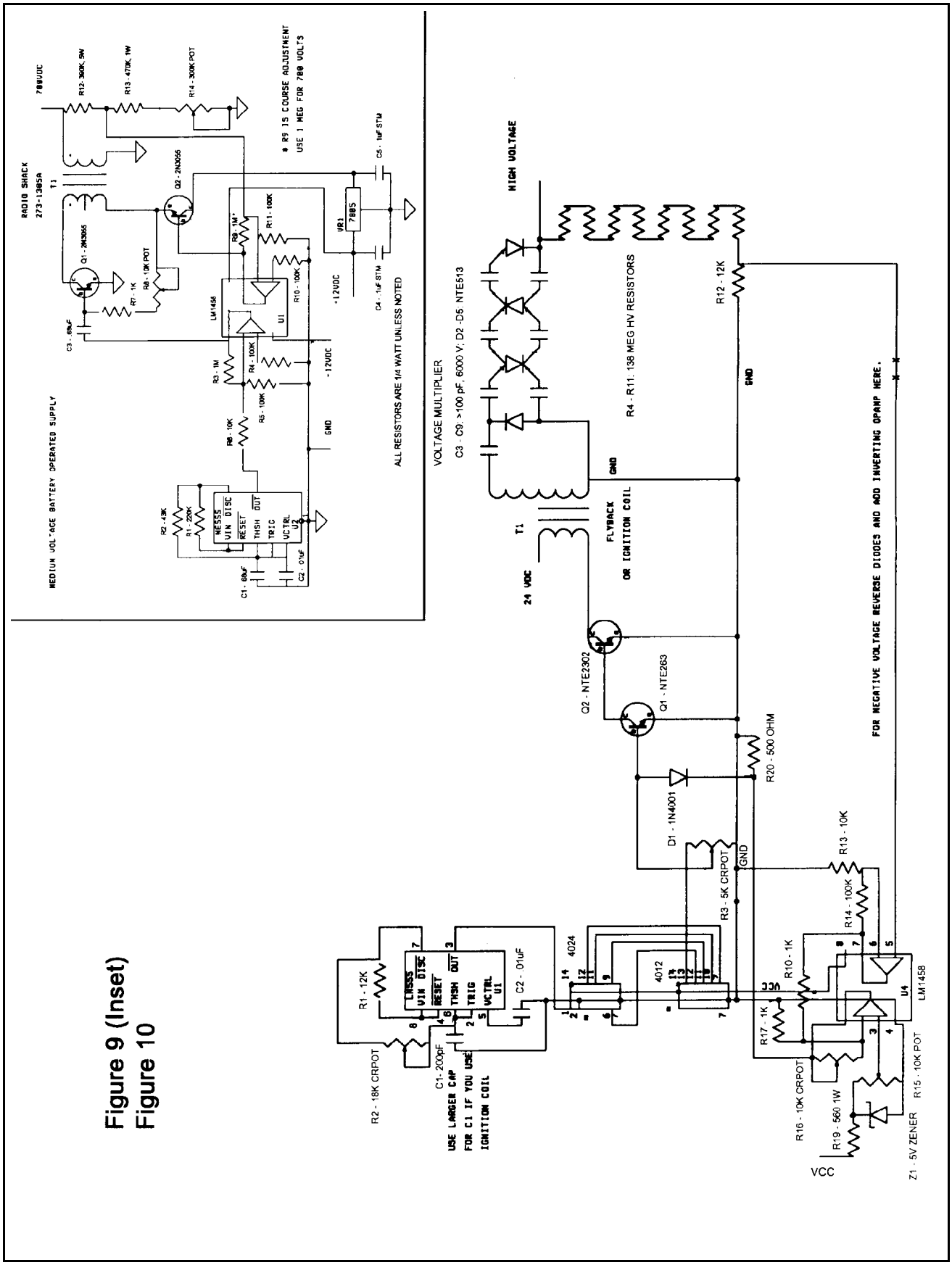


Figure 9 (Inset)
Figure 10

Plasma Accelerators - An Introduction

Steve Hansen

The principÌle of the magnetically driven rail plasma accelerator is covered. This simple apparatus requires only modest vacuum yet is capable of producing some rather interesting effects. Subsequent articles will cover power supply design and refinements to the basic rail gun design. This project involves the use of high voltages and very high value currentpulses. Do not attempt any experimentation in this area unless you have an appropriate level of knowledge and experience.

I. INTRODUCTION

The basic principles behind magnetic plasma acceleration are fairly simple and may be explained without even referring to plasma physics. The acceleration mechanism has been understood since Ampére's time when it was shown that a magnetic force exists between two current carrying wires. One of Ampére's experiments involved a loop of wire floating on and bridging two parallel troughs of mercury (see Figure 1). He found that when a current was passed through the circuit, the bridge moved so that a larger area was contained within the circuit. What ultimately

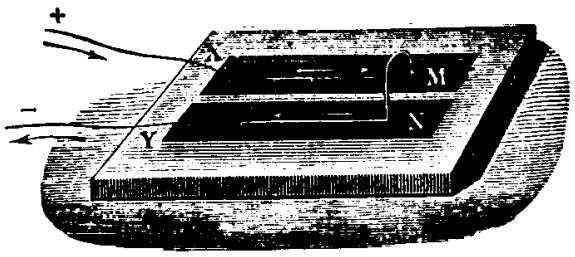


Figure 1 - Ampére's experiment showing mutual self-repulsion between elements of a circuit.

evolved was a mathematical understanding of the relationship between current flow in a circuit, the magnetic field (which arises from that source current), and the resultant force which acts on the circuit elements. This relationship is stated as the vector equation:

$$F = J \times B$$

where *F*, the force vector, is the vector product of *J*, the current vector, and *B*, the magnetic field vector. (For those of you who may be unfamiliar with vectors, they are simply values (like amps) with a direction assigned.) In the above relationship, it is not important from where the magnetic field arises: it could be from the current flow solely or there could be a supplemental field.

All of the above becomes a bit clearer if Ampére's device is redrawn as a pair of rails with a rolling bar setting across the rails as shown in Figure 2. When current flows through the circuit loop (think of it as a half turn solenoid) a magnetic field will be developed with a direction up from the page (remember the right hand rule; curl you fingers in the direction of the current and your thumb will point in the direction of the resultant magnetic field). Going back to the force relationship, what it says is that there will be a force produced from the interacting current and magnetic field vectors and that force will be in a direction at right angles to each of the other vectors. That ends up being toward the right. (If you still have your hand curled and your thumb sticking up, the force will be in the direction your index finger would point if extended.) Thus, given a smooth enough set of rails and adequate current, the bar will accelerate to the right.

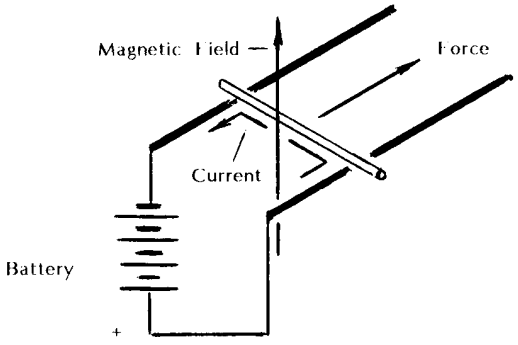


Figure 2 - Schematic representation of the rail gun principle.

four goal is to accelerate the sliding element to as high a velocity as possible, the mass of the moving rod (given practical limitations with regard to how much current may be dumped into the circuit) is a serious limitation. Now imagine that the rod is replaced by a highly conductive gas, a plasma (by definition, a

plasma is an electrically neutral collection of ionized atoms and electrons). For a given amount of applied force, the acceleration of a low mass moving element will be much higher than would be the case with the massive (relatively speaking) solid rod (we are simply trading mass (*m*) for acceleration (*a*) in the well known relationship $F = ma$). The current source also needs optimizing: a very fast dump of charge is needed. A battery or other steady state source would neither be adequate nor efficient.

An appropriate process for generating a plasma would be to discharge a high voltage capacitor bank through a very fine metallic wire element. Such a rapid discharge would result in the very rapid heating of the wire ending in vaporization and plasma formation. A bright flash and a sharp sound (when the discharge occurs in air) will ensue. This is the "exploding wire" phenomenon. This should not be confused with the melting and popping one would associate with, for ezample, a fuse blowing; the sonic shock resulting from an exploded wire is akin to a gun shot. To get an idea of the energy release involved, first consider that the energy stored by a capacitor is

$$E = \tfrac{1}{2}CV^2$$

where E is the energy in watt-seconds (or joules), C is the capacitance in farads, and V is the charging voltage. For a "home size" capacitor bank suitable for this sort of experiment typical values are 200 lf and 2000 volts. This would translate to 400 watt-seconds. Now, if the capacitor were to discharge in a period of 10 microseconds (not very fast by pulse standards) the *power impulse* in watts (assuming a nice rectangular pulse) would be E (400 watt-seconds) divided by the pulse length (10 lsec) or 40 megawatts. Current flow would be that power divided by the voltage or 20 kiloamps. (All of this is quite idealized; in reality the loop inductance and other characteristics will result in non-rectangular pulse shapes and a variety of losses.)

Returning to the magnetic rail gun, if the moving rod is replaced with the plasma source (the fine wire) and the battery is replaced by a capacitor, the discharge of the capacitor will explode the wire forming a conductive bridge between the rails. With current still flowing from the capacitor the $J \times B$ force will cause the metallic plasma to move toward the ends of the rails. If you've gotten the hang of the right hand rule, you'll also note that no matter which way the current flows the force will always be in the same direction. Thus a ringing capacitor will have no effect on the general direction of plasma motion. (From an efficiency point, however, it is most desirable to have a damped system which dumps all of the capacitor's energy into the plasma by the time the plasma has reached the rail tips.) Once the plasma reaches the end of the rails it will continue to move in the same general direction.

When the above takes place in a moderate vacuum (a few Torr or below), the plasma will be accelerated to quite high velocities, on the order of 10^6 to 10^7 cm/sec (higher at lower pressures). The very high plasma velocities attainable by such means led to considerable study of the rail gun during the late 1950s and 1960s as a method of propulsion for interplanetary space travel. Due to a variety of inefficiencies in the rail gun approach, this approach was pretty much abandoned by 1970 in favor of other configurations. However the rail gun is easy to fabricate and some interesting studies may be performed with it. An example would be the study of the impact patterns generated by the plasma with targets positioned at varying distances from the rail tips. The rail gun also provides a good entrée to several other types of pulse plasma source, many of which are within the capabilities of the home experimenter. and build upon the same basic equipment i.e. capacitor discharge power supplies and rough vacuum apparatus.

III. APPARATUS OVERVIEW

This section is only intended to provide a general overview of a rail gun system. Please note that the combination of high voltage and high capacitance make a deadly combination. The common connections must be connected to a good "earth", any control circuits which one might touch must be suitably isolated from the high voltage side of the supply, and a means of shorting the capacitor(s) terminals must be provided and must be in place when working on any part of the device.

The basic rail gun apparatus is shown in Figure 3. Within the bell jar is a pair of copper rails arranged as shown. One rail may be connected to the base plate as part of the system ground. The other rail is connected to a simple feedthrough which is fabricated from a KF (QF) centering ring and other hardware as shown. The wire is suspended between copper rails: The type of solid copper wire used for house ground connections is adequate for the rails. #24 to 26 copper wire is good for the plasma source. A low current (50 ma or so) high voltage power supply is used to charge the capacitor(s). Things will be simplified if voltages do not exceed about 2 or 3 kv. The capacitor bank (one or more caps in parallel) should have a value of at least 100 μf. The apparatus will work at a vacuum of several Torr however 100 mTorr to 1 Torr is a good operating range. Suitable oil dielectric capacitors may be found at

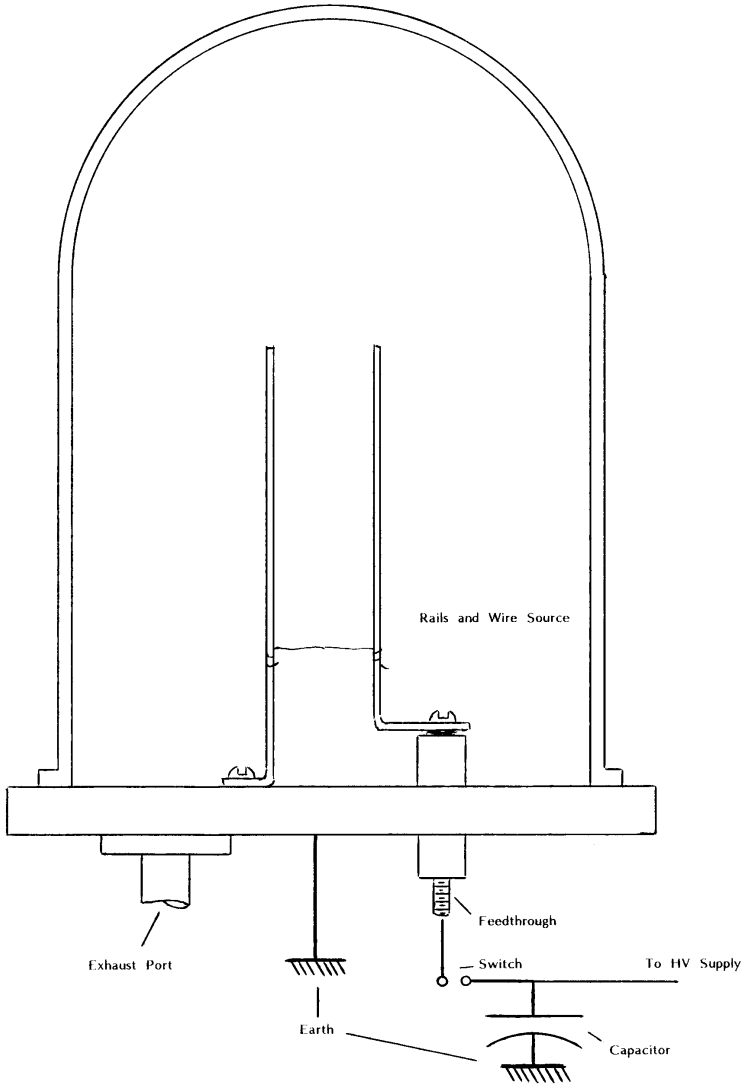


Figure 3 - Rail Gun

surplus outlets such as Fair Radio Sales and C&H Sales **Do not** use electrolytic caps as they are too inductive and may be destroyed by the polarity reversals of the discharge cycle. Once charged, a mechanical switch in the discharge circuit is closed; this will result in the high current pulse which will cause the wire element to explode. Thick/short connections are needed in the discharge circuit. I've found RG-8U coax to be adequate for most purposes. Targets (typically glass plates) may be fixtured above the gun's "muzzle."

IV. OPERATION & AN EXPERIMENT

After assembling the apparatus and with the terminals of the capacitor well shorted (a shorting bar may be made with a stiff piece of metal long enough to bridge the terminals affixed cross wise at the end of a long insulating handle) place a piece of thin wire across the rails at the indicated position indicated in Figure 3, i.e. just above the bend. For demonstration purposes a piece of window glass cut to a convenient size makes a good target. To examine the directional characteristics of the gun, place one target below the rails, another one in close proximity to the rail ends, and another positioned vertically and parallel to the rails. Provide some sort of support for the targets. Next, place the bell jar on the plate and exhaust the system with a mechanical pump. When a vacuum of at least one Torr has been reached remove the shorting strip and begin to charge the capacitor. When charged, discharge the capacitor through the rail gun (a simple switch is just another insulating rod with a shorting bar at the end which can be used to touch the "hot" rail connection to the capacitor terminal). It is recommended to wear hearing

protection while doing this - the discharge at the switch is noisey and will leave your ears ringing. If all has gone well the wire element will vaporize in a brilliant flash.

Release the vacuum and note the patterns on the various glass plates. If the plate at the end of the gun was very close to the rail tips you will note a distinct pattern near the rails themselves indicating a focussing effect. You will also note some deposits on the other plates indicating that, while the gun is quite directional, there are losses. Microscopic examination of the targets will reveal some very interesting features such as a variety of types of metallic deposit (films and imbedded particles), fracture patterns, etc.

The lossy nature of the rail gun is one reason for its limited usefulness as a means of propulsion. However, the approach has evolved over the years, first to configurations with parallel metal plates, and then to coaxial arrangements. The latter device in a "wireless" configuration (the discharge takes place across a gap with the eroded electrode material and gas within the gap serving as the source of ions) is now being investigated as a source for high intensity soft x-rays.

FURTHER READING

Good coverage of rail guns and similar devices may be found in Frank Früngel's *High Speed Pulse Technology, Volume 1* (Academic Press, New York, 1965).

This article was originally presented in Volume 1, Number 2.

A Coaxial Plasma Gun

Steve Hansen

Abstract: A simple magnetically driven plasma accelerator with a coaxial geometry is described. This gun may be used to generate beams of copper ions, as a shock tube, and to demonstrate the principle of plasma propulsion.

I. INTRODUCTION

Magnetically driven plasma accelerators were discussed in an article in the Spring 1992 issue of this journal. The example given was a simple rail gun using two parallel wires as the electrode rails and a thin piece of wire as the fusable element from which the plasma is formed. The Summer 1992 issue contained details on a high voltage capacitor discharge power supply suitable for powering small plasma guns of the rail type.

Disadvantages to the rail geometry as described include sizable side losses of the generated plasma (i.e. only a small portion of the generated plasma is projected along the rails) and the long time between subsequent "shots" as a result of having to break vacuum to change the fuse wire.

This article describes a coaxial configuration of the rail gun which provides for better capture and focusing of the plasma. Also incorporated is a spark gap which permits plasma generation without the wire fuse thereby permitting relatively short cycle times between shots without the need to open the chamber.

The entire accelerating portion of the gun is fabricated from copper. Thus, the beam is substantially composed of copper ions and neutrals.

II. CONFIGURATION

The coaxial configuration may be thought of as a simple extension of the rail gun where one of the rails becomes the central electrode and the other rail is rotated around the first rail to form the coaxial outer cylinder. The operating principle is the same. A current loop is formed between the electrodes (here the inner rail and the cylinder), some metal between the electrodes becomes intensely heated forming a plasma, and then, by virtue of the geometrical asymmetry coupled with the magnetic field developed by the current, the plasma travels down the rails.

A simple coaxial gun is shown in Figure 1. The outer electrode is a short length (8 inches to a foot is reasonable) of 1 inch copper water tube (1.125" od). The inner electrode is fabricated from 0.125" copper hobby tubing (e.g. K&S Metals). Alternatively a straightened piece of refrigeration capillary tube could be used. Do not use anything too thin as the localized heating will cause very rapid erosion.

Instead of the wire fuse used in the previously discussed rail gun, the plasma is generated in a spark gap which is located at the base of the accelerator tube. The gap is simply a "fat" region of the center electrode. Since the separation between electrodes is reduced at the

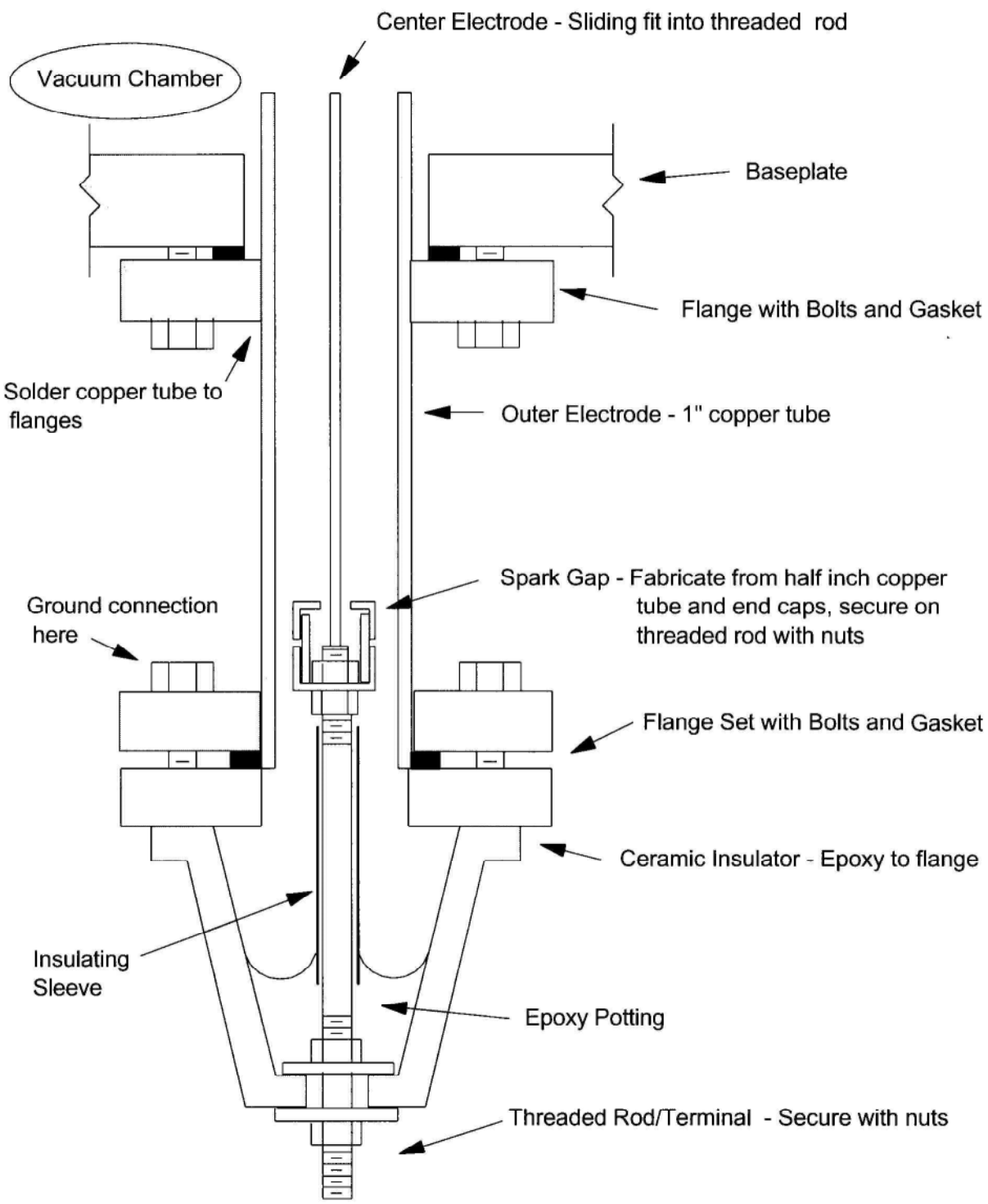


Figure 1 - Coaxial Plasma Gun

gap, the plasma generating spark will initiate here.

The components of the plasma will consist of ionized molecules and electrons, as well as neutral (uncharged) matter. The former will predominantly be composed of the electrode material, in this case copper. Keeping the electrode stucture to one material will result in a beam which is overwhelming comprised of that material.

The inner electrode of the gap is fabricated from two half inch copper end caps and a short length of half inch copper tubing (0.625" od). The lower end cap is drilled with a 0.250" hole. This is used with a pair of 1/4-20 nuts to secure the electrode to its support structure, a piece of threaded rod. The upper cap is drilled to clear the center electrode (0.250" is fine).

The structure below the gap is for current introduction and mechanical support. None of the parts here are involved in the plasma generating or accelerating process. The central electrode is supported by a length of quarter inch threaded rod (preferably brass). The upper end of this rod is drilled to hold the center electrode (a tight sliding fit). The lower end is held by an insulator. I used half of a conical feedthrough although other variants could be improvised. A vacuum tight seal is provided by potting this area with hardware store variety white epoxy. As some copper residue will collect in this area it is important to have enough insulation to prevent arcing between the rod and the outer electrode below the spark gap. I found that by adding an insulating sleeve with the lower end embedded in the potting epoxy this problem could be eliminated. The sleeve can be a ceramic or glass tube or just a piece of shrink tubing applied to the rod. The gun is divided into two sections, the upper accelerating structure and the lower area. For maintenance (cleaning and replacing electrode elements) the gun should be able to be dissassembled at the division between these regions. I've used the homemade stainless flanges as described in the Winter 1992 issue. A satisfactory commercial alternative would be the knife edge UHV type flange (e.g. 2.75" diameter x 1.125" bore). However, given the non-critical nature of the assembly, I could imagine several workable kluges based on old insulators, epoxy, and steel washers. Another flange is used to attach the gun to the vacuum chamber.

For a good electrical connection as well as for sealing, the outer electrode is soldered to the flanges. Low melting tin-silver is good for this. The ground connection from the capacitor bank is made at the base of the accererator. The hot connection is made to the central electrode at the bottom of the gun. The integrity of the ground is important. Make sure that there is a good connection (via the bolts) between the upper flange and the vacuum chamber base plate and that, in turn, the entire system is well grounded. As we are dealing with current transients on the order of thousands (up to tens of thousands) of amps, having a heavy wire leading to a dedicated ground rod is not overkill.

As the vacuum requirements are not particularly rigorous, the gaskets used with the flanges may be rubber or any of the cheaper synthetics, e.g. neoprene.

This gun is designed to be "fired" into a vacuum chamber. My chamber is a 9 inch diameter by 17 inch tall cylinder. The center of the gun muzzle is located off center, about 3 inches from the chamber wall, to provide room for target holders. The gun extends about 2 inches into the chamber. (The length of the gun as well as its protrusion into the chamber may be increased by simply adding extensions in the form of more 1" copper tube joined with a copper sleeve. There is no need to solder. A longer center electrode would have to be supplied as well.)

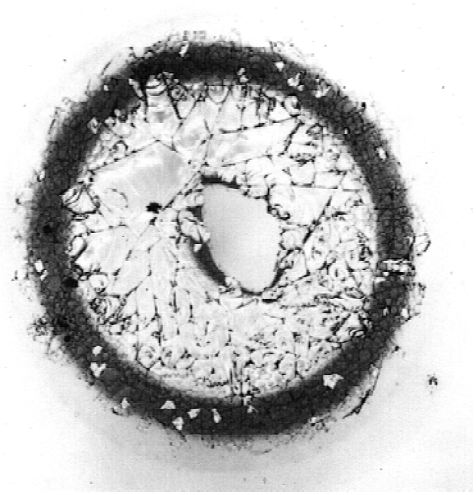
III. OPERATION

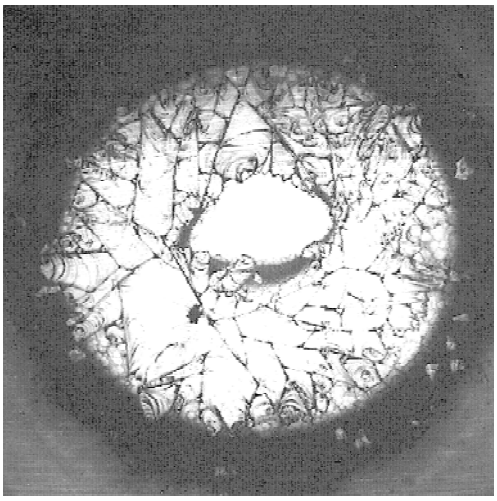
The gun works quite well with a capacitor bank as small as 100 μf at 2 to 3 kV. The desirable vacuum range is from about 1 Torr down to 100 mTorr (lower and the gap might not fire).

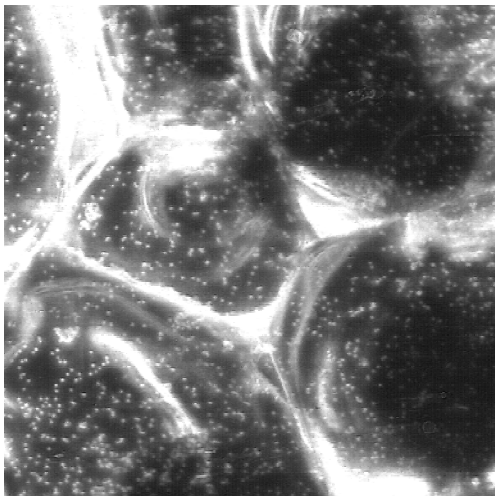
The center electrode may be biased positive or negative with respect to ground. As shown in the earlier article, current direction has no effect on the direction of the beam. However, there are some interesting differences in the focusing properties of the plasma depending upon bias.

When the gun is observed in a darkened room, a bright and slightly diverging beam will be seen to emerge from the muzzle. The beam, consisting of fast moving constituents, has an observable force. This may be demonstrated by placing a piece of stiff paper over the muzzle. When the gun is fired, the paper will fly away quite rapidly (remember, this is an airless environment - the motion will be quite dramatic). At high beam energies the paper target may even be crumpled or perforated by the force of the beam.

Inspection of the target will reveal traces of copper. This is easier to see if a piece of plate glass is used as the target (do rig some sort of support for this target). Another thing that will be noticed is the erosion of the center electrode. The entire length of the electrode will have a variety of lengthwise tracks but the tip will have a higher degree of erosion. This is due to the focusing action of the magnetic field as the plasma reaches the end of the gun. Much of the plasma will continue to move in the direction of the axis of the gun. However,







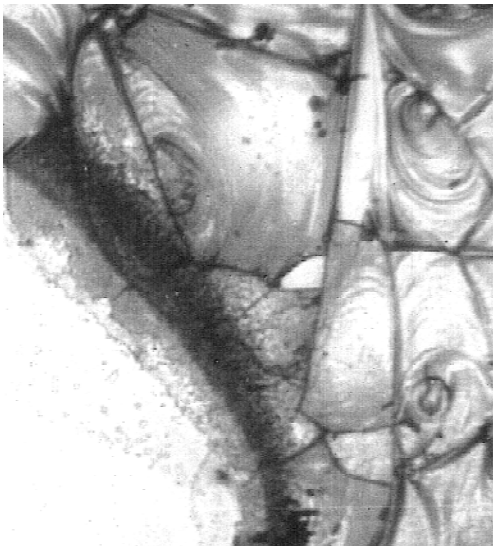


Figure 2 - Micrographs of Plasma Gun Target

Plate glass target was placed approximately 1 mm from the end of the plasma gun. All photographs are of the same target. One shot was fired using a 240 μF capacitor charged to 2 kV at a system pressure of 200 mTorr. All photographs are by the author.

Upper Left: Clear central area is the shadow of the 1/8" diameter center electrode. As the electrode was cut with a wirecutter, this area has an elliptical shape. Adjacent to the electrode shadow is a region of fractured glass. This fracturing is due to the shock wave and/or the intense heat caused by the pulse. The dark circular area is a copper rich region denoting the focusing effect of the plasma gun.

Upper Right: Same as the upper left photograph except with dark field illumination. The fracture region stands out in better detail.

Middle Left: Dark field closeup of the fracture region. Note the small dots. These are copper macroparticles that have been imbedded in the target.

Lower Left: Fracture region directly adjacent to the central electrode. Note the sharply defined copper-rich region at the perimeter of the electrode and the fracture and strain patterns in the fracture zone.

some portion will be accelerated radially inward toward the axis, at the tip of the electrode. This plasma focus effect has received quite a bit of attention amongst the plasma physics community, particularly in scaled up versions of this simple gun. One potential practical application of plasma focus devices is the generation of intense bursts of soft x-rays (here the tip of the central electrode would contain a tungsten insert) for fine feature semiconductor lithography.

The difference in beam pattern may be studied by placing glass targets a successively further distances from the muzzle of the gun. The patterns obtained in close proximity will show various well defined regions revealing different types of metallic deposit and damage patterns. Further away, the deposit appears more uniform, as one would expect from an accelerated ion beam.

III. EXPERIMENTS

Personally, I find experimenting with plasma guns to be extremely interesting. On top of that, the devices are very simple and very undemanding in terms of vacuum requirements (0.1 to 1 Torr is fine).

When a coaxial gun fires, several things happen and each one of these can be explored and exploited by the experimenter. For one thing, a hypersonic shock wave is created. This wave will carry along macroscopic particles of the copper electrode structure. As the velocities are in the same realm as micrometeorites, this type of gun has been used to study the effects of dust impact on spacecraft materials.

The gun will also project a puff of plasma. This can be shown by placing a magnet near the muzzle of the gun. In a darkened room the bending of the beam is quite visible. In fact, several beams can be seen as the copper ions will have varying degrees of ionization.

The region near the gun's muzzle is quite interesting as it is here that there is a pinch effect i.e. the plasma will be drawn toward a focus point about the axis of the gun. The pictures and text of Figure 2 show some characteristic patterns that were produced on a plate glass target placed at the gun's muzzle.

This article was originally presented in Volume 2, Number 1 with additional material from Volume 3, Number 3.

New Life for Old Microwave Ovens

Steve Hansen

Abstract: A variety of thoughts and information sources concerning how the components of scrapped microwave ovens may be used for interesting projects and experiments.

I. INTRODUCTION

Those readers who have read the first volume of this journal will be well acquainted with my adventures in the local dump (or, rather, "recycling center") scrounging such treasures as refrigeration compressors and dehumidifiers. About six months ago I turned my attention toward microwave ovens. I've averaged about one per every two or three weeks and have thus far accumulated the major components from upwards of ten ovens.

To give some context to this, I've long had an interest in high power microwaves. Before the age of consumer microwave ovens I had put together a magnetron pulse modulator (WWII variety) using surplus components but was foiled in my attempts to acquire a working tube still attached to its magnet (this was around the time when the mail order surplus houses (like Edmund Scientific in its earlier days) were making money by selling magnets for retrieval purposes). My zeal had somewhat subsided by the time the home "radar range" came along but I maintained a lingering interest as well as a feeling that something else besides cooking could be done with this new technology. Confirmation came when the ham radio publication *73 Amateur Radio* came up with an article on adapting an oven to amateur TV. Since then I've noted a variety of articles, especially in the professional literature (surprise!) regarding the use of oven components to construct experimental apparatus.

This article is not intended as a construction article but rather to provide some background information on microwave ovens and then to discuss the basics for conversion. Several sources of further information will be referenced for those who want to delve into this more deeply. This is not to preclude a future article or articles having to do with specific implementations - in fact, I am in the process of building a plasma reactor along the lines of what will be discussed here.

Hopefully this introductory article will provide enough information to generate some interest in such conversions amongst the amateur community. I will be most interested in reporting any results or good ideas in this journal.

II. MICROWAVE OVEN BASICS

Figure 1 depicts the basic organization of a microwave oven. The most important components are the magnetron power tube, the power supply for the magnetron, the cavity (i.e. the cooking chamber), the waveguide coupling between the magnetron and the cavity, and the control circuitry (timer, etc.).

The magnetron is a self contained oscillator which operates at 2.45 GHz (2450 MHz), a standard industrial frequency. Depending upon the size of the oven the associated tube may put out anywhere from a couple hundred to nearly a thousand watts. The tube is a current operated device and power output will vary directly with the amount of DC input power. Spectral purity and frequency will vary somewhat depending upon the amount of current fed into the magnetron by the power supply. David Pacholok in his article in *73 Amateur Radio* [1] noted a 1% change in frequency with a 2:1 change in current. While such characteristics would be undesirable for communications purposes, for cooking and plasma generation, these problems are not an issue.

The cavity is where the microwave energy ultimately gets absorbed by the food. The tube is connected to the cavity by means of a short length of rectangular waveguide (1.5 x 3 inches). Depending upon the design of the oven (i.e. where the microwaves enter the cavity) this waveguide may be only a couple inches in length or as long as a foot (the smaller ovens seem to be more generous in this regard). The magnetron has a probe which enters the waveguide at one end; the output to the cavity is a simple opening. The length of the waveguide, location of the tube probe and output window, and the size of the cavity are all calculated so as to transfer the maximum amount of power to the load (food) with a minimum of power being reflected back to the tube. If excess power is reflected, the tube will overheat. All ovens have a number of overtemp sensors which will shut off power should such a condition exist. One of the sensors will be found on the magnetron itself.

The power supply consists of a leakage (current limiting) transformer with two secondaries - a high current/low voltage (about 3 volts) filament winding and a high voltage (about 2 kV at up to 250 mA) winding. High voltage line frequency DC pulses are provided to the magnetron by means of a simple capacitor-diode circuit. These pulses are superimposed on the filament current; the magnetron anode (case) is at DC ground. In newer ovens the diode and capacitor are integrated into one unit - older ovens will have discrete components.

Other essential components to be found in an oven will include the fan (which keeps the tube cool and also ventilates the cavity), the aforementioned thermal cutouts, fuses, and surge protectors. The control circuitry may be as simple as a spring wound timer but today's norm (except in the cheapest ovens) is a microprocessor based controller.

Not to be overlooked are the interlocks and shielding. Microwave radiation is dangerous. (This reminds me of a summer job experience I had at a company that manufactured microwave antennas: one of the technicians (an experienced one) was quite impressed at how the microwaves coming from a feed could warm his hand!) When working on or attempting any modifications to an oven, the integrity of the shielding system must be kept intact. Fortunately, leakage testers are cheap and easy to come by. Surplus ones may currently be had from American Science & Surplus for under $10.

III. PARTING OUT AN OVEN

Microwave ovens all look very much alike on the inside. Unlike many consumer products they are easy to take apart and I've yet to find one that did not have the schematic glued to the inside of the case. Opening is done by removing the several screws around the cabinet. Just about all of the disassembly may be performed with a set of screwdrivers, a small adjustable wrench, and some wire cutters. You will want to take out the following items: the magnetron (and its associated thermal cutout switch and mesh gasket), power transformer, diode/capacitor assembly, fan (and its associated ducting, if any), and primary circuit protection components (fuse, varistor). Also save the schematic and the various slip on connectors. If the oven has a waveguide of any substantial length, try to save it as well. One wall of the guide is usually part of the cavity and removal will require some cutting of the sheet metal. Saving the control board is optional; you might be able to think of something creative to do with it in another application.

If you obtained the oven from a scrap heap, one thing to think about is why the oven was discarded. There are three primary reasons that I can think of: the magnetron failed (most likely reason for concern), the control board failed (not an issue here), or the previous owner decided it was time to upgrade to something newer, bigger, more stylish. Since many of the parts

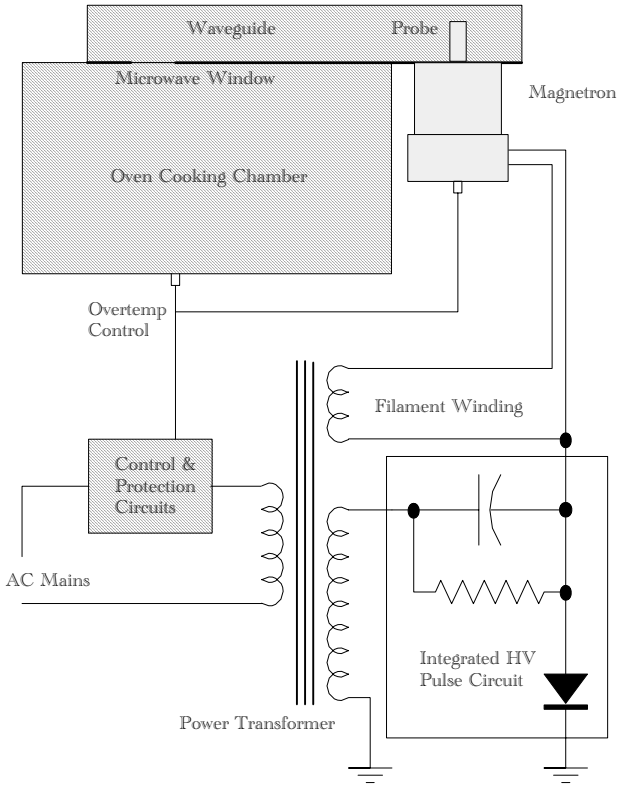


Figure 1 - Microwave Oven Organization

that we are interested in are largely interchangeable, acquiring several scrapped ovens will almost certainly yield enough working parts to build up a working microwave power supply including a goodly set of spares.

After parting out your find, return the carcass to the dump.

IV. SOME CONVERSION IDEAS

What follows is some information gleaned from two particularly relevent articles which discuss microwave oven conversions. One is from a hobby publication, the other is from the professional literature.

ATV Transmitter from a Microwave Oven! This article [1] deals with the adaptation of a microwave oven to amateur TV. While most of the text is concerned with the design and construction of a modulator, the author does describe a simple set of modifications to couple the oven magnetron and waveguide to coax cable by means of an adjustable E-field probe. The probe components are standard radio and hardware store items.

Simple Low-Cost Microwave Plasma Source. The "standard" way to couple a magnetron to a cavity is through conventional microwave “plumbing.” The plumbing, at the minimum, would consist of such things as isolators, circulators, dummy loads, forward and reflected power meters, and so on. Not simple, easy to acquire, or cheap. This paper [2] describes a simple tuned cylindrical cavity which has the magnetron tube mounted directly on the sidewall with the probe extending into the cavity. Tuning is accomplished by sliding brass end plates, one at each end of the cylinder. Power is adjustable by inserting a variable autotransformer (120 volt, 10 amp) in the primary circuit. Input power to the tube is monitored by means of a wattmeter placed before the autotransformer. (Conversely, a voltmeter/milliamp meter pair could be placed in the secondary circuit of the high voltage transformer.) Since the filament winding is incorporated in the power transformer, a separate transformer is introduced for the filament. This is just a second oven transformer. The “load” in the particular application is the gas flowing in a quartz tube (other materials are too lossy) which passes lengthwise through the center of the cavity. The authors note that,

in order to limit the cavity to only one resonance mode, the diameter is set so that only a single half wavelength could be accomodated between the plasma boundary (i.e. the inside of the quartz tube) and the cavity wall. At 2.45 GHz, a half wavelength is about 6.1 cm. The described cavity with a tube inside radius of 0.9 cm and cavity radius of 10 cm meets this requirement. The final settings (with a well struck plasma discharge at 1 Torr pressure and cool operation of the magnetron) were with the magnetron probe 2.5 cm from the downstream end plate and about 7 cm between plates.

While not explicitely stated in the article, reflected power may be indirectly monitored by means of a temperature sensor on the magnetron (something like Radio Shack's digital temperature probe would do).

V. SOME APPLICATIONS IN PLASMA CHEMISTRY

All of the above is quite nice but what does one do with a few hundred watts of microwave power? One area of potential interest to the amateur would be to generate plasmas for use in facilitating otherwise difficult or impossible chemical reactions. Plasma chemistry has become a major area of research and numerous applications now exist within industry. Plasmas (i.e. an electrically neutral collection of partially ionized gas comprised of ions, electrons, and neutrals) may be created through the action of applying energy in the form of very high temperatures or intense electric or magnetic fields to the gas medium. In plasma chemistry the driving force is most frequently an electrical discharge. The current may be directly or indirectly coupled to the gas. In the case of the former, a DC or low frequency AC current may provide the excitation by means of electrodes placed within the chamber. With the latter, the energy is coupled either inductively or capacitively through the containing vessel's walls and the exciting field is in the form of RF (e..g. at the industrial frequency of 13.56 MHz) or microwave radiation. Since this article is focused on microwave applications, we will limit the discussion to that mechanism.

Alexis T. Bell [3] notes that “in a discharge, free electrons gain energy from an imposed electric field and lose this energy through collisions with neutral gas molecules. The transfer of energy to the molecules leads to the formation of a variety of newspecies including metastables, atoms, free radicals, and ions. These products are all active chemically and thus can serve as precursors to the formation of new stable compounds.” In everyday life, plasma-assisted reactions are used in the manufacture of semiconductor devices (in many cases eliminating the expensive and environmentally hazardous wet processes), to harden the surfaces of metals, produce osmotic membranes, improve the adhesion characteristics of polyester tire cord, deposit films of synthetic diamond, and produce new polymers.

A basic reactor facility is shown in Figure 2. This example is based on the simple microwave source described in [2]. This is called a "downstream" or “remote” reactor configuration: a carrier gas is passed through the plasma region within the cavity and then the reacting species are introduced outside the region downstream from the excitation area. The reaction is induced by the active species that emerge from the discharge region. This configuration permits the reaction to proceed in a stable environment and also keeps the reaction products from contaminating the chamber walls in the discharge zone.

A couple of interesting articles which describe research into the upper atmosphere reactions which cause the airglow appeared in the March 1966 and January 1972 issues of Scientific American [4,5]. The airglow is one of the natural sources of light in the sky and is caused when atoms and molecules are dissociated by the action of the sun's UV radiation which then subsequently recombine in various ways. One of the articles [4] describes a laboratory experiment using a microwave energized downstream plasma reactor. The carrier gas is common molecular nitrogen (N_2) which gets dissociated into atomic nitrogen (N) in the plasma discharge region. The gas admitted downstream is nitric oxide (NO) and the reaction products are atomic oxygen (O) and molecular nitrogen (N_2). The excited oxygen atoms de-excite through collisions and by the emission of light. The spectral lines and bands emitted during these reactions require unique conditions and are seldom seen in the laboratory as they arise from rarely occuring changes within the structures of the atoms and molecules (these are so-called “forbidden emissions”).

Getting down to earth, there is a considerable level of research into plasma assisted chemical vapor deposition (PACVD). Here, reaction products are formed which condense on a substrate. An industry which makes wide use of PACVD is semiconductor manufacturing where well characterized high purity thin films are of great importance. Another application is for the formation of diamond films. [6] describes a relatively simple reactor for the PACVD of diamond. The described reactor also uses adapted microwave oven parts along with a surplus pyramidal cavity which covers the reaction chamber, a small quartz bell jar. The reaction gases are methane and hydrogen which are maintained in the chamber at a pressure of between 60 and 140 Torr. The plasma keeps the temperature of

the substrate (a silicon wafer) at a temperature of 800 to 900 degrees C. The article goes into some detail on the safety features incorporated in the reactor design due to the gases being handled.

From an amateur's perspective, a simple plasma reactor could be used to demonstrate a variety of simple reactions involving ashing, surface modification, or film deposition. Two excellent references on plasma chemistry are the books by Boenig [7] and Hollahan and Bell [3].

REFERENCES

[1] David Pacholok, 73 Amateur Radio, July, 1989.

[2] L. G. Meiners and D. B. Alford, Review of Scientific Instruments **57(2)**, 164 (1986).

[3] John Hollahan and Alexis Bell *Techniques and Applications of Plasma Chemistry* (Wiley-Interscience, New York, 1974).

[4] Robert A. Young, Scientific American, March, 1966.

[5] M. F. Ingham, Scientific American, January, 1972.

[6] M. A. Brewer, I. G. Brown, M. R. Dickenson, J. E. Galvin, R. A. MacGill, and M. C. Salvidori, Review of Scientific Instruments **63**, 3389 (1992).

[7] Herman Boenig, *Plasma Science and Technology* (Cornell University Press, Ithaca, 1982).

This article was originally presented in Volume 2, Number 1.

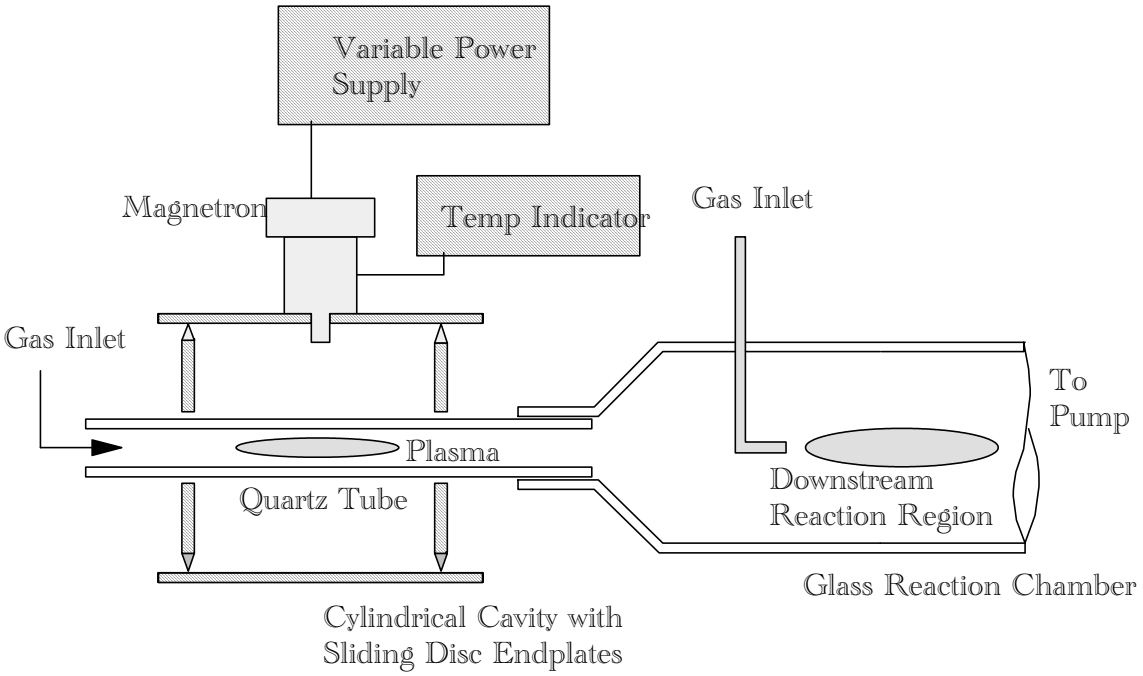


Figure 2 - A Simple Microwave Plasma Reactor

Microwave Miscellany

A variety of additional thoughts concerning recycled microwave ovens

The article on the use of microwave oven components to make such things as plasma reactors must have made an impression based upon the amount of mail which your editor has received during the intervening three months. What follows is a selection of the various pertinent comments and an update from yours truly on another application.

Bill Fain of Martinsville, VA writes "I read with great interest your article on the microwave oven exciter. I actually witnessed FM transmission through a microwave while attending an ATV (amateur television) meeting which was held in conjunction with the Dayton Hamfest (which is a must for any hands on experimenter). I believe it was David Pacholok that demonstrated the device (he also had an article about it in a microwave industry magazine (*this is pretty much the same article which appeared in* 73 Amateur Radio-Ed.). I can remember his telling about how it worked and then the moment of truth when he turned it on. He had a piece of teflon coax as the dummy load as it is not a good conductor of microwaves at that frequency. I remember looking around the room for a quick way out if the expensive microwave leakage tester (Radio Shack) he used had shown in the red zone. I also covered my eyes and other things near and dear to me since they did not have any heat distinguishing capability. Well anyway, he turned it on and there was a great picture on the receiver across the room of the color bars he was transmitting. After a while the coax started smoking because it didn't like 600 watts of rf going through it. He then shut it off. Before seeing this demonstration, I had thought about using a microwave oven and my satellite dish to try to communicate to someone on the other side of the world using moonbounce. I haven't calculated the free path losses and all that stuff yet so I don't know if it's actually possible. I have noticed that research scientists have been able to achieve 55 terawatts of laser power using wave stretching and compression techniques. Maybe this could be done with the microwave oven to achieve greater power."

Bill concludes by noting that he now has a microwave oven squirreled away in his garage - safe from cooking duties.

Bill Williams of Boulder, CO, has pointed out the book *Microwave Oven Service and Repair* by Clayton L. Hallmark (Tab Books No. 962). Some useful items in the book include information on detecting failure modes in microwave oven tubes:

- Lost vacuum - detectable by oxidation of the internal copper parts of the tube.
- Tipoff cracks - axial or transverse cracks in the sealoff area due to mechanical or thermal stress. Will cause lost vacuum.
- Suck in - caused by overheating of the glass around the output (antenna) glass to metal seal. Will cause lost vacuum.
- Insulation breakdown - burns around the heater leads from arcing or shorting. Caused by the ferrite beads on the leads being too close to the glass or from contamination on the surface.
- Open heater - broken due to shock or vibration. Normal heater resistance should be 1 ohm or less.
- Cathode exhaustion - low power or slow to reach normal power; current too low for oscillation at the normal voltage. Sometimes the exhausted cathode layer can be burned off by briefly overheating the heater. Increasing the tube voltage may help.

Bill continues "Some other things of interest from the book include the presence of a 10 ohm 2 watt resistor to ground in the high voltage power supply. This has a jumper bypass which is removed to measure the plate current and hence the microwave power. Also, in many of the units the high voltage transformer and capacitor have selectable connections to adjust the voltage. Finally, the schematics in the book show several methods of power reduction as "defrost" circuits. Most common is a cycling relay or triac, an approach not too useful for our purposes. However, one circuit adds a 1200 ohm resistor and bypass switch from the diode to the ground side of the high voltage transformer. There are other variations of the theme using RC networks. It would seem that a 2 kΩ variable resistor would see less than 600 volts and allow a wide range of adjustment. One last method of power control consists of a coil around the magnetron magnet which allows the field to be strengthened thereby limiting the current passing through the tube. These kinds of tests should precede disassembly and should be run under a load of a quart of water. Overall, EXTREME CAUTION is indicated when experimenting with these

units as they have a wide selection of potentially fatal hazards."

Jeff Morris of Orlando, FL passed along a description of a commercial plasma etch system which is based on a standard microwave oven. The unit, called *Plasma-Preen* by its manufacturer, Plasmatic Systems, Inc. of North Brunswick, NJ has a rectangular pyrex chamber within the oven cavity (it looks roughly like a pyrex baking dish and is 8 x 6 x 2 inches) with the gas feedthroughs entering the chamber from the floor of the oven compartment (i.e. the "baking dish" is open end down). The floor of this chamber is water cooled via an installed aluminum cold plate. Power is adjustable from 10% to full (about 700 watts). Gases used are oxygen or argon with the flow controlled by means of a standard ball-in-tube flowmeter rated at 5 cu-ft/hour maximum. Stated applications include cleaning surface contamination from semiconductor devices, removal of plastic encapsulants, surface preparation of plastics for gluing, cleaning optics and other glass parts, and reduction of some metal oxides. Operating pressure is typically 1 to 10 Torr. What do you pay for the customization and the baking dish? About $6000. I would think that such a device would be well within an amateur's capabilities and could be quite handy for such purposes as preparing samples for microscopic examination (e.g. you can remove organic tissue from the mineral rich skeletal matter in both plant and insect samples, etc.) and for general purpose high quality cleaning. It would be important to monitor the tube temperature for excessive reflected power and the waveguide may need some adjusting.

Finally, a recent article in *Review of Scientific Instruments* ("An inexpensive x-ray source based on an electron cyclotron", H. R. Garner, T. Ohkawa, A. M. Howald, A. W. Leonard, L. S. Peranich, and J. R. D'Aoust, February 1990) shows how a microwave oven magnetron running at about 100 watts can produce electric fields within a small cylindrical cavity which in turn accelerate electrons (produced by a filament) to an energy of about 150 keV. The accelerator differs from a traditional cyclotron in that the "Dees" are replaced with the tuned microwave cavity. The cavity itself is 7.2 cm high and 12.7 cm in diameter. The filament is located centrally with the microwave feed (a loop) and the target (molybdenum rod) entering from opposite sides of the cavity. The magnetic field required for operation is moderate, about 1000 gauss. In the version described, the field is produced by an electromagnet. As the maximum orbital radius of the electrons is only about 1 cm, it may be possible to shrink the size of the cavity, thereby reducing the bulk of the magnet.

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Apparatus Splits Glass Tubes Longitudinally

From NASA Tech Briefs
January, 1993
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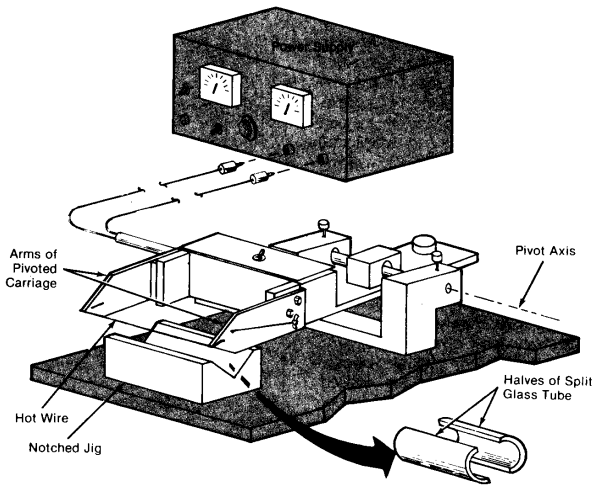
The apparatus shown in the figure splits glass tubes in half longitudinally by the well known hot-wire/thermal-shock method. A tube to be cut is placed on a notched jig in the apparatus.

A nichrome wire is stretched between the arms of a pivoted carriage and oriented parallel to the notch. The wire is heated by electrical current while it rests on the tube. After heating for about 1 minute for each millimeter of thickness of glass, the tube is quenched in water and is split by the resulting thermal shock.

The apparatus has been used to split tubes in sizes ranging from 9.5 mm in diameter by 25.4 mm long to 38.1 mm diameter by 102 mm long.

This work was done by Ernest Shaw and Robert O'Neill Manahan of Lockheed Space Operations Co. for Kennedy Space Center. For further information contact NASA CASI, Manager, Technology Transfer Office, PO Box 8757, Baltimore, MD 21240-9985. Refer to KSC-11547.



Some Resources and Ideas for Plasma Experiments

Plasma Experiments with Commercial Gas Tubes and More Ideas for Microwave Oven Conversions

I. INTRODUCTION

Over the past few months I have received a considerable amount of material from Prof. Robert Jones of the Department of Physics at Emporia State University in Emporia, KS. Prof. Jones' interests lie primarily with experimental plasma physics and he has constructed an interesting array of simple benchtop apparatus for plasma studies.

II. PLASMA EXPERIMENTS WITH GAS TUBES

Prof. Jones brought to my attention a number of articles that have appeared in the *American Journal of Physics*, a publication of the American Association of Physics Teachers. Each of these articles deals with experiments that may be performed with commercial gas tubes such as the OA4-G (argon-filled cold cathode triode), 884/885 (argon-filled thermionic triode), and 886 (mercury-vapor rectifier). All of these tubes (or equivalents) may be obtained for prices in the $5 to $12 range from suppliers such as Fair Radio Sales.

The use of commercial tubes permits a considerable amount of experimentation without the need for vacuum apparatus. However, the techniques, once understood, are completely applicable to "real" applications.

In this note I won't go into the details of the experiments but will only outline the experiments that are described. Detailed explanations of the concepts may be found in almost any text on plasma physics. Since most of these books are almost totally incomprehensible to the average mortal, a suggestion will be made at the end of the article.

The first article is ***New Elementary Experiments in Plasma Physics*** (I. Alexeff, J.T. Pytlinski and N.L. Oleson, September 1977). Four experiments are described:

- Plasma Familiarization - Measurement of *e/m* (charge to mass ratio of the electron) and the ionization potential of argon using the 884.
- Plasma Diagnostic Experiment - Measurement of plasma electron temperature and electron density by a single Langmuir probe using the OA4-G.
- Observation of the plasma electron frequency using the 866-A.
- Investigation of the decaying plasma using the 866-A.

The second experiment, as adapted by Jones, is diagrammed in Figure 1. I note this experiment because of the importance of the Langmuir probe in plasma diagnostics.

A Langmuir probe is nothing more than a wire that is inserted into a plasma to measure its potential. Early experimenters let the probe float and measured the voltage with a high impedance meter. That gave totally erroneous measurements because the floating probe would permit charges to accumulate. Langmuir's technique involved connecting the probe to a source of variable potential. The probe voltage is swept and the resulting current *vs.* voltage characteristic, will yield the electron and ion currents to the probe.

The figure shows how the OA4-G is connected for this experiment. A discharge is triggered between the cylindrical cathode at pin 2 and a ring shaped anode at pin 7. The electrode at pin 5 serves as the probe. This electrode is surrounded by a glass sleeve to a point at the plane of the ring anode. The unsheathed portion extends about 6 mm beyond the sleeve.

In the experiment, a discharge is struck between the anode and cathode. This may require about 200 volts. Once the discharge is started the voltage must be reduced to about 60 volts to avoid damaging the tube. After a period of warm-up, the probe is swept by incrementally varying the variable supply. A curve of the type shown in the figure will be developed.

As many plasma devices utilize magnetic confinement fields, a couple of articles describe experiments in which the OA4-G is immersed in a field. Now, all OA4-A tubes are not created equally. The above described tube with its long iron-alloy cathode is not appropriate for experiments with magnetic fields as the cathode quite effectively shields the discharge. However, there is a variation with a very short cathode in which the anode and probe structures are above the cathode, exposed. As the tube number is the same, you will have to do a bit of digging to find the right tube.

Experiments in a solenoidal field are described in ***Behavior of a Single Langmuir Probe in a Magnetic***

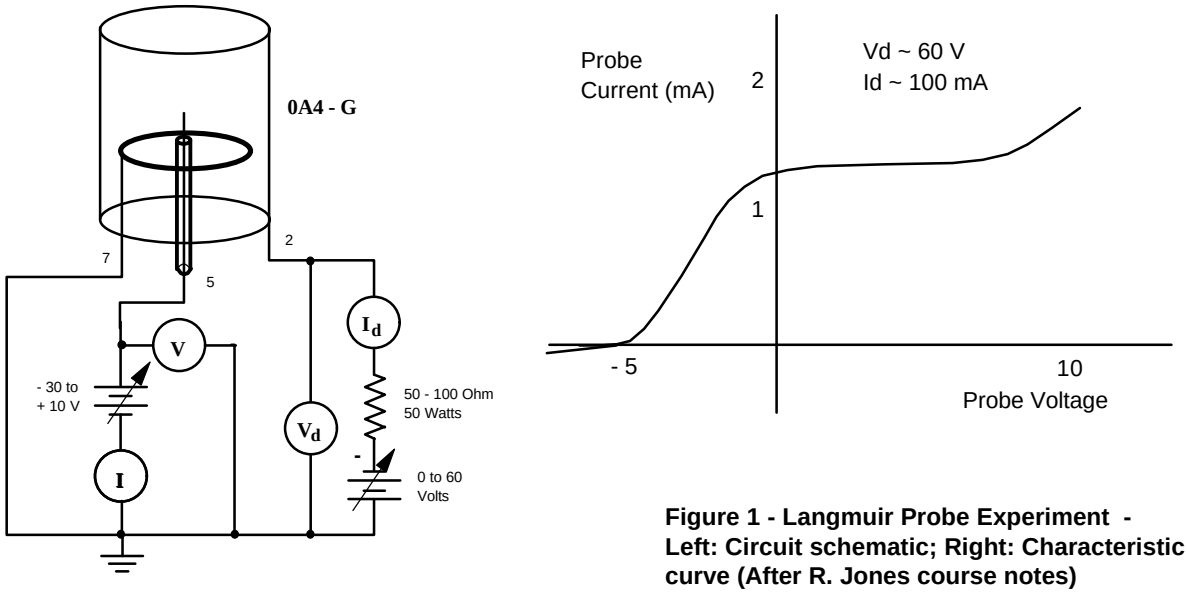


Figure 1 - Langmuir Probe Experiment - Left: Circuit schematic; Right: Characteristic curve (After R. Jones course notes)

Field (J.T. Pytlinski, H.J. Donnert and I. Alexeff, December 1978).

Experiments in more complex magnetic fields are detailed in ***Characteristics of a Langmuir Probe in a Magnetic Field*** (Jonathan Katz, Edward F. Gabl, Eugene K. Tsikis and Karl E. Lonngren, August 1984). Here, multi-dipole magnetic fields as might be encountered in plasma apparatus such as fusion reactors and ion sources are simulated by surrounding the tube with up to 16 small disk magnets that are attached to the inside of a steel coffee can, 3 lb. size.

Some more complex experiments using the OA4-G are contained in ***Some Plasma Physics Experiments on Electrical Conductivity and Similarity Laws*** (J.T. Pytlinski and I. Alexeff, December 1977). Let's just say that if you have the courage to try some of the above experiments, you'll probably like these too.

Seriously, the experiments detailed in the first noted article are easy to set up and conduct and any amateur seriously interested in plasma studies will get a lot out of them. I have obtained a small supply of gas tubes and will be trying some of these exercises in the near future.

III. MICROWAVE OVEN BASED PLASMA SOURCES

The article on page 3-51 addressed in general terms the use of microwave oven components for plasma experimentation. Since that time I have been puttering on a microwave plasma reactor. The status of this will be covered later in this article. However, Dr. Jones has had a considerable amount of experience with simple plasma sources based on oven components and several of these will be detailed.

Jones notes "Microwave plasmas are used as ion sources, for plasma chemistry, in ion implantation, isotope separation and in spectroscopy. 120 watt commercial units are available and sell for about $4000." Unable to afford such a unit, Jones pursued several alternatives.

Referring to Figure 2A, Jones states "In a typical microwave oven the magnetron tube is connected to the oven cavity via a waveguide formed out of folded sheet metal. In my microwave plasma source the 2M172J magnetron and power supply are removed from the oven along with a section of waveguide. The sheet metal section of waveguide (to which the 2M172J mounts) is cut from the oven proper and the open end of the guide is sealed closed with a folded sheet metal cap. This forms a microwave cavity 9 cm wide, 9 cm long and 3 cm thick. A hole is made in the cavity using a chassis punch. This hole enters the cavity from the side opposite to the face holding the magnetron. The hole is sized to the diameter of the Pyrex tube used as the discharge chamber. A 1" tube was used in the prototype and a standard lipless test tube may be used. A 1-1/2" tube would have the advantage of being able to mate with a 1-1/2" sink trap fitting. The tube is supported so that it is parallel to the magnetron's probe.

"An alternative source was made by removing the magnetron entirely and attaching it with epoxy cement to a 9 cm long section of 5 cm diameter copper tubing,

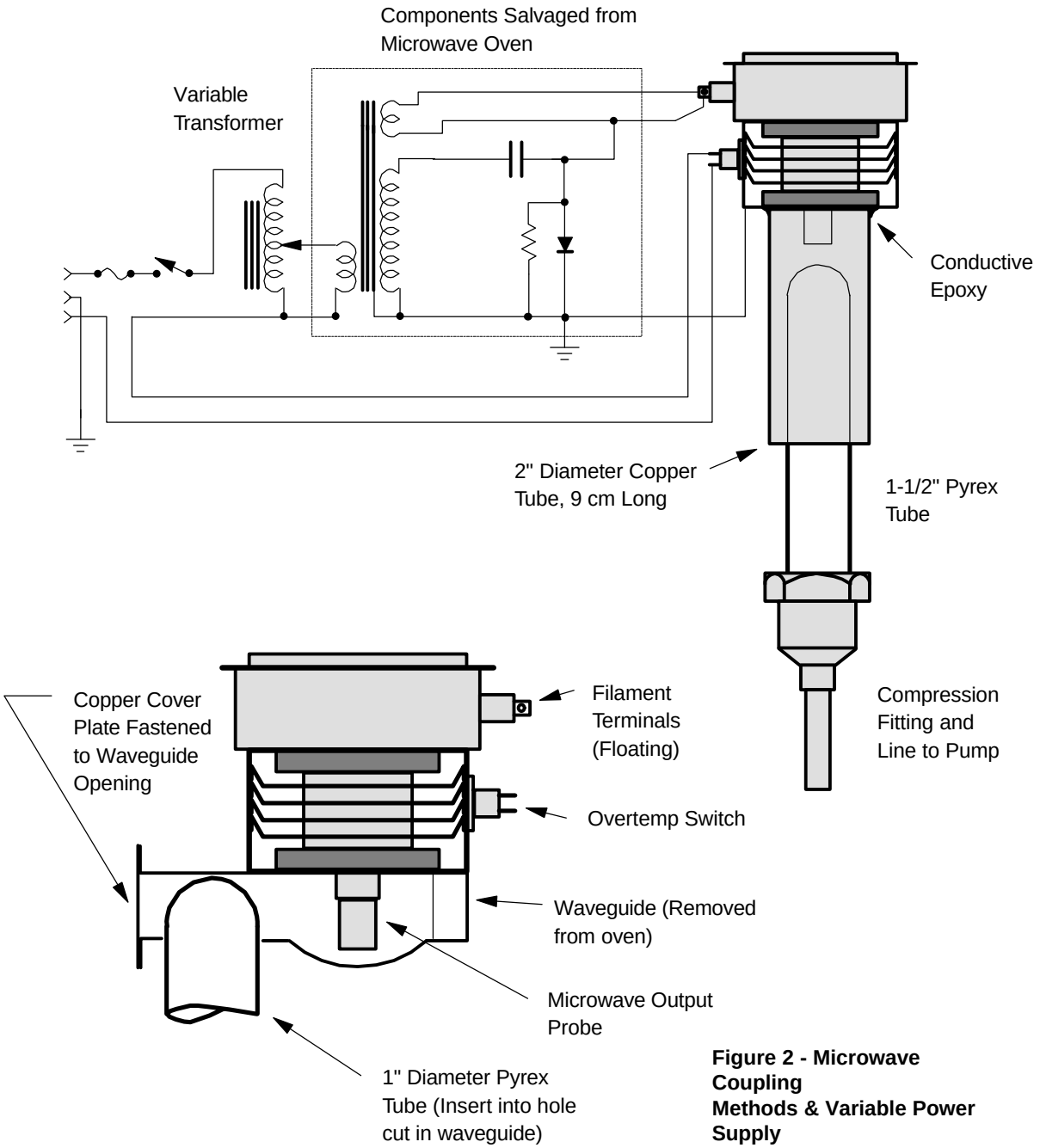


Figure 2 - Microwave Coupling Methods & Variable Power Supply

2A (Left) - Coupler Adapted from Oven Waveguide

2B (Above) - Coupler Adapted from Copper Tubing; Tube

coaxial with the magnetron's probe. (See Figure 2B.) The adhesive is preferably electrically conductive. However, it will also work with a poor electrical contact. The glass vacuum tube then slips down axially into this coaxial cavity."

Figure 2B also shows Jones' simple power supply. A variable transformer is used to control the power to the tube. Normally the filament voltage is held constant but this would require another transformer. Throttling both the high voltage and the filament permits the power to be cut back to just a few watts. The tube, at full power, will put out over 600 watts. Striking a plasma requires much less power. Scaling back the power also reduces tube heating. Nonetheless, the overtemp protector should be left in the circuit. The cooling fan may be eliminated for low power, intermittent operation.

Depending upon the particular oven you pull apart, the circuit and components might differ somewhat. For example, in some ovens the resistor is paralleled with the capacitor and is in the same can. Fortunately, ovens generally have a schematic pasted to the inside of the cabinet.

If you wanted to eliminate all of the "plumbing", it is possible to make a plasma in a glass chamber simply by bringing the probe of a bare magnetron up to it. This is rather inefficient and there is more microwave leakage. Regarding this, Jones continues "I do worry about microwave leakage, particularly at high power. The cheap *Rapitest* tester is a simple tool that can detect problems. But, even with the bare probe and will low powers, the microwave level can be kept to safe levels a meter or two behind the magnetron."

The vacuum requirements for this sort of device are modest. Plasma reactors typically operate at a few Torr. Rotary refrigeration compressors will work nicely. Jones has found that even a cheap metal water aspirator will work. This allows one to make a very cheap device.

Furthermore, even if you lack any sort of vacuum pump, you can still do some interesting plasma experiments by exciting the gas within the aforementioned OA4-G tube as shown in Figure 3.

Occasionally the discharge needs some help to get started. This can be done by "tickling" the discharge tube with a hand-held Tesla coil of the sort used for vacuum leak testing or with a high frequency TV flyback supply of the type shown on page 18 of this issue.

Jones concludes by saying "The rectangular cavity source is more efficient than the coaxial cavity source. With each of my microwave plasma sources there are component parts which could be optimized. For instance: What tube diameter is best? What length? And so forth. One could use a photo light meter (or a Langmuir probe) to judge which components give the most plasma for a given energy input. For the most part if something worked reasonably well I did not seek to optimize it. I just used it."

IV. THE EDITOR'S REACTOR

One of Jones' plasma sources used the entire oven. He bought a Sears and Roebuck *Capri* oven (about $80) and punched a 1-1/8" diameter hole through the top of the oven cavity, 2-1/2 inches left of center. The plasma

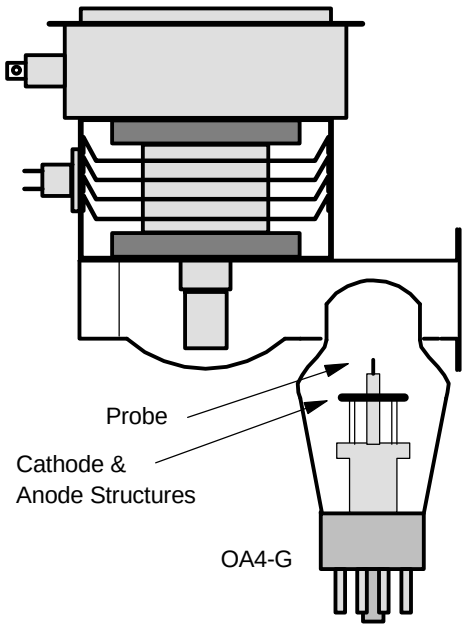


Figure 3 - Langmuir Probe Experiments in a Microwave Plasma Using an OA4-G Argon Gas Tube

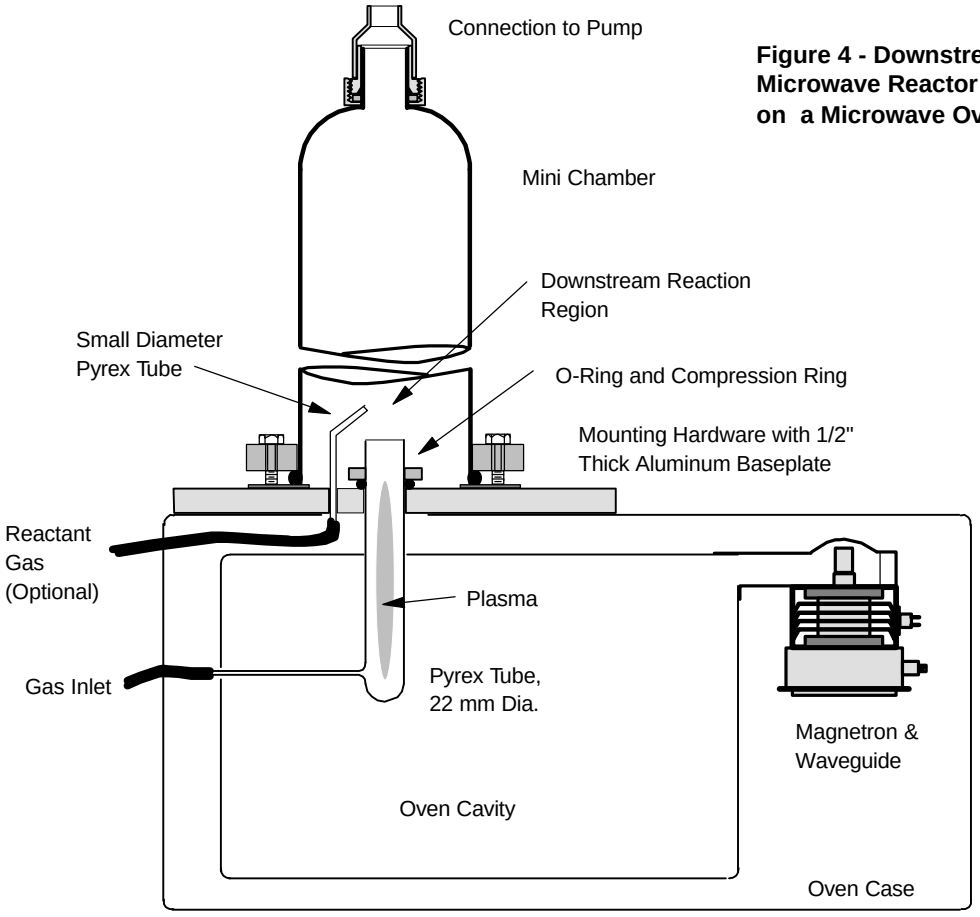


Figure 4 - Downstream Microwave Reactor Based on a Microwave Oven

was generated in a test tube inserted a few inches into the oven cavity. The plasma streams up the tube and out of the oven to a region where magnetic fields can be applied and experiments performed. In this version, power is controlled in the same way as noted above.

My system is being built along a similar fashion. Figure 4 shows the general layout. My oven, Goldstar brand, was one of several obtained from the appliance pile at our dump, a.k.a. recycling center. The reactor is constructed in a downstream configuration, i.e. the carrier gas enters the plasma tube at the end away from the reaction chamber. The main chamber is made from a VK-005 Mini Chamber which is pumped from the top.

A second gas inlet is provided above the plasma tube. Through this tube may be passed a reacting species. Reactions are induced by the active species coming from the plasma tube. These may include atoms, metastables, free radicals or ions. This configuration permits the reaction to proceed in a stable environment, i.e. away from the discharge, and also keeps the reaction products from contaminating the chamber walls in the discharge region.

Gas flow through each inlet is controlled by stainless steel needle valves. I obtained the valves from American Science and Surplus.

Since I obtained several ovens of similar size and since the major components are all interchangeable I have opted to add a second transformer to keep the filament voltage constant.

This added transformer also serves as the power control. I removed the high voltage secondary, a task that left a pile of fine copper wire all over the driveway. I then carefully pried every other primary turn away from the adjacent turns just enough to allow me to scrape away a bit of the enamel insulation and solder a piece of #18 wire to each of the selected turns. After soldering, insulating varnish was applied and allowed to dry. Pieces of fabric were then inserted before the displaced windings were returned to their original positions. Then another coating of varnish was applied.

The end leads and the five taps are brought out to banana jacks. Since the taps are non-symmetric, depending upon which way the transformer is connected to the mains, outputs of 12, 25, 32, 40, 52, 60, 68, 76, 88 and 100% may be obtained.

Another advantage of using the whole oven is that you can also use it to heat your lunch while you are producing strange chemical reactions.

FURTHER READING AND RESOURCES

The noted articles from *American Journal of Physics* may be found at your nearby technical library. Another source is UMI (formerly University Microfilms) which may be reached at (800) 521-0600. Reprints obtained through UMI typically cost in the $10 to $15 range with all copyright fees paid. UMI is a wonderful resource and the service is excellent. A call will also get you their informational brochure.

The article *Microwave Discharge Atom Source for Chemical Lasers* by R.A. McFarlane (Review of Scientific Instruments, Vol. 46, August 1975, pg. 1063) deals with an oven-tube powered plasma discharge apparatus with a plasma tube that passes through the waveguide. Regarding safety the author notes, "Where the discharge tube left the waveguide structure, radiation levels of 10 mW/cm^2 were detected, falling rapidly to less than 1 mW/cm^2 at a distance of 15-20 cm. It is concluded that no hazard exists for normal operating procedures, but the experimenter should be aware that 10 mW/cm^2 is an upper limit to avoid cornea damage and some caution should be exercised when viewing the discharge directly."

Elsewhere in this issue I noted the AVS monograph "Electric Probes for Low Temperature Plasmas" by David N. Ruzic. If you want to play with plasmas and probes and understand what you are doing, this little book is a must.

"Microwave Oven Repair" by Homer L. Davidson (McGraw-Hill/TAB, 1991) has a wealth of practical information concerning the innards of microwave ovens. This book is usually available even at well-stocked chain book stores. One reason for the proliferation of microwave ovens at the town scrap pile is the high cost of repair *vs.* the modest purchase price of the smaller ovens. A lot of ovens get scrapped for want of a 50¢ fuse or $10 rectifier. Since these ovens are incredibly easy to troubleshoot and repair, this book might save you some money even if you don't want to play around with plasma reactors.

Replacement tubes and other oven parts may be obtained from Richardson Electronics (40W 267 Keslinger Rd., LaFox, IL 60147) or MCM Electronics (650 Congress Park Dr., Centerville, OH 45459-4072).

This article was originally presented in Volume 4, Number 2.

Some New Books in the American Vacuum Society's *Classics* Series

The American Vacuum Society, through the American Institute of Physics (AIP), has added a few new titles to its series of reprints of classic vacuum publications.

Of particular interest to the amateur would be Walter Kohl's **Handbook of Materials and Techniques for Vacuum Devices**. This book provides information on the construction techniques associated with vacuum tubes along with a comprehensive discussion of the properties of the materials used in such devices. Construction topics include joining (soldering and brazing, glass to metal seals, metal to ceramic seals), cathodes and heaters, grid structures and coatings, getters, and secondary emission and voltage breakdown in vacuum. Materials discussed include glass, ceramics, mica, carbon/graphite, iron, copper, nickel, precious metals, refractories, molybdenum, and a host of others and their alloys. 623 pages, $45.00 non-members and $36.00 to members of AIP societies.

Several other previously released titles that have been mentioned in this journal and which are highly recommended include Alexander Roth's **Vacuum Sealing Techniques** (498 pages, $35.00/$28.00), Fred Rosebury's **Handbook of Electron Tube and Vacuum Techniques** (597 pages, $35.00/$28.00) and A. von Engel's **Ionized Gases** (325 pages, $35.00/$28.00).

Two other titles that may be of interest to the more advanced amateur would include **The Physical Basis of Ultrahigh Vacuum** by Paul A. Redhead, J. P. Hobson and E.V. Kornelsen (498 pages, $35.00/$28.00) and **Vacuum Technology and Space Simulation** by Holkeboer, Jones, Pagano and Santeler (339 pages, $35.00/$28.00).

AIP books and further information may be ordered by writing to the American Institute of Physics c/o AIDC, P.O. Box 20, Williston, VT 05495-0020 or by calling 1-800-809-2247 or fax 1-802-864-7626.

A Brief Tutorial on the Glow Discharge - Part 1

Steve Hansen

I. INTRODUCTION

"Of all the new phenomena wherewith the world has been enriched by modern physics, there is none more beautiful than the glow in a suitably rarefied gas. In the tube between the electrodes, the "viewless air" takes form and color; it seems to condense into luminous mists, to gather itself into islands of variously tinted cloud, which zones of darkness divide.

"The charm of these phenomena is nothing lessened by remembering that in history they make their first appearance as precursors of the conquest of our present wisdom. Modern atomic physics is the child of the vacuum pump. When pressures of the order of a millimetre were attained, the glow revealed itself in its greatest splendor. Attracted by the sight, physicists forged onward to lower and ever lower pressures as fast as better pumps were made. The splendor waned; but the change was simplification. Ultimately the glory of luminous clouds was gone; but by that time there were sharp and clearcut beams of radiation in the tube: one proceeding from the cathode into the zone of the discharge, others in the opposite sense....and a radiation arising from the places where the beam of the first mentioned hit the wall or any other obstacle. It then transpired that the rays of the first type were free electrons, those of the second were free ionized atoms, the last were photons of a frequency higher than any yet known. So came about the discovery of electricity released from matter, of ionized atoms freely wandering in space, and of the X-rays."

This is how Karl K. Darrow begins the chapter on the self-sustaining glow discharge in his 1932 book "Electrical Phenomena in Gases" [1]. These two paragraphs poetically summarize the study of electrical discharges in vacuum beginning with Jean Picard's observation in 1676 of luminous flashes in the "empty" space of a barometer tube to Wilhelm Roentgen's discovery of the x-ray in 1895.

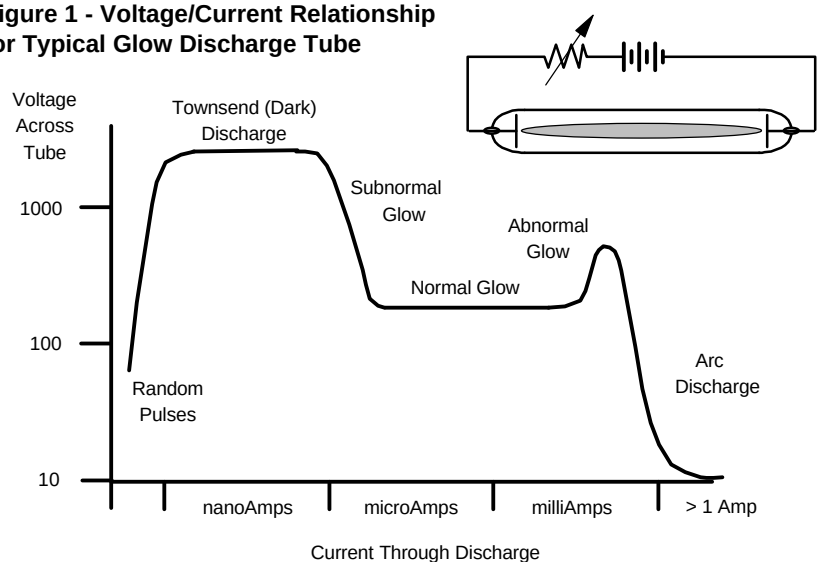
An earlier article in discussed some of the visual phenomena in a glow discharge tube. What follows is a thumbnail sketch, in far less eloquent terms than Darrow's description, of some other important characteristics of the glow discharge. A future article will cover additional aspects.

II. EVOLUTION OF THE GLOW DISCHARGE

A glow discharge takes place in an evacuated tube with two electrodes. When a suitably high voltage is impressed across the tube, there will be a breakdown and the gas will form into a plasma, a neutral mix of positive ions and electrons.

Figure 1 depicts the voltage vs current relationship for a glow discharge tube of the dimensions that an amateur experimenter might work with, i.e. 1 inch diameter electrodes placed about 18 inches apart. This tube would be operated at a pressure where the characteristic forms of the discharge will be revealed, i.e. about 1 Torr.

Figure 1 - Voltage/Current Relationship for Typical Glow Discharge Tube



If the current is slowly ramped by decreasing the value of the resistor, the gas in the tube will go from a non-conducting state to one in which there are very low current random pulses (induced by, in the absence of anything else, the passage of naturally occurring ionizing rays) which create a very dim, sporadic illumination. Through this region the current rise is small compared with the increase in voltage drop. This eventually changes to a region where the voltage rise slows as current increases more drastically. In this region, the discharge is called *self-sustaining*

as the current becomes independent of the external ionizing source, the applied voltage.

Increasing the current further leads to what we usually see as the typical glow discharge. There are three regions: the sub-normal glow, the normal glow, and the abnormal glow discharge. A discharge tube in the normal condition will show a bright region in close proximity to the cathode, the *negative glow* (see Figure 2). At first this sheath will only cover a portion of the cathode area. As the current through the tube rises, the negative glow will expand to cover the entire cathode. When the cathode is covered with the sheath, further current increases will drive the discharge into the abnormal region and the voltage will rise.

Note that the voltage across the electrodes is very constant while the discharge is in the normal state. This is the operating region that gas regulator tubes work in. (For those of you with fewer gray hairs, they were the equivalent of zener diodes.) Finally, with higher levels of current, the tube will rise and then, with enough current, will go into a very low voltage arc discharge mode. This happens when the current density is high enough to vaporize material from the electrodes, causing a drastic drop in resistance as the resulting vapor plasma fills the tube.

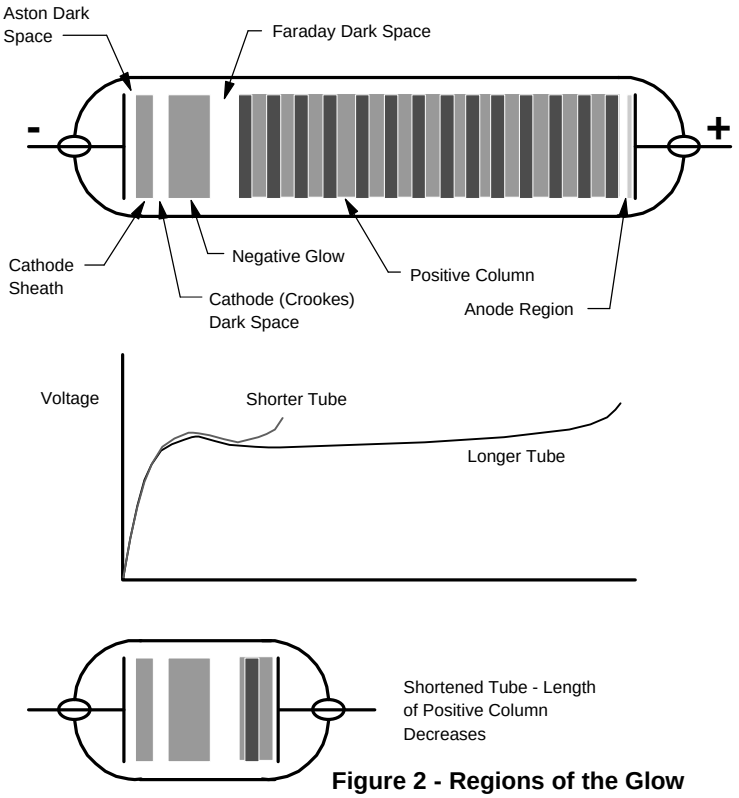


Figure 2 - Regions of the Glow Discharge and Voltage Distribution

The relatively low voltage that exists across the tube in the normal mode is a surprise to many people, particularly when such things as neon signs are driven by transformers in the 5 to 10 kV range. However, such transformers are really constant current devices: the high voltage capability serves to get and keep the tube in the normal discharge region but, as the current rises, the voltage drops to maintain a normal glow. If the transformer were not of special construction, the current would escalate resulting in arc formation and the destruction of the tube and transformer.

In other applications, such as in cold cathode electron guns, very high voltages can be sustained across the gap by supplying the potential in brief pulses. This drives the discharge into the abnormal region but keeps electrode heating to a low enough level that vaporization, and hence arc formation, does not occur.

III. GLOW DISCHARGE REGIONS AND CHARACTERISTICS

When a fully formed glow discharge is viewed, its appearance is similar to that of Figure 2. The details will change according to pressure and type of gas present but suffice it to say that there are two main groupings: the regions associated with the cathode (up to and including the Faraday Dark Space) and the regions associated with the anode).

The voltage distribution across the glow discharge is interesting. As shown in Figure 2, it is not linear. Instead, most of the voltage drop is near the cathode. This results in a high electrical field and this is the region where the electrons that are emitted from the cathode are accelerated. The fast electrons enter the negative glow where there is a high degree of ionization and this area tends to have a high positive space charge. In the positive column, the field strength is constant (as indicated by the gradual rise of voltage over the length of the column) and there is an equal distribution of positive ions and electrons.

At any given set of conditions (voltage and pressure) the negative region is a constant entity. If the pressure is lowered, the negative region will expand and the positive column will shrink. Also, as is shown in the lower illustration of Figure 2, if the electrodes are moved closer, the positive column will shrink to the point where it may almost completely disappear.

A nice, long and healthy positive column is needed in neon sign tubes. Such devices are operated at pressures well over 1 Torr where the negative region is

small. The sputtering process relies on the activity that occurs in the negative region and the dimensions and pressures are adjusted such that the positive column is of minimal length.

IV. THE OBSTRUCTED DISCHARGE

If the anode is moved even closer to the cathode, such that it closely approaches then enters the negative region, the voltage will have to rise in order to maintain a discharge. This is because there is less space for ionization to occur. The general form of the relationship is shown in Figure 3. Note that the sustaining voltage relates to the product of the pressure (in Torr) and the separation (in cm). A discharge that takes place in this regime to the left of the voltage minimum is called an *obstructed* discharge.

A cute device that demonstrates this is the so-called *detour* tube developed by Hittorf in 1884. (See Figure 4.) Here the direct path between electrodes becomes shorter than the cathode region as the pressure in the tube is lowered. When that occurs, the discharge takes the longer route through the side tube where a normal glow discharge can form.

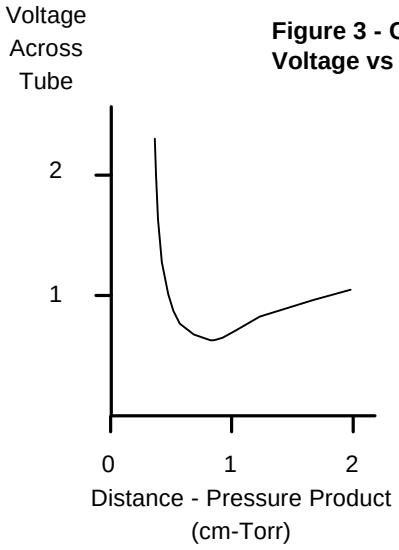


Figure 3 - Cathode Fall Voltage vs *D* x *p*

The occurrence of the voltage minimum in Figure 3 and the action of the Hittorf tube illustrates the *Paschen effect*, also known as the minimum sparking potential.

One practical use of this effect is for high voltage feedthroughs into vacuum chambers. Instead of trying to insulate the negative connection (which will more than likely result in virtual leaks and other problems), if the lead-in is surrounded by a coaxial anode with a spacing that is smaller than the negative region, the gap will hold off a quite high voltage. This type of feedthrough is discussed in [2].

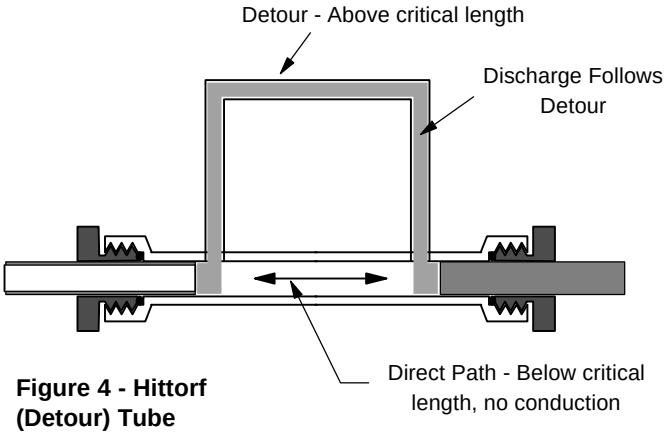


Figure 4 - Hittorf (Detour) Tube

REFERENCES

[1] Karl K. Darrow, *Electrical Phenomena in Gases* (Williams & Wilkins Co., Baltimore, 1932).
[2] L. Holland, *Vacuum Deposition of Thin Films* (Chapman and Hall, London, 1966).

See also A. von Engel *Ionized Gases*. This has been reprinted by the American Institute of Physics as part of the *Classics* series.

This article was originally presented in Volume 5, Number 1.

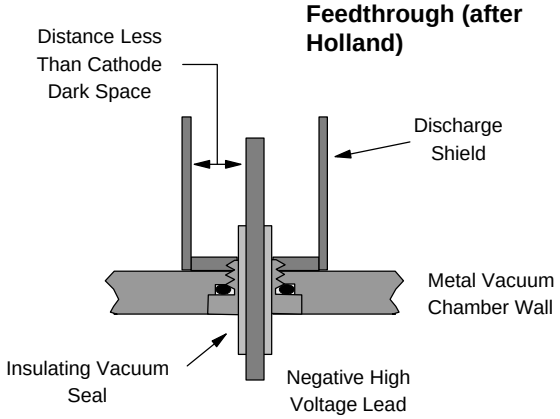


Figure 5 - High Voltage Feedthrough (after Holland)

A Brief Tutorial on the Glow Discharge - Part 2

Steve Hansen

I. INTRODUCTION

In Part 1 of this series we looked at the general characteristics of the glow discharge, how the self sustaining glow fits within the continuum from the dark discharge through the high current arc, and the nature of the obstructed discharge.

In this part we will look some more at the pressure - electrode separation relationship. We will also look at the hollow cathode effect.

II. AN EXPERIMENTAL GLOW DISCHARGE APPARATUS

The setup shown in Figure 1 can be used to investigate the characteristics of the glow discharge that were discussed in Part 1. A specific set of components will be detailed. However, substitutions can be made depending upon the availability of specific materials.

The discharge tube consists of a 300 mm length of 15 mm id Ace Glass chromatographic column (catalog number 5820-16. (Ace Glass columns and accessories were discussed in Volume 2 Number 2.) The nylon bushing/O-ring assemblies (2 ea.) are Ace Glass #7506-06. These Ace components will cost about $38.00.

The 15 mm bushings make a snug fit on a rod of 0.562 inch outside diameter. I made the pumpout tube from a piece of K&S Engineering 9/16" od brass tubing. I made the moveable solid electrode from 1/2 inch aluminum rod with a layer of shrink tubing over the rod as shown in the illustration. The shrink tube builds up the diameter enough to make a good fit in the bushing. It also insulates the surface of the rod, making the rod end the only exposed surface. While shrink tubing isn't a great vacuum material, at the rough and medium vacuum levels we are using and with modest tube currents (to avoid substantial electrode heating), it works fine. The aluminum rod will be the cathode and the pumpout tube will be the anode, at least for the first set of experiments. Aluminum makes a good cathode material especially as it has a low sputtering rate and, therefore, will tend to take longer to build up a deposit on the glass at the cathode end of the tube.

My power supply is a bit of overkill. It's a fully adjustable 500 to 10,000 volt regulated and current limited supply rated at 40 mA max. However, all that's needed is a dc supply with an adjustable resistor string. An autotransformer in the primary circuit of the supply is handy. Current is measured with a simple multimeter with several current ranges. For tube voltage measurement I used an old VTVM with a high voltage divider probe. A TV focus divider is cheap and equally usable.

Since the setup requires that the pressure be measured, a gauge is attached to the exhaust tube, as

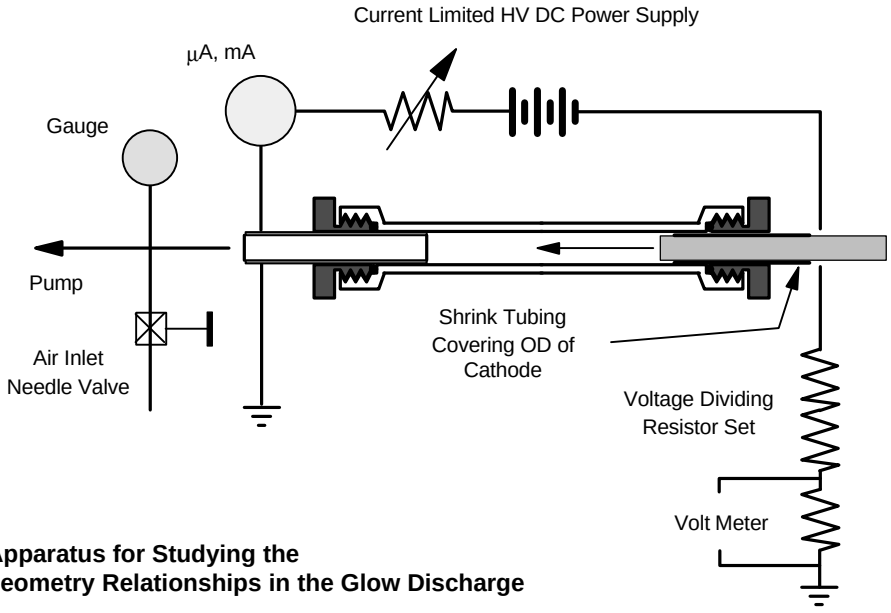


Figure 1 - Apparatus for Studying the Pressure-Geometry Relationships in the Glow Discharge

close as possible to the tube. As you will probably use a thermocouple or Pirani type gauge, remember that their responses will be considerably different if gases other than air or nitrogen are introduced into the tube (we'll discuss gas sensitivities of thermal conductivity gauges in the next issue).

Finally, some way of holding the pressure constant is required. A small needle valve, acting as a bleed, will do this quite admirably. American Science and Surplus has carried nice stainless/Teflon needle valves for about $5.00 each.

With all of the aforementioned put together, you can:

1. With a set pressure and electrode spacing, ramp the current to create a curve of the form shown in Figure 1 of Part 1. Then, investigate what effect differing pressures and electrode spacings have on the curve.
2. At varying combinations of pressure and electrode spacing, see how the tube voltage varies with the product of pressure and electrode spacing (see Figure 3 of Part 1).

These exercises show how pressure and geometry can be manipulated to produce low voltage drops as well as high voltage drops across discharge tubes. The former is desirable in such devices as neon signs and gas laser tubes. Referring back to Figure 3 of Part 1, these devices work on the right side of the voltage minimum where the distance-pressure product is higher. On the other hand, cold cathode x-ray tubes and electron guns, where high voltages must be sustained, work at low distance-pressure products, i.e. on the left hand side of the voltage minimum.

Finally, the breadth of the normal glow discharge plateau as well as the pressure/geometry relationships are key to the operation of gas-type voltage regulator tubes.

III. THE HOLLOW CATHODE

I mention the hollow cathode effect elsewhere without really explaining it. Von Engel [1] discusses the evolution of the effect and Figure 2 follows his explanation. If moveable disk cathodes are located on opposite sides of a ring shaped anode, each cathode will have a negative region associated with it. However, if the cathodes are brought close to each other such that the negative regions merge, then the current through the discharge, and its intensity, will rise abruptly. The effect will take the form of the curve in the figure: at lower values of the distance- pressure product, the current for the hollow cathode discharge will be higher than that for a the single electrode geometry by some multiplication factor.

How this happens, at least to the extent that it is understood at present, has been detailed by Schaefer and Schoenbach [2]. There are several mechanisms but the significant one causes electrons within the hollow cathode to be reflected from the opposing surfaces of the cathode and modify the electrical field in that area. The electrons also increase the level of ionization in the negative glow. Additionally, energetic neutral particles (including photons) that are created in the region will be more likely to hit to cathode surface, resulting in a further increase in electron emission.

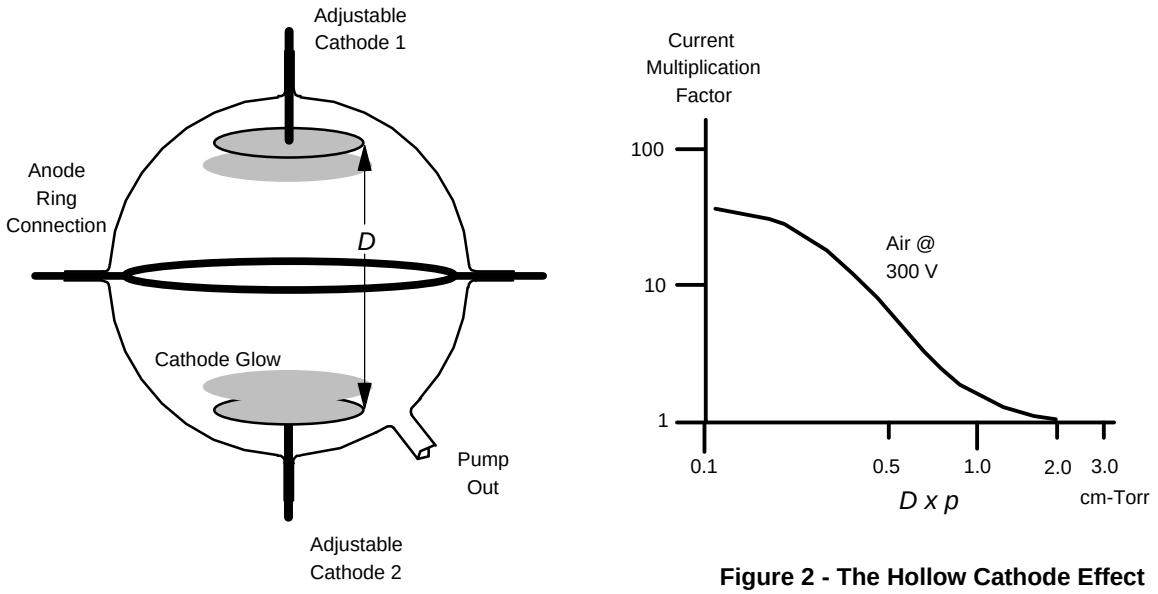


Figure 2 - The Hollow Cathode Effect

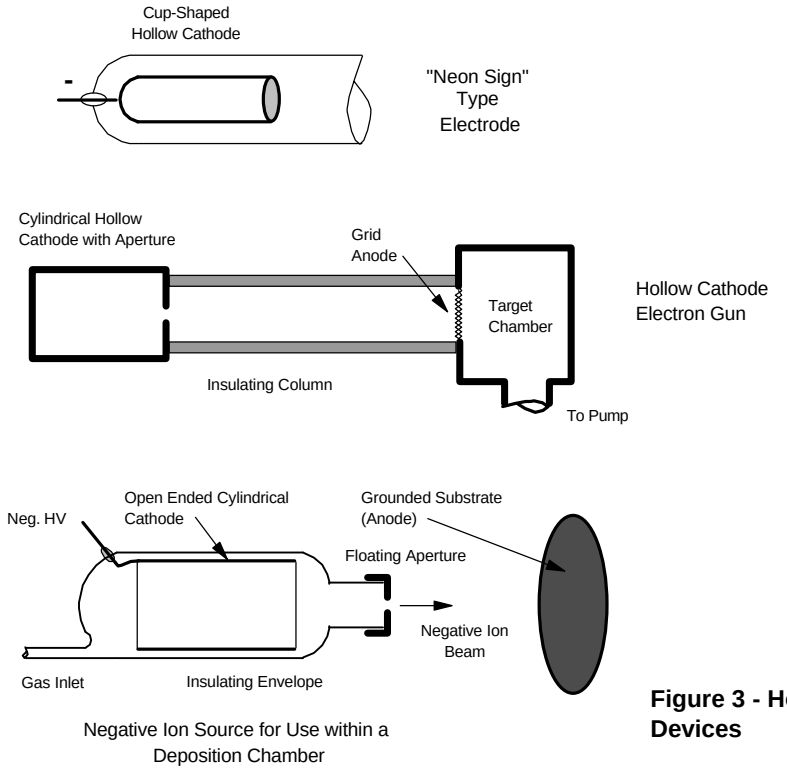


Figure 3 - Hollow Cathode Devices

A common application of the hollow cathode can be seen in neon sign tubes, some glow discharge spectral lamps and small cw gas lasers (see Figure 3, top).

The device shown in the middle of Figure 3 was used recently [3] to study pulsed electron beams produced by a hollow cathode with small aperture. Beam formation was studied at various pressures and with different cathode apertures. The impressed voltage pulse was on the order of 50 kV. Under some conditions, the tube would break down to a low voltage discharge. Under favorable conditions the full voltage remained impressed across the tube, resulting in a high energy electron beam.

The simple hollow cathode negative ion source shown at the bottom of Figure 3 has been used [4] to produce O^- ions. Placed within the chamber of an aluminum evaporator and oriented so that the ions impinge on the substrate, an aluminum oxide film is produced.

To conclude this part, although the serious study of the glow discharge had its roots in the gas tubes of the last century, there are many applications today. The hollow cathode effect is of particular value in high voltage and/or high current beam and switching devices and a considerable amount of research into both the fundamentals and applications is ongoing. The simple nature of the devices also makes them suitable for amateur study.

REFERENCES

[1] Alfred von Engel, *Ionized Gases* (American Institute of Physics reprint).

[2] G. Schaefer and K.H. Schoenbach, *Basic Mechanisms Contributing to the Hollow Cathode Effect* in "Physics and Applications of Pseudosparks" (Martin A. Gundersen and G. Schaefer, eds., Plenum, 1990).

[3] M. Favre, L. Arroyo, P. Choi, H. Chuaqui and E. Wyndham, *Experimental Investigation of Electron Beam Formation in High-Voltage Low-Pressure Linear Discharges* in "Small Plasma Physics Experiments" (S. Lee and P.H. Sakanaka eds., World Scientific, 1988).

[4] J. Ebert, in *Proceedings of the Society of Photo-Optical Instrumentation Engineers (SPIE), Optical Thin Films*, edited by R.I. Seddon (SPIE, Washington, 1982), p. 29.

This article was originally presented in Volume 5, Number 2.

Some Experiments with Glow Discharge Produced Electron Beams

Steve Hansen

I. INTRODUCTION

In the article on page 2-28 I described a set of modular metal and glass components which can be used for a variety of electron, ion and molecular beam experiments as well as for plasma studies. In this article I will concentrate on some experiments with electron beam production using a cold cathode glow discharge source. These experiments are conducted in rough vacuum, 30 to 100 mTorr being the recommended range. The reader should also refer back to the two articles which covers some of the characteristics of the glow discharge. Additionally, several references are noted with this article.

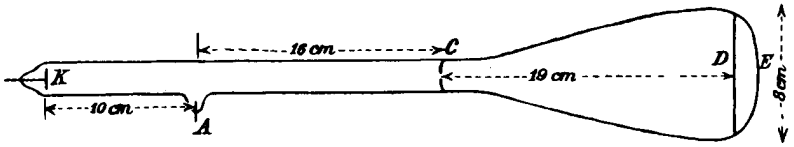


Figure 1 - Braun Tube (from *Annalen der Physik*, 1897)

Early cathode ray tubes made extensive use of glow discharges to produce electron beams. The Braun tube, dating from 1897 (see Figure 1), represented a practical application of the stream of development which progressed from Geissler to Crookes. This tube was the prototype of the modern phosphor-screened cathode ray tube. The Braun tube consisted of a narrow neck containing a pair of electrodes and a plate with a small aperture. With a discharge between the end mounted cathode (K) and the anode (A), electrons are liberated and accelerated through the anode region. A beam is formed when the electrons pass though the aperture (C). The beam then progresses to the screen (D) which provides the visual indication. External magnets could be used to deflect the beam as could internal electrostatic deflection plates.

When the electron beam passes through the coarse vacuum of the tube, there is a further focusing of the beam through an effect called *gas focusing*. This is caused by the electron beam's ionizing of the residual gas in the tube which then creates a positive space charge along the path of the beam. This positive 'channel' helps to prevent divergence of the beam.

The cold cathode CRT continued to be used even after the introduction of the more modern hard vacuum thermionic (filament) tubes. Particularly suited to the gas tube was the Dufour oscillograph where the phosphor was replaced by a piece of photographic film. The ruggedness of the Braun configuration along with

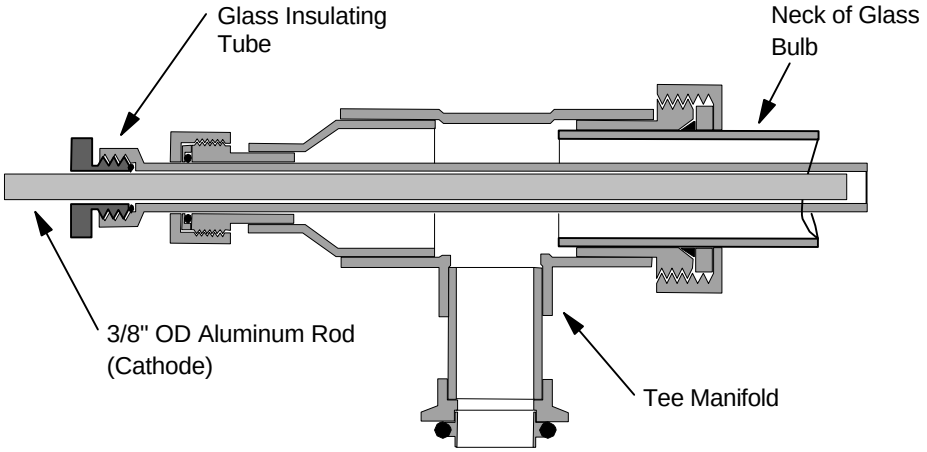


Figure 2 - Manifold and Cathode Assembly

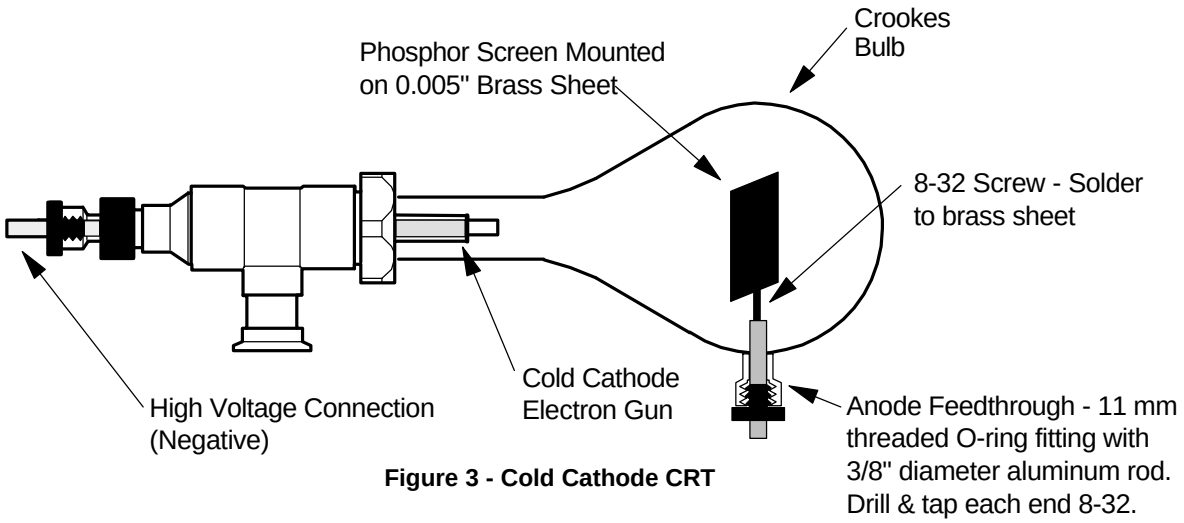


Figure 3 - Cold Cathode CRT

its modest vacuum requirements was particularly suited to this type of application.

II. APPARATUS

For the first set of experiments, the cathode is configured as shown in Figure 2. Here the aluminum electrode rod is simply inserted into the glass insulating tube with the end of the rod roughly flush with the inner end of the tube. For the chamber, I have shown a Crookes bulb, configured as shown in Figure 2.

In order to show the profile of the electron beam, a plastic encapsulated zinc-cadmium sulfide phosphor screen is attached to the anode feedthrough. These screens, normally used for industrial x-ray fluoroscopes, fluoresce bright green and have a moderate persistence, a good feature for analyzing pulsed beams. The particular screen used here is very flexible and can be cut to size with scissors (Note: cadmium sulfide is classified as a toxic material. When cutting a screen, do this in a well ventilated area and wash your hands afterward.) The active side of the screen is a yellow color.

The holder for the screen consists of a piece of flexible brass sheet (model builders stock from K&S Engineering or shim stock, either of which is available locally) of about 0.005 inch thickness. Cut a piece to about 1 x 2 inches and then solder a 1 inch long brass 8-32 round head screw to one of the short ends of the sheet. The feedthrough is made from 3/8 inch diameter aluminum rod, 2-3/4 inches long with holes tapped for 8-32 screws at each end.

A piece of phosphor screen is cut to about 1 x 2 inches and is then attached to the brass with a couple of 2 inch pieces of bare copper wire (#18 to #22). Simply wrap a piece of wire around the screen at the top and another at the bottom and crimp each around the brass sheet with a pair of pliers. Screw the screen assembly into the aluminum rod until there is about 1/2 inch of exposed screw thread. If the threaded hole is deeper than 1/2 inch, it might be helpful to put a nut on the screw and tighten it against the aluminum rod to keep the screen from rotating.

Getting the screen into the bulb is a bit like making a ship in a bottle. Feed the assembly rod first into the bulb. With a bit of juggling and perhaps some prodding from a metal rod, you can get the end of the feedthrough to go into the side port of the bulb. Here is where the flexibility of the screen and brass backing help. When the rod is in place, slip an O-ring over the rod and screw the nylon compression bushing into place. Make sure that the phosphor side of the screen is facing the opening of the bulb. With the screen centered in the bulb, the rod will just protrude from the nylon bushing.

At this point the bulb may be assembled to the manifold's compression fitting.

III. POWER SUPPLY

One or more power supplies are required for the experiments. You will need a dc supply capable of delivering several milliamps with an output adjustable from 1 to 10 kV. This supply should have a current limiting resistor string between the output and the discharge tube. Additionally, a higher voltage supply is also required. This can be an induction coil type of supply such as the one shown elsewhere in this issue. Both supplies should be wired to provide a negative high voltage output with the positive end grounded.

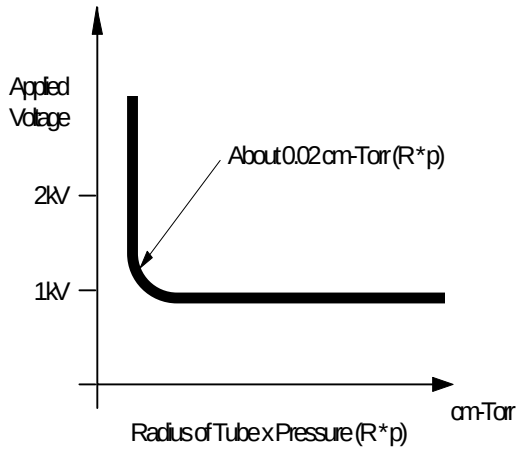


Figure 4 - Cathode Fall Voltage for a Constricted Discharge (after v. Engel, 1965)

IV. EXPERIMENTS

All of the following are best performed in a darkened room.

1. With the cathode rod end just flush with the insulating tube and with about 2 kV applied to the cathode, begin the pumpdown cycle. At a few Torr a bright discharge will commence and the phosphor screen will be uniformly illuminated. As the pressure decreases to below 100 mTorr, the discharge will become more beam like and eventually a coarse spot will form on the screen. (Be careful about exposing the phosphor to high current beams for long periods of time as the film will discolor or even melt. This is usually noticeable before any real damage is done.) You will also notice the pencil-like ionized beam projecting from the center of the cathode. This is the region that causes the gas focusing effect.

2. Make several runs with the cathode progressively withdrawn further and further into the insulating tube. You will notice that, for a given degree of vacuum, it will take higher voltages to maintain the discharge as the cathode recedes. This is called a *constricted* discharge. With the cathode about four inches inside the glass tube, it will not be possible to maintain a discharge and beam at pressures below about 100 mTorr, even with an applied potential of 10 kV.

The constricted discharge effect sets in at the point where the radius of the discharge tube is on the order of the mean free path of the ions in the tube. (Mean free path is defined as the average distance between collisions of molecules or ions in a system. For the molecules in air, m.f.p. corresponds to about 0.005 cm at 1 Torr and varies linearly with pressure.) When the product of the radius of the tube (in cm) and the pressure (in Torr) is less than 0.02, the characteristic of the discharge changes and only much higher voltages can sustain the discharge. It is believed that x-ray production plays a role in sustaining these higher voltage constricted discharges.

A crude approximation of this characteristic is shown in Figure 4. A fuller explanation of the effect is provided in von Engel's book [1].

Referring to the figure, you will note that the voltage across the tube in the unconstricted mode is only several hundred volts. Then why does one need a multi-kV power supply for 'normal' discharge tubes? The answer is that the high potential is needed to start the discharge. After that, the potential falls to some sustaining level. Neon sign transformers, for example, are designed to supply a constant current (usually on

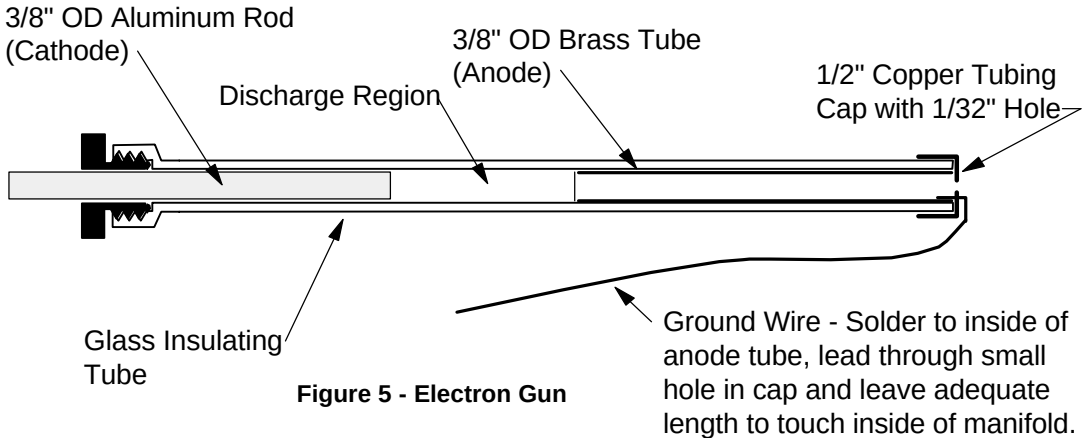


Figure 5 - Electron Gun

the order of 20 mA). Once the discharge is initiated, the transformer output voltage drops considerably. If that were not the case, the current would escalate rapidly (with the transformer trying to maintain full output voltage) and either the tube or the transformer would fail from overheating. In the case of our dc supply, the excess voltage drop is consumed by the current limiting series resistor string.

Going back to the constricted discharge, this is not a desirable condition for neon signs. Tubes are initially filled to a pressure that will avoid this condition - well over 1 Torr. Sometimes, as tubes age, the internal pressure will drop due to gettering and the resulting increase in glow discharge maintenance voltage will ultimately exceed the transformer's capabilities. Flickering and then darkness will be the result. On the other hand, the constricted discharge is important for cold cathode electron guns as this configuration permits high accelerating voltages to be attained. For the amateur, constricted discharges permit the easy production of moderate current high energy electron beams without the need for hard vacuum conditions, fragile filaments, or complex focusing optics.

V. A PRACTICAL COLD CATHODE GUN

Figure 5 shows a very simple form of cold cathode gas discharge gun. This may be fabricated using the insulating tube and aluminum cathode but adding an internal tubular anode and a focusing aperture. The anode tube is made from a piece of 3/8" diameter brass tubing (e.g. K&S Engineering), about 4 inches long. The aperture is a standard 1/2 inch copper tubing cap (5/8 inch inside diameter) which can be obtained from a hardware or plumbing supply store. A small hole, about 1/32 inch, is drilled in the center of the cap. This serves to constrict the electron beam. Another small hole is drilled near the edge. A piece of bare copper wire (#22-26) is soldered to the inside of the brass anode tube and is then passed through the off-center hole in the cap. About 8 inches of wire should hang from the cap. This is pushed into the manifold to ground the cap and anode. With the insulating tube installed in the manifold, insert the anode tube and then place the cap over the end. To help the cap stay in place, squeeze it slightly out of round with a pair of pliers. Make sure that the ground wire is pushed into the manifold and is bent sufficiently to ensure contact. Set the cathode rod to provide a cathode to anode separation of 2 to 4 inches. At this point, reattach the glass bulb and check all O-ring seals for tightness.

Connecting the cathode terminal to the negative end of a high voltage power supply (here is where you will want to use an induction coil), and with the manifold and phosphor screen grounded, begin to evacuate the device. At around 100 mTorr a very well defined spot will begin to form on the screen. Assuming the voltage is high enough (40 to 60 kV), the beam will remain down to the few mTorr range.

Now that you have a beam, you can try some experiments using permanent magnets or electro-magnets to deflect the beam.

VI. ADAPTING A SURPLUS OSCILLOSCOPE TUBE

Instead of the glass bulb with the separate phosphor screen, an old CRT can be adapted to work with the cold cathode gun and the manifold. "Easy" I originally thought. All I would need is a CRT with a 1-1/2 inch neck, one that a surplus house like Fair Radio Sales would have in big supply for 10 bucks each. It wasn't quite that easy. It seems that the usual diameters of small (2 to 7 inch) CRT necks are 1, 1-3/8, 1-5/8 and 2

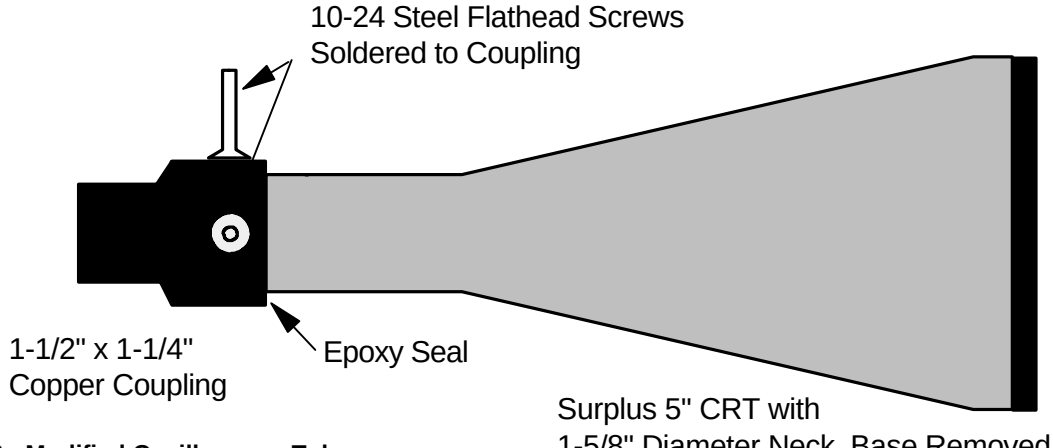


Figure 6 - Modified Oscilloscope Tube

inches. (For this bit of knowledge I have to thank the people at Fair Radio Sales. They kindly sorted their available tubes by neck size for me.) But, not to give up, my perverted knowledge of plumbing supplies said that I could adapt a 1-5/8 inch neck to my 1-1/2 inch manifold compression fitting with a standard 1-1/2 x 1-1/4 inch copper reducing fitting as the inside of the big end is 1-5/8 inches and the outside of the small end is 1-1/2 inches. All I would need to do would be to cut off the base and epoxy the fitting to the modified CRT. As Fair Radio has a good stock of compatible tubes, I purchased a couple of 5DEP1s for $17 each.

Figure 6 shows the converted tube. The first step is to remove the base and cut the tube. I just broke the pins off with a pair of needlenose pliers. This cracked a couple of seals, letting air in (it has to happen sometime). Then I scratched a line around the neck with a triangular file. Cutting the neck was accomplished with an improvised hot wire cutter. The final step is to pull out the electron gun assembly.

When opening and cutting, be careful not to let bits of glass or dirt get into the tube as they can scratch the phosphor. I would also imagine that cleaning the inside of an opened CRT would be almost impossible without harming the phosphor or aquadag coatings.

The copper coupling is prepared by soldering two 10-24 steel flathead screws to the larger part of the coupling as shown in the figure. These should be at right angles to each other. These screws are used to attach a pair of solenoids for use in magnetic deflection experiments.

For final assembly, degrease the coupling and the outside of the tube neck. Then smear a thin layer of white epoxy on the inside of the coupling and insert the tube neck until it is seated. (Check this fit before glue is applied to make sure that the tube indeed fits. You might have to remove some copper from the fitting using either a lathe or a small sanding drum.)

Before placing the modified CRT bulb on the manifold, attach another ground wire to the one which connects the gun anode to the manifold. This wire should extend into the CRT and touch the conductive aquadag coating. When assembling, make sure that the exit aperture of the gun is behind the deflection solenoid attachment screws.

When operating the tube, keep the current low, especially when the beam spot is stationary. Otherwise the phosphor will burn.

Deflection coils may be made by winding wire around the screws. You can also use coils which have been salvaged from old relays, buzzers, or other electromechanical devices. Lissajous patterns may be created by feeding 60 Hz to one coil and the output of a variable frequency audio oscillator to the other.

Suitable 1-5/8 inch neck CRTs also include the 5UP1 and 3KP1. 1-3/8 inch neck tubes, of which there seem to be a greater variety, can also be used with the same coupling. Just file away the tube stops from the inside of the small part of the copper coupling and glue the CRT neck to this area. Tubes with 1-3/8 inch necks include 2AP1, 2BP1, 3RP1, 3WP1 and 5FP4 & variants (5FP7, 5FP15, etc.) A number of these are available from Fair Radio Sales at prices ranging upward from $12.

REFERENCES AND SOURCES

[1] A. von Engel, *Ionized Gases* (Oxford, 1965). This book has been recently republished as part of the *AVS Classics* series. See the note in the Winter 1994 issue.

Another useful book is James Cobine's *Gaseous Conductors* (McGraw-Hill, 1941). This book contains a good discussion of cold cathode electron guns. Dover reprinted this book as a paperback but I am not sure if it is still in print.

The manifold, glass bulb, and phosphor screen are available through the author. K&S Engineering's metal products are widely available through local hardware stores and hobby shops. Fair Radio Sales can be reached at P.O. Box 1105, 1016 E. Eureka St., Lima OH 45802, (419) 223-2196.

This article was originally presented in Volume 3, Number 2.

Demonstrate a Transmission Electron Microscope

Steve Hansen

This demonstration was part of my talk at the AVS Education Outreach session and was inspired by a column in *The Amateur Scientist* (9/73) in which a group of high school physics students built a cold-cathode single lens TEM as well as a hot cathode compound TEM. Their simple TEM could magnify about 10 to 100 times while the compound instrument was capable of magnifications to 10,000x. Both instruments used fairly large electromagnetic lenses with iron pole-pieces internal to the vacuum chamber.

This device is built using the manifold and gun assembly as detailed earlier. I used the front of a commercial surplus CRT for the drift and display section.

Instead of an anode with a small, single perforation as was used in the previous article, the TEM uses an anode made from a piece of brass screen with a fairly fine mesh. The shadow of this “grid” is what will be projected and magnified by the "microscope", the shadow image falling on the phosphor screen.

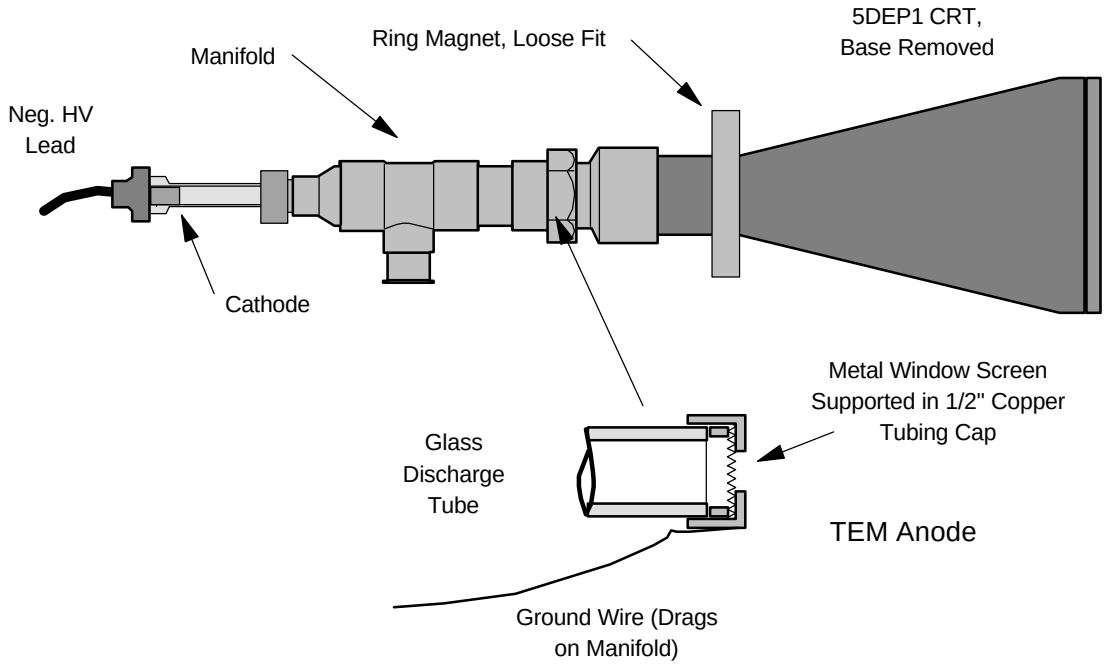
Power is provided by a small (1/2" spark or better) spark coil. The “hot” electrode is the cathode and this must be biased negative.

The single focus lens is a ring magnet of the type used for loudspeakers. These are readily available through surplus outlets. Nothing is critical about the magnet except that it should make a very sloppy fit over the neck of the modified CRT. The magnet is wrapped with vinyl electrical tape to provide some cushioning.

With the magnet placed around the neck of the CRT, begin pumpdown. Once the pressure approaches a few Torr, the spark coil is turned on. Eventually a diffuse glow will appear which will then become more defined as the pressure declines. Eventually the grid will become visible, with the image getting crisper as a pressure of about 100 milliTorr is approached. This effectively demonstrates the concept of mean free path.

With the image now well defined, moving the magnet will produce a magnified image of the screen mesh. Allowing the members of the audience to adjust the magnet gives them a feel for how the magnetic focusing of electrons is much like using a glass lens to cast a magnified image of an object.

This article was originally presented in Volume 4, Number 4.



The Multiplate Chamber (MPC) Electron & Ion Beam Source

Production of intense charged particle beams by means of a simple pulsed "pseudospark" device

Steve Hansen

I. INTRODUCTION

This article describes the principles behind a relatively simple device which can be used to produce high intensity energetic beams of both electrons and ions. This device, variously called the multiplate chamber source (because of its electrode configuration) or pseudospark source (for the type of discharge which produces the beam), relies on the special properties of its electrodes, a stack of metal disks, each having a center hole and separated from each other by insulating spacers.

Since the device both produces and accelerates the electron and ion beams, it may be thought of as a simple implementation of a particle accelerator. For background, we will begin with a discussion of the potential drop accelerator.

II. POTENTIAL DROP MACHINES

Most amateurs have some familiarity with particle accelerators, in particular those of the dc or potential drop variety. Typically powered by a Van de Graaff electrostatic generator or by a voltage multiplier of the Cockcroft-Walton design, these machines accelerate particles to an energy determined by the potential difference between the high voltage terminal and ground. Typically this is in the range of 100,000 to a few million eV. The upper end of the energy range is restricted by the practical limits (mainly insulation) of high voltage dc power supplies. For higher energies alternative approaches are used in which successive "kicks" are given to the accelerating particles, thereby avoiding the need for high voltages. These "multi-transit" accelerators include linear accelerators, cyclotrons, synchrotrons, and so forth.

Potential drop accelerators have found some interest in the amateur community. In the 1950s F. B. Lee built a 250 keV electron accelerator powered by a Van de Graaff generator. This was described in *The Amateur Scientist* column of Scientific American and was later included in C. L. Stong's compilation "The Scientific American Book of Projects for the Amateur Scientist" (Simon and Schuster, 1960). A later treatment is found in a contribution by Larry Cress to *The Amateur Scientist* of August, 1971. This machine incorporated a simple ion source for the generation of protons and deuterons.

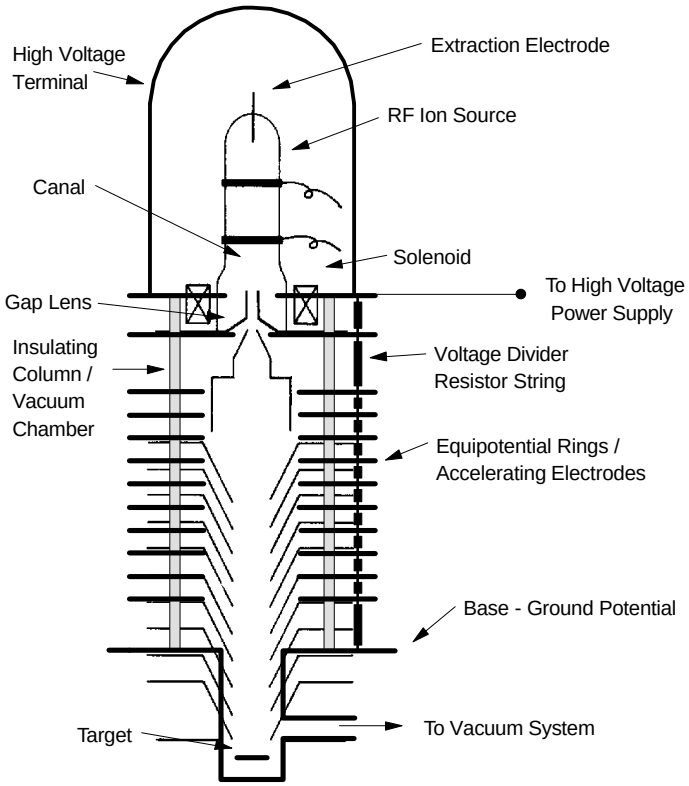


Figure 1 - Schematic Representation of a Potential Drop Particle Accelerator

Some plans are presently commercially available from at least one company but, from what I've seen, you are better off looking up the old Scientific American information.

While this article is not on potential drop machines, some information on this approach will help to put the MPC into context.

Figure 1 shows a schematic representation of a simple potential drop accelerator. Such machines, operating at about 250 keV, are used for neutron activation analysis. Equipped with an electron source and higher potential (up to several million volts), such machines are also used for industrial radiography.

In the figure, the high voltage terminal contains an RF driven ion source. The RF, usually at the industrial frequency of 13.56 MHz, is capacitively coupled by means of the two rings around the glass chamber or bottle. Gas (e.g. hydrogen or deuterium) is introduced into the source in a slow, controlled manner by means of a leak. The RF creates a plasma within the bottle which is extracted by the combined action of a positive potential applied to the extraction electrode (at the top, closed end of the bottle) and a negative potential applied to the gap lens at the open end of the bottle. Through this action, positive ions will emerge from the narrow canal into the acceleration column. All of the RF and dc power supplies for the ion source are contained within the high voltage terminal with the ion source base acting as the voltage reference.

The acceleration column consists of a series of electrodes which may be in the form of a series of disks, tubes, or funnel shaped structures. The total potential of the accelerator is divided between the electrodes by means of resistors or, in smaller machines, by corona points. The electrodes are configured to not only keep the beam focused and narrow but also to keep the beam from striking the insulating walls of the column. (If the beam was allowed to strike the chamber, the charge buildup would eventually lead to loss of focus, internal sparking and possible rupture of the column.)

The bottom, grounded end of the accelerator contains the target, or, if the beam is to be used external to the accelerator, a thin window. The connection to the vacuum pump is made here as well.

The potential drop accelerator requires high vacuum (10^{-5} torr or better) to prevent internal arcing as well as to ensure an unimpeded path for the accelerated particles. Beam currents are typically low, in the microAmp to low milliAmp range. Voltage regulation can be very good in a well designed machine. This results in a well controlled, steady state beam.

III. THE MULTIPLATE CHAMBER SOURCE

Figure 2 is a schematic representation of the multiplate ion/electron source. Its construction is very simple, consisting of only a series of metal disks with coaxial apertures separated by insulating plates. The driver is a small value capacitor which is connected to a high voltage power supply. Thus, this source is a pulsed device as contrasted with the steady state operation of the dc potential drop accelerator with its RF ion source.

The MPC was first described by J. Christiansen and C. Schultheiss in 1979. Initial work involved the use of the configuration as a high power switch, comparable to the action of a thyratron. Later work began to exploit the fact that intense beams are developed along the axis of the chamber.

The principles behind the discharge mechanism are still not totally understood, but considerable progress has been made in the past few years. The following description follows the explanation provided by E. Boggasch and M. J. Rhee [1].

The discharge action begins with a very low current (microAmp range) predischarge (called a Townsend discharge) of several microseconds duration. During this period, a light emitting zone travels from the anode toward the cathode. At this point, the current rises to a few hundred microAmps and a constant current glow discharge is established. Positive ions begin to accumulate along the axis, forming a positive space charge. When a critical level of charge is reached at the cathode hole, a strong discharge is ignited. (This is called a hollow cathode discharge and the characteristics are related to the geometry of the tubular cathode.) Ionization waves move into the cathode at velocities of about 1 million meters/second and an intense electron beam is ejected at the anode. The voltage then begins to drop with an accompanying steep rise in current. The high current densities (on the order of 100 kA/cm^2) are attributed to field enhanced emission from the melted electrode surface.

Typical electron beam parameters for the MPC include a peak beam current of a several kiloamperes with a pulse duration of about 50 nanoseconds. The ion beam arises mainly from the action of electrons on the target material.

Vacuum requirements for an MPC source are modest; the usual range is 40 to 80 mTorr. As higher voltages can be sustained at the lower pressures, the beam will be more energetic in the low mTorr range.

The discharge can be initiated by simply reaching the breakdown voltage of the device, or, for either lower voltages or lower pressures, the discharge can be initiated with a trigger spark in the cathode region. Typical operating voltages shown in the

literature are in the range of 10 to 60 kV. At higher voltages external path breakdown can be a problem. I've operated an oil insulated device at voltages well over 100 kV.

The geometry of the MPC creates a pinch effect which forces the discharge to occur along the axis of the device. This pinching keeps electrode erosion to a minimum and also helps to avoid damage to the insulating plates. X.L. Jiang and N. Xu [2] showed that multiple apertures would create multiple beams as long as the apertures in each plate were well aligned with the corresponding apertures in the other plates.

The number of plates represents a balance between desired holdoff voltage (worse with fewer plates) and beam attenuation (worse with more plates). A good range is five to seven.

Jiang and Xu in reference 2 give some guidelines for hole and separation dimensions. They state a desirable ratio $r/d = ½$ where r is the radius of the electrode hole and d is the distance between adjacent electrode plates. They caution that, as the ratio is increased to over 4, the MPC will begin to behave as a "normal" glow discharge tube. Hole diameters used by them ranged from 1 to 5 mm with higher currents being achieved with the smaller holes.

In my experiments I have used standard storm door type Plexiglas in 0.100 inch thickness as the insulating material. For electrodes, an acceptable ratio can be obtained (close enough anyway) by soft silver soldering together a pair of standard stainless steel fender washers of 5/32 inch id and 7/8 inch od. The resulting thickness is also compatible with a standard 7/8 inch by 1/8 inch O-ring. All of these components and materials are available at a well stocked local hardware store.

As stated before, the capacitor value required is modest. High voltage ceramic capacitors of the doorknob type are quite suitable and are available at modest cost as surplus from suppliers such as C&H Sales. 500 to 2000 pF is a good range to work with. To minimize inductance (the MPC is a high speed device), the capacitor should be located very near to the MPC itself.

Because of the nature of the discharge, the energy/intensity spectrum is not uniform over time. The first effort at producing a time-resolved spectral study of the MPC electron beam was undertaken by B. N. Ding *et al.* and reported in *Review of Scientific Instruments* just this past June [3]. This study used a 3 plate MPC charged to a maximum voltage of 15 kV and operating at about 80 mTorr. In a typical run, the beam energy was seen to peak at 14 keV, nearly the full bias voltage, after about 7 nanoseconds. The current at this point was around 30 mA. The current continued to escalate to about 230 mA as the beam energy declined to 8 keV after 15 nanoseconds. After this, both the energy and current continued to decline, reaching extinction by 25 nanoseconds.

Thus, the MPC can be seen to be a simple high brightness source of electrons and ions that will work at fairly high pressures. Compared to the potential drop accelerator, the intensity/energy distribution is highly time variant and only pulse operation is possible.

IV. APPLICATIONS

The previous referenced articles cite a number of applications which are being explored for the MPC source. These include very efficient production of soft x-rays (with a heavy metal target), rapid solid annealing, pulsed microdoping, irradiating graphite to form diamondlike particles, micromachining, production of superconductors, and the generation of microwave energy.

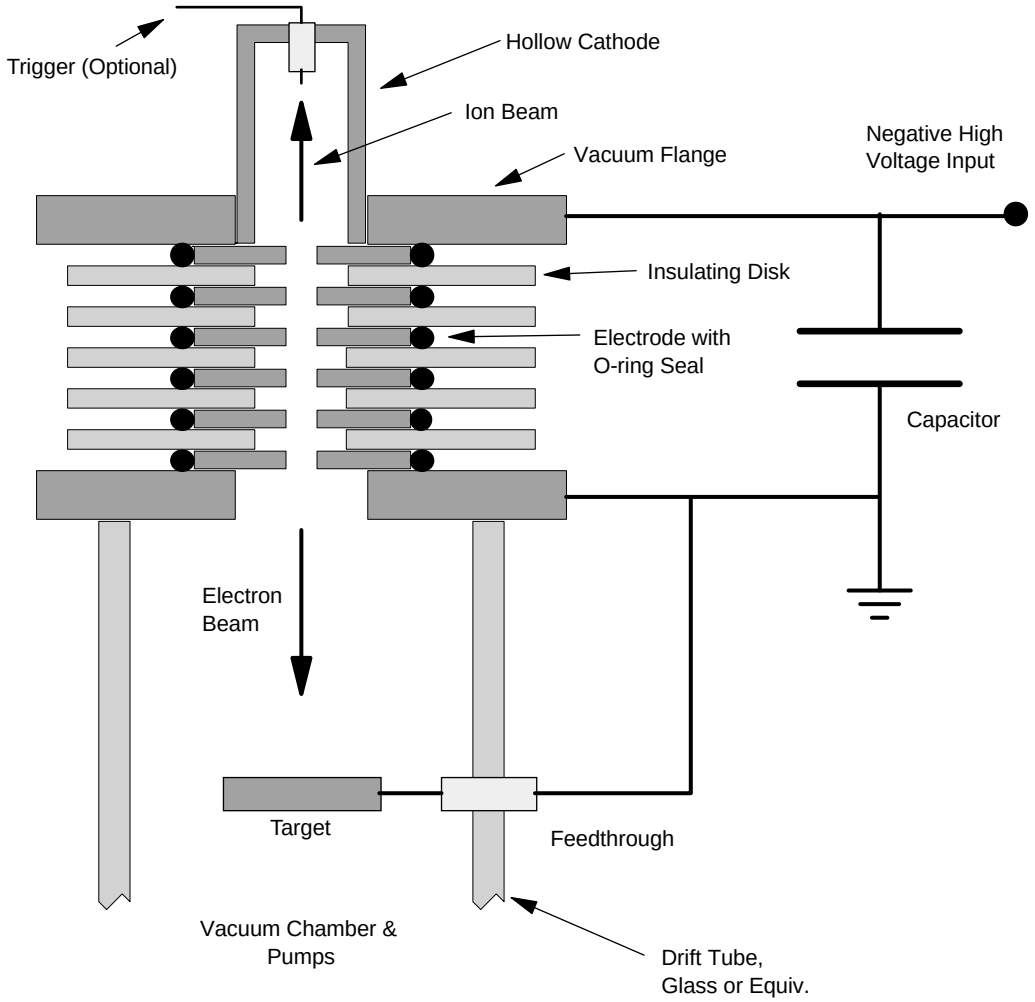
REFERENCES

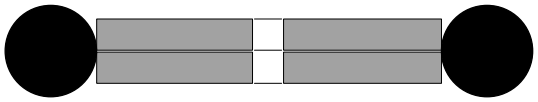
[1] E. Boggasch, M.J. Rhee, Second International Conference on Dense Z-Pinches, Laguna Beach, CA, 1989 (edited by Nino Pereira, Jack Davis and Norman Rostoker). AIP Conference Proceedings #195.

[2] Xing-Liu Jiang and Ning Xu, Review of Scientific Instruments **61**, 644 (1990).

[3] B.N. Ding, T.J. Myers and M.J. Rhee, Review of Scientific Instruments **64**, 1442 (1993).

This article was originally presented in Volume 2, Number 4.





Suggested Electrode Configuration - two 7/8 inch diameter x 1/16 inch hole stainess steel fender washers soldered together with 7/8 inch ID by 1/8 inch diameter O-ring.

Figure 2 - Schematic Representation of Multiplate Chamber Electron/Ion Source

Build a Multiplate Chamber Beam Source

Steve Hansen

I. INTRODUCTION

In the previous article I discussed the general principle of the multiplate chamber (MPC) source as a means for generating pulsed beams of both ions and electrons. This article will focus on the details of construction of such a source.

The unique features of this design include (with the exception of a brass coupling and CF flanges) the exclusive use of easy to obtain hardware store components. Also, the source may be insulated with oil, thereby permitting the use of high discharge voltages without the risk of external arc-over.

The source is designed to be attached to the beam experiment apparatus which was also described in the previous issue.

The general layout of the device is shown in Figure 1. While it looks fairly complex, the elements are simple and generally non-critical. Construction details will be provided in a step-by-step fashion.

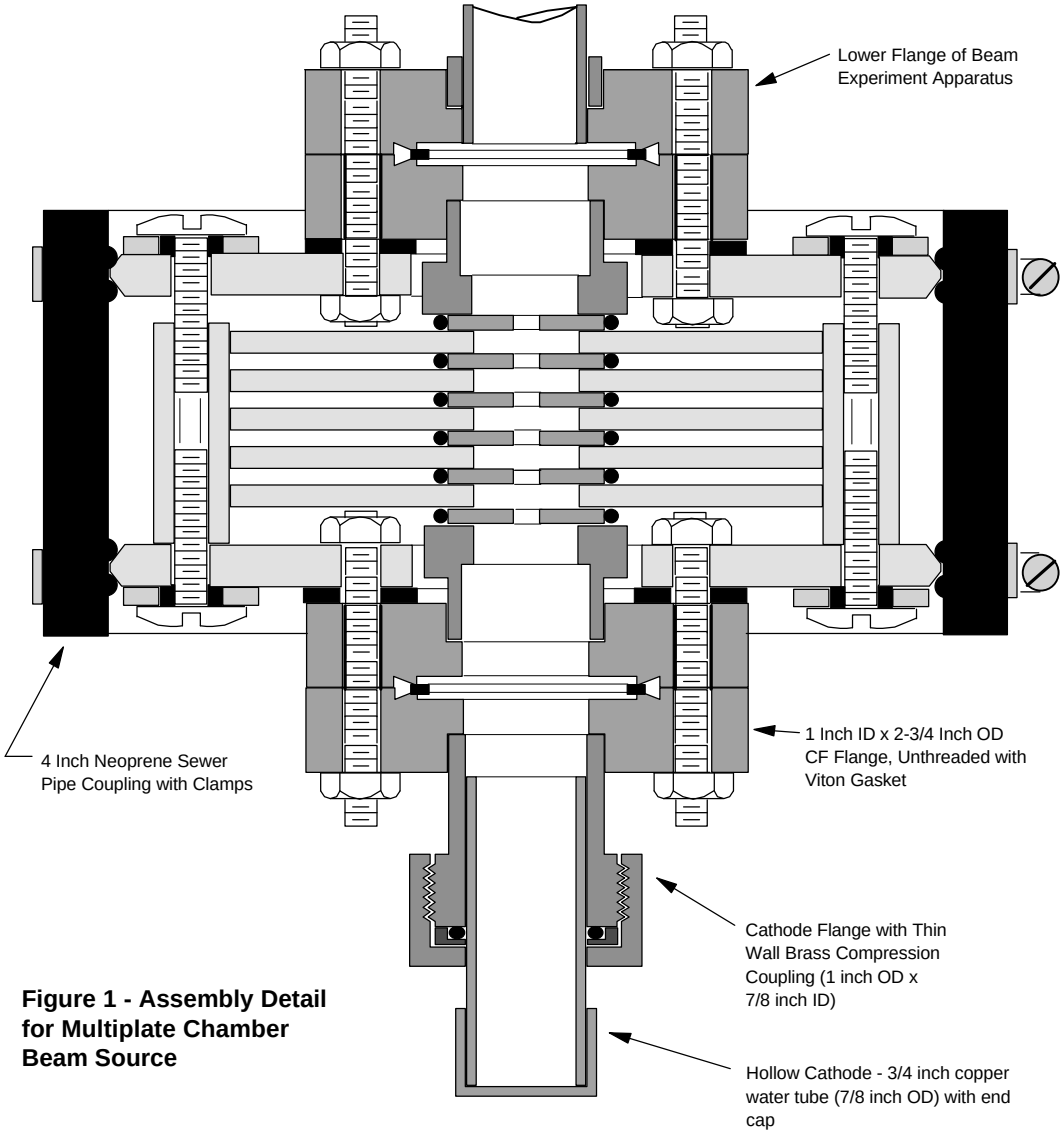


Figure 1 - Assembly Detail for Multiplate Chamber Beam Source

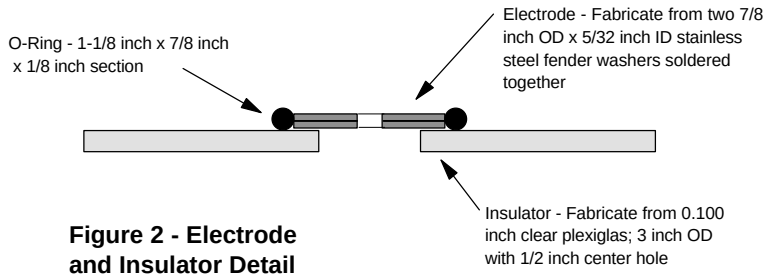


Figure 2 - Electrode and Insulator Detail

II. ELECTRODES AND INSULATORS

Select a handful of flat stainless steel fender washers, 7/8 inch od x 5/32 inch id. You will have to solder together pairs of these washers in order to fabricate the floating electrodes. This is fairly easy. First, clean the washers in alcohol. Then apply flux to one side of each of the washers. With a gentle torch flame tin one washer with 4% silver - tin 'hobby' solder. Do not deposit too much solder. Then, place the other washer on top of the first, fluxed side down. With a little more heat, the washers will join with surface tension pulling the two into alignment. If you didn't go overboard with solder, the hole will still be clear and the solder will be seen to have flowed to the edge but not bubbling out. Six sets are needed but make a few more and select the better ones.

The insulating spacers are made from storm door plexiglas. Most hardware stores stock odd sizes so you should not have to purchase an entire sheet. Using a fly cutter or hole saw in your drill press, cut out five disks, three inches in diameter. In the center of each, bore a hole ½ inch in diameter.

Since the washers and spacers are components of the vacuum chamber, these must be air tight. This is done using O-rings as shown in Figure 2. As the soldered washer pairs are about 0.110 inches thick, O-rings with a cross section of 1/8 inch are suitable.

Two end plates as shown in Figure 3 are required. I made mine from ¼ inch thick tinted Lexan, obtained as an odd piece from a glass shop.

The six holes in the inner ring mate with the CF flange studs. The outer row is for the nylon screws which pull the flanges together, compressing the electrode stack O-rings and holding the entire assembly in alignment. The center hole has to be large enough to clear the brass pedestal on the end flange.

The reason for the curious bevelling of the perimeter is to provide a snap-fit into a neoprene sewer pipe coupling. Referring to Figure 1, this coupling (*PlumbQwik*, a standard plumbing supply store item), which is about 2-1/8 inch long, has two pairs of raised ribs on the inside. The center to center distance between the grooves formed by the ribs is about 1 - 9/16 inches. The edges of the Lexan are bevelled in order to properly mate with these grooves. (Note that the coupling is only needed if the MPC is to be oil insulated. If you plan to operate the source at potentials under about 50 kV this feature is not needed.)

One disk should have a hole drilled and tapped for the oil filler plug. Again, this is not necessary if the source is to be operated in air.

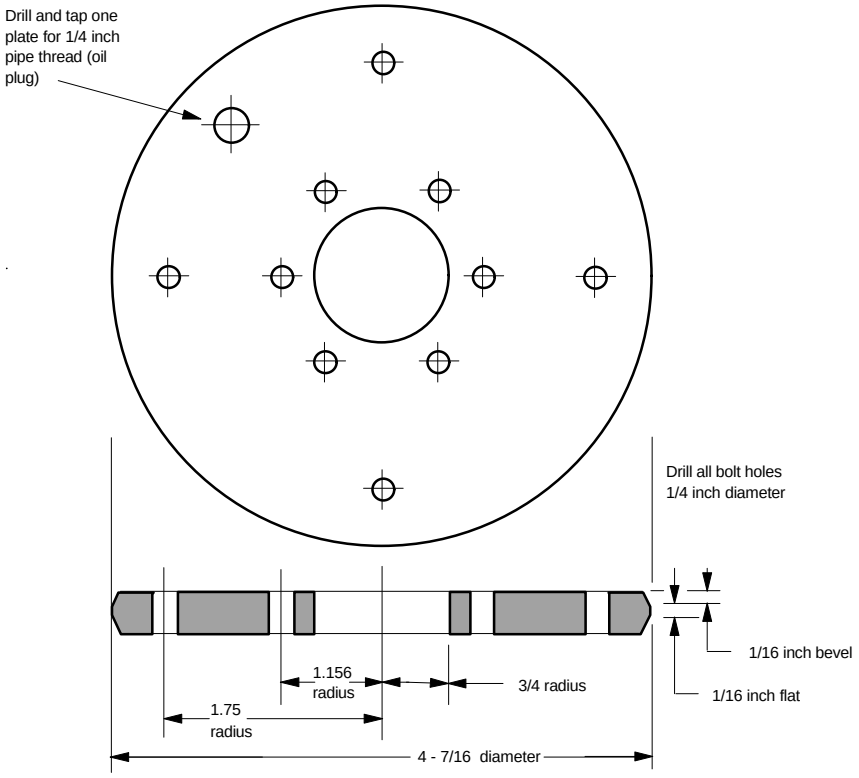


Figure 3 - Lexan End Plate

III. END FLANGES

Two end flanges are required as shown in Figure 4. The parts for each flange include a standard CF vacuum flange (stainless, 2-3/4 inch diameter with a 1 inch bore, tapped for ¼-28 bolts), six 2 inch lengths of either stainless steel (preferred) or brass threaded rod, a brass compression fitting for 7/8 inch tubing, and some 3/32 inch oil resistant (e.g. neoprene) gasket stock.

The purpose of the modified compression fitting is to provide a raised pedestal for the stack of electrode plates. If you do not wish to do the machine work required to modify the fittings, feel free to improvise. The height of the fitting is consistent with the bevel dimensions of the Lexan plates, an actual thickness of 0.090 inch for each of the plexiglas disks, and a typical thickness of 0.110 for each of the electrodes. Again, if you elect not to oil insulate the source, this dimension is not critical. But, if you do want the oil feature and if your electrodes or other components in the stack deviate significantly from these dimensions, you will need to adjust the height of the pedestal.

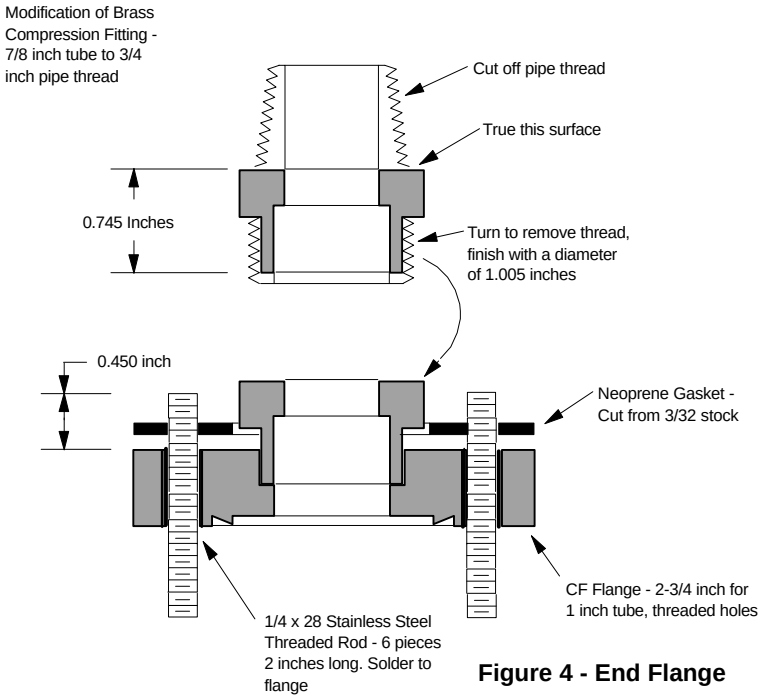


Figure 4 - End Flange

The threaded studs are screwed into the flange as shown. Pay attention to the protrusion from the flange or the studs could interfere with the insulating plates. With the studs and pedestal in place, solder these to the flange with 4% silver - tin solder.

Finally, prepare a pair of neoprene gaskets per the dimensions shown in the figure.

IV. FINAL ASSEMBLY & TESTING

All soldered components will need to be thoroughly cleaned with hot, soapy water and an abrasive pad (the type made by 3M for cleaning metal works fine). Also wash all of the other components and dry with a lint free towel. From this point, avoid contacting any surface which will be exposed to vacuum with your bare hands.

First, put together the end assemblies consisting of the end flanges, gaskets, and Lexan plates. Note that the bevels on the Lexan plates are not symmetrical. The shorter bevel needs to be closer to the outside of the device. Tighten the six nuts enough to compress the gasket but avoid over tightening. Place a drop of Loctite on each nut.

A set of four nylon screws and spacers is used to hold the device together and compress the electrode O-rings. Assemble these to one of the Lexan plates as shown in Figure 5. If the source is to be oil filled, place O-rings on each of the screws as shown. The nylon hardware is manufactured by Jandorf and is readily available locally in hardware stores.

With one end assembly completed, begin the process of stacking the electrodes and insulators. It is important that the electrode holes be well aligned. A simple jig may be made with slightly undersized straight rod (e.g. piano wire) mounted perpendicular in a block of wood. With the flange centered over the rod, just slide the electrodes over the rod during the assembly process.

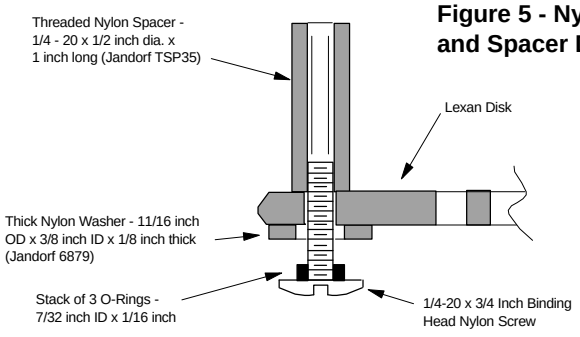


Figure 5 - Nylon Screw and Spacer Detail

The insulating plates will self align within the nylon spacers.

After the electrodes and spacers are located, place the other flange assembly on the stack with the screw holes aligned with the nylon spacers. Finally, insert three more nylon screws with their associated washers and O-rings and tighten everything just enough so that the electrode O-rings are compressed and the flanges are parallel.

Fabricate the hollow cathode as shown in Figure 1. The compression fitting could be eliminated to save a few dollars. However, eventually, you will want to modify this part of the source either to add a trigger wire (for higher vacuum/higher voltage applications) or to access the positive ion beam which will emerge from this end of the source.

Before filling with oil, test the device to ensure that the assembly is vacuum tight. The materials used in construction should allow you to easily pump to the 10^{-5} Torr range. However, typical operation will be at 25 to 75 milliTorr and will only require a halfway decent mechanical pump.

Operation of the source can be tested by discharging a small doorknob type capacitor (2000 pf at 20 to 40 kV) across the device. The capacitor should be located at the source with short wires connecting to the flanges. Operation can be verified by observing the effect of the electron beam on a phosphor screen.

In upcoming issues, use and applications for this source will be discussed in more detail. Also there will be information on a simple beam transport and focusing scheme based on the 'dielectric waveguide' principle. In the meantime, I would be happy to respond to any inquiries concerning this interesting type of electron/ion beam source.

This article was originally presented in Volume 3, Number 1.

Addendum: In the 10 years since this article was originally published I have made a number of these pseudospark MPC sources. I have abandoned the oil insulated design in favor of designs with larger diameter plates. Besides being simpler and easier to modify, the air insulated versions work well to the 100 kV range. The design shown in the next article is amenable to such scaling.

Something Different: Freeze-Drying

the Bell Jar has mostly been concerned with applications of vacuum in physics. However, there are a great many other areas that involve vacuum technology. The freeze-drying of biological materials is one of these. Preservation of the freshness of coffee and making astronaut food area couple of the well known examples of freeze-drying. Freeze-drying is also used in the manufacture of pharmaceuticals (an area that I occasionally get involved in) and for the preservation of animal specimens. Museums now use freeze-drying as an alternative to taxidermy and a couple of companies will sell you freeze-dried rattlesnakes or even freeze-dry your dear departed pet so you can keep it curled up on the sofa in perpetuity (and never have to feed it or take it for a walk). About a year ago, T.J. Lindsay (Lindsay Publications, Inc.) brought to my attention a book authored by Rolland Hower of the Smithsonian Institution that covers the processes and equipment for the freeze-drying of biological specimens ranging from organs to complete animals. Basically, freeze-drying (or lyophilization) just consists of freezing the specimen and then drawing off the water under conditions where the ice sublimates (i.e. turns directly from solid ice to vapor). By avoiding melting, the form of the specimen is preserved in a stable condition. Sublimation requires a vacuum better than about 1 Torr as well as gas-ballasted pumps, often with refrigerated condensers, to handle the water vapor that is drawn off. Hower's book, *Freeze-Drying Biological Specimens: A Laboratory Manual* (Smithsonian Institution, 1979), describes the freeze-drying process, practical apparatus of varying complexity (including one based on a chest-type home freezer), photos of preserved specimens, representative process cycles (20 hours for a praying mantis, 7 days for a small snake, 9 months for an alligator) and descriptions of related processes including the preservation of documents and wooden marine archeological materials. Lindsay doesn't carry this book but you can order it through your local bookseller. The price is about $35 in hardcover.

Compression Plates for Stacked Electrode Assemblies

I. INTRODUCTION

A variety of interesting pieces of apparatus may be assembled from coaxial planar stacks of metal apertures and/or grids separated by suitable dielectrics. Examples would include dc accelerator columns, energy and mass analyzers, electron guns, gaseous and metal vapor ion sources, and so forth.

The MIT and Penn State electron optics kits described on page 3-87 are examples of a flexible approach to particle optics studies. As noted, the Kimball Physics *eV Parts* components provide a relatively easy way to fabricate complex research quality UHV compatible electron optics with a standard set of parts. All of these approaches create structures that are flange mounted for insertion into a vacuum chamber.

At the other end of the spectrum are the stacked and brazed or glued structures that have been used to make potential drop particle accelerators. Amateur built accelerators have used brass disk electrodes with intervening glass tubes which serve as segmented insulating columns as well as the walls of the vacuum chamber. Research accelerators have used ceramic tubes and, more recently, polyethylene disks as the insulating structures. In most cases, the seals between the electrodes and insulators have been made with epoxy cement. While this makes for a simple structure, the use of adhesive seals makes changes, even minor ones, very difficult (or impossible) to accomplish.

The O-ring based approach to stacked structures as shown in the article on the multiplate electron and ion source can be generalized to encompass other structures. This article will give details on a simple set of plates between which such structures may be placed.

II. COMPRESSION PLATES

The compression plates are fabricated as shown in Figure 1. I cut the plates from ½" Lexan sheet using a 3-¾" hole saw. This produced a disk about 3-5/8" in diameter. As the edge was a bit ragged, I turned the disk down to 3-½" with a lathe.

The central pedestal represents a totally perverse use of a 1.33" fixed Conflat flange. My only excuse is that it works quite nicely and the flanges are fairly cheap. The central bore is 5/8" diameter and is compatible with ½" copper water tube. The stub provides extra support to the tube and has a slight step in the end which perfectly matches the id of a standard 5/8" brass washer. To assemble the metal parts, clamp the water tube gently in a vise such that, when the flange is placed over the tube, the end of the tube is flush with the washer's upper face. With all mating surfaces cleaned and fluxed, solder with 2 to 4% silver-tin alloy.

As the flange is designed to be flush with the 5/8" compression fitting on the manifold, it is important that the heads of the 8-32 screws be recessed. Three equally spaced holes on a 2-½" circle hold the tie-rods. I would recommend that one of the plates be drilled and tapped

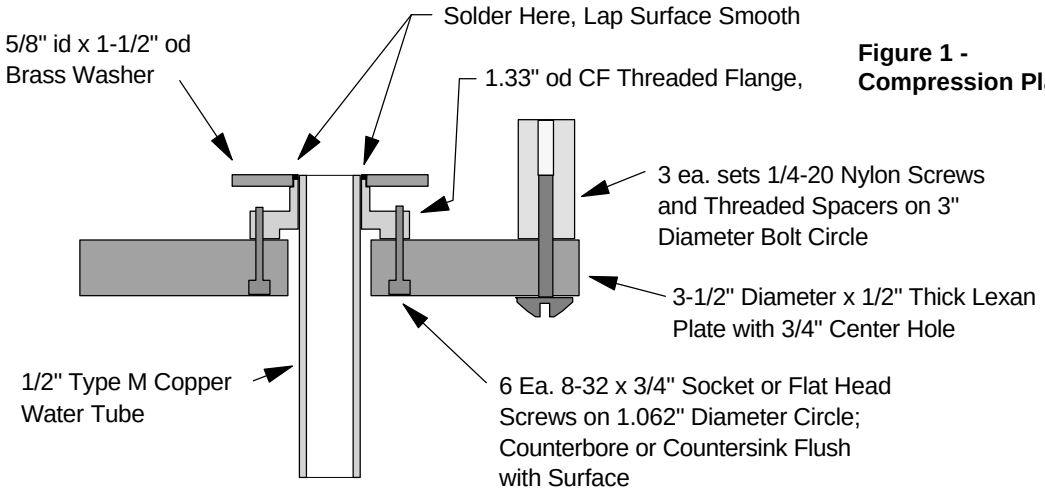


Figure 1 - Compression Plate

for a ¼-20 thread. The other should be drilled to clear a ¼" thread.

For low voltage applications, the tie rods can be made with stainless steel or brass threaded hardware. Where insulation is important, nylon screws and threaded spacers (Jandorf brand, available in hardware stores) are quite satisfactory.

III. A MPC BEAM SOURCE

Figure 2 shows the now familiar MPC electron and ion beam source adapted for use with the compression plates.

This particular version was operated at 10 kV with a 2000 nF capacitor. A well defined electron beam was produced at about 100 mTorr and the discharge ceased at 70 mTorr. As my high voltage supply was ripped apart for modifications, I have not yet had a chance to test it at higher pulse voltages. I believe that this source should be able to stand off voltages in excess of 50 kV without arcing.

IV. OTHER CONFIGURATIONS

As the MPC source uses floating intermediate electrodes, the surrounding O-rings do not present a problem. However, in most cases it is necessary to make electrical connections to the electrodes. Figure 3 shows several ideas for such structures.

Figure 3A shows a segment of an electrode stack for a high voltage electron gun. The insulating disk is high vacuum compatible polyethylene and the electrodes are fabricated from brass QF center rings, and brass and stainless steel washers. The plastic disk is cut from a sheet of material using a hole saw. The center rings have their flanges partially removed from one side and totally removed from the other side. It is important to have the aperture considerably smaller than the insulator diameter to prevent the beam from hitting the insulator and building up charges.

Figure 3B shows a gridded structure built up with 3/32" Teflon rings. Teflon in this thickness is not too expensive, has good electrical and vacuum properties and works well with 1/8" section O-rings.

Plastics such as UHMW polyethylene and Teflon are available in a variety of shapes and sizes from United States Plastic Corp., 1390 Neubrecht Rd., Lima, OH 45801, (800) 537-9724.

Figure 3C shows an accelerating column made from copper fittings and 38 mm od Pyrex tubing. This column is similar in geometry to the original particle accelerator of Cockcroft and Walton. QF25 center rings are used at each end for mating with the compression plates and/or other electrodes. The tubes are glued to the copper fittings with epoxy.

Given the vagaries of O-rings, this approach has its limitations. However, the simplicity and flexibility should permit many interesting experiments to be conducted.

This article was originally presented in Volume 3, Number 4.

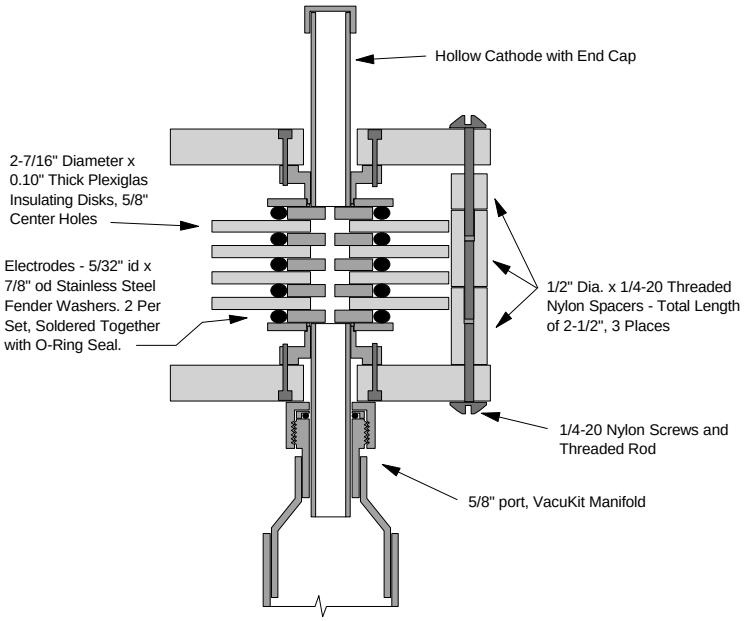


Figure 2 - Compression Plates Configured as a Multiplate Chamber Electron Source

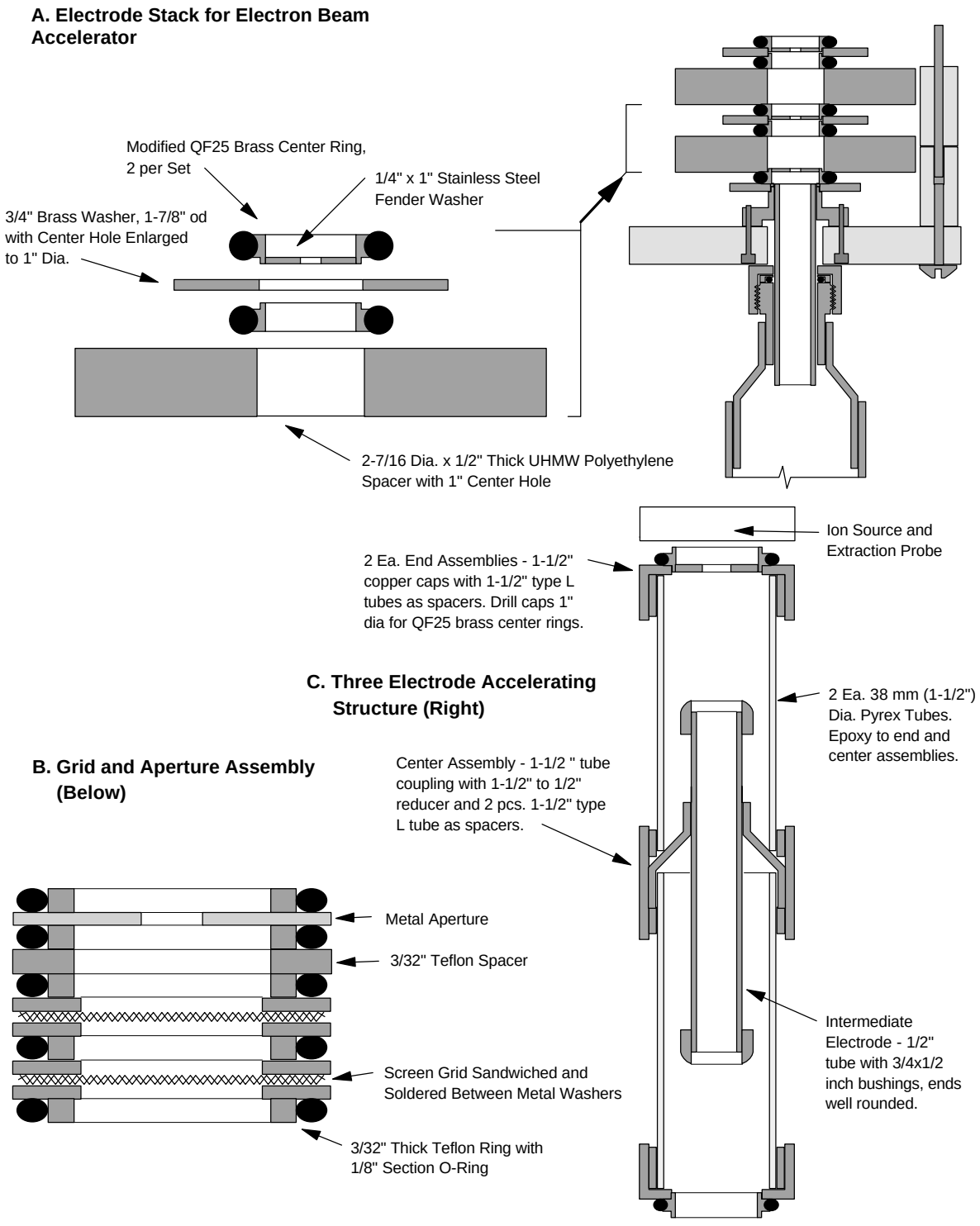


Figure 3 - Ideas for Various Electrode Structures

Electron Optics Kits

An inquiry by subscriber Jack Sieber concerning some kit form vacuum tubes that were available some time ago (1950s per Jack's recollection) has generated some interesting correspondence from the readership.

Dr. Bruce Kendall of State College, PA provided some information with regard to vacuum tube/electron optics kits that were in use in that general timeframe. Bruce writes "Jack may be referring to the 'Harries Physikit Vacuum Tube Set' in use at MIT and possibly other universities in the late 1950's. These kits allowed construction of operating glass vacuum tubes with joints made by 'solder glass'. The kits were apparently developed by J. H. Owen Harries, a consulting engineer in Bermuda who had a background in commercial vacuum tube design.

"I remember seeing an example of the Physikit in Canada around 1960. At about the same time, Owen Harries published a description in "American Journal of Physics." Unfortunately, his planned large-scale production never eventuated. His work did, however, show what could be done with relatively simple equipment.

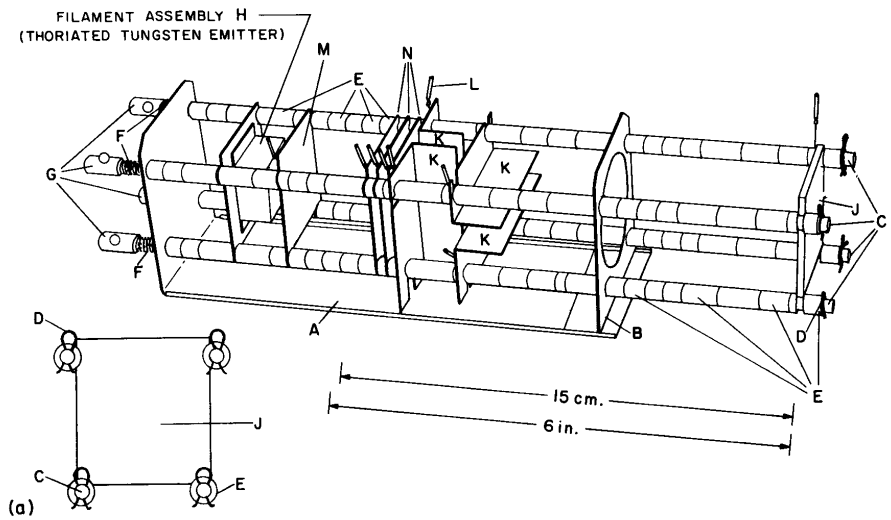
"Around 1964 we tried unsuccessfully to obtain several Physikits for use in laboratory classes at The Pennsylvania State University. After reviewing various alternatives, including the later and different kit of parts also developed at MIT, I became involved in developing our own system based on reusable, planar, interchangeable electrodes. With the aid of Holger Luther (then a graduate student) and Don David (instrument technician), two kits were developed with an associated vacuum system for continuous pumping. The 'elementary' kits could be used to make working thermionic diodes, triodes, pentodes, electron guns and a cathode ray tube. The 'advanced' kit extended this to include working ion guns, electrostatic lenses of various types, ionization gauges, and electron multiplier, and a mass spectrometer.

"After this equipment won an award from the American Association of Physics Teachers and was exhibited in New York in 1966, we were approached by various prospective vendors regarding commercial production. Don David left Penn State and began manufacture of the kits which were sold for several years by the Ealing Corporation, based in the Boston area. My impression is that most of them were bought by colleges in the Northeast. Some users operated them in bell jars, instead of their original housings which were based on food processing components. During this period extensive jigs and tooling were constructed in an attempt to reduce production costs.

"Eventually Don's business expanded and diversified and kit production ceased. The jigs and other tooling were discarded and after a long period of storage passed into my hands so that occasional requests for spare parts could be filled."

A drawing of this kit, assembled as a CRT, is shown in the figure below. Some relevant references include the following:

- J.H.O. Harries, American Journal of Physics, **28**, 698 (1960).
- C.K. Crawford, Review of Scientific Instruments, **36**, 844 (1965). Describes the MIT kit.



Electron Optics Kit
The main features include the main frame A, ceramic rods C, retaining clips D, ceramic spacers E, compression springs F & clamps G. Electrons are generated at H, focused by elements M, N, and are deflected by plates K. The beam is detected by phosphor plate J. (a) shows the self-aligning feature. Reprinted from *Experimental Vacuum Science and Technology,* p.196, by courtesy of Marcel Dekker, Inc.

- B.R.F. Kendall, H.M. Luther, American Journal of Physics, **34**, 580 (1966). This article, *Apparatus for Teaching and Research in Electron Physics*, discusses the design of the Penn State kit in detail.
- B.R.F. Kendall, Journal of Vacuum Science and Technology, **5**, 45 (1968). Describes a simple pumping system for the optics kit.
- B.R.F. Kendall, H.M. Luther, D.R. David, American Journal of Physics, **37** (1969). This article, *Apparatus for Studying the Principles of Electron Physics*, describes the integrated pumping system and electron optics kit for student use.
- B.R.F. Kendall and H.M. Luther, *Construction and Use of a Cathode Ray Tube*, in "Experimental Vacuum Science and Technology" (Marcel Dekker, NY, 1973) This book, edited by the American Vacuum Society Education Committee, contains a wealth of useful information and experiments. This particular chapter describes a project using the Penn State kit.

The only contemporary equivalent of this kit that I am aware of is a set of parts which one can obtain from Kimball Physics, Inc., a manufacturer of electron and ion optical systems. Known as 'eV Parts', three kits of varying complexity are available as well as smaller assortments of components. All parts are UHV compatible and consist of plates, cylinders, mounting rods, insulating spacers, brackets, phosphor screens, springs, grids, etc. From these can be made ion and electron sources, Knudsen cells, electrostatic lenses, LEED equipment, Faraday cages, mass spectrometers, scanning microscopes, etc. Prices for the kits range from nearly $1000 to over $4000. For those with deeper pockets than mine, Kimball Physics can be reached at Kimball Hill Rd., Wilton, NH 03086.

This article was originally presented in Volume 3, Number 2.

Build a Plasma Sphere

Steve Hansen

I. INTRODUCTION

"Plasma Spheres" are the little globes that are seen in Radio Shack, science museums and other places. A single electrode in the center of the glass sphere is connected to a source of high voltage, high frequency (important!) current. The gas mixture and pressure in the sphere are adjusted to a set of conditions where delicate tendrils of electrical "flame" will jump from the sphere to the globe. Voltage, frequency and waveform will also have an influence on the discharge. Touching the globe with the fingers or hands will modify the appearance.

Like the store-bought radiometers, store-bought plasma spheres don't allow for any tinkering. While it may be difficult for the average amateur to precisely duplicate the visual effects in a professionally built globe, a number of interesting experiments may be conducted. Of course, the fact that you "built it yourself" is significant. The pressures used in these globes is typically well over 1 Torr (usually close to 1 atmosphere) so a modified air-conditioner compressor may be used for these experiments.

The major components, as shown in Figure 1, are several plumbing components, a 7 inch length of 11 mm Ace chromatographic tubing with a Nylon bushing, a piece of brass tubing and a modified brass hose barb. The globe itself is made from a 1 liter Pyrex flask blank. In addition to these components you will need a small quantity of two-part white epoxy cement, soft solder and flux (2 to 4% silver - tin "hobby" solder is preferred) and a suitable solvent (e.g. denatured alcohol).

The Pyrex glass insulating tube must be able to pass through the copper coupling. Use a small round file or rotary burr to remove the stops from the coupling until the glass tube just slips through the coupling.

Clean the copper fittings and the glass tube with solvent and let dry. Avoid touching the parts during the gluing process. Insert the glass tube into the coupling. Withdraw the tube about 1 inch and apply a light coat of epoxy to the tube. Insert the tube as far as it will go into the coupling, rotating slightly to spread the glue. Arrange a support to hold this assembly upside down while the epoxy cures. Make sure that the coupling is level and the tube hangs plumb. Let the epoxy cure overnight.

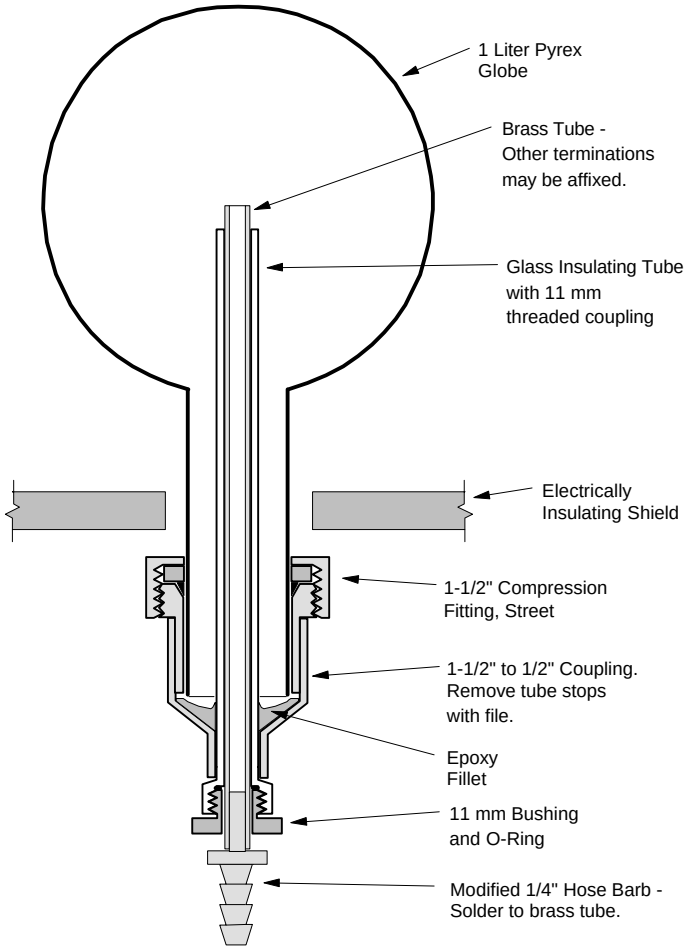


Figure 1 - Plasma Sphere Assembly

From the wide end of the coupling, apply a liberal amount of epoxy, filling the conical metal portion of the coupling. Avoid getting epoxy on the upper part of the glass tube and try as much as possible to work air bubbles out of the glue. Then smear a thin coat of epoxy around the inside of the coupling mating surface and insert the body of the compression fitting into the coupling. Rotate the fitting to evenly distribute the epoxy. Excess epoxy will ooze out of the bottom of the coupling. Should any squeeze into the coupling's threads, immediately clean the threads with a paper towel dampened with solvent. Support the assembly in an upright position and let the epoxy cure.

The hose barb is made from a 1/4" x 1/8" male pipe-thread brass barb. This is available from hardware or plumbing supply stores. Turn or file the threaded portion so that it makes a tight fit in a piece of 13/32" od brass tubing (K&S Engineering). Solder the barb into one end of the brass tube and clean with an abrasive pad (e.g. 3M metal polishing abrasive pad) and soapy water. Rinse with clean water and let dry.

II. FINAL ASSEMBLY AND NOTES ON OPERATION

The 11 mm fitting on the glass tube permits the position of the brass tube to be adjusted. For initial tests, position the brass tube so that about 1/2 inch extends beyond the end of the glass tube, into the globe. The globe seals to the compression fitting with the brass compression nut, a hard rubber compression ring and a Buna-N O-ring, in that order. A gentle final tightening with a wrench is advisable.

For experiments at moderate vacuum, the globe may be evacuated through a length of standard 1/4" id clear PVC tubing, available locally. DO NOT USE AUTOMOTIVE RUBBER TUBING: this tubing is usually electrically conducting. If the globe is operated

while the pump is connected, a portion of the discharge may follow the tubing to the pump. This parallel discharge will diminish the intensity of the discharge within the globe. This may be alleviated by pinching the tube with a clamp after the globe has been evacuated. As this is a constricted discharge, by making the tube fairly long (i.e. over about 3 feet in length) there will be no conduction through the tube. Under high vacuum conditions, very little separation is required.

In order to confine discharges to the end of the brass electrode, the compression fitting must be electrically connected to the brass tube. Since these areas are electrically "hot," an insulating shield should be built to surround the area below the bulb.

Only high frequency, low current power supplies should be used with the globe. Unrectified TV flyback based supplies (see Figure 2) and small Tesla coils are suitable.

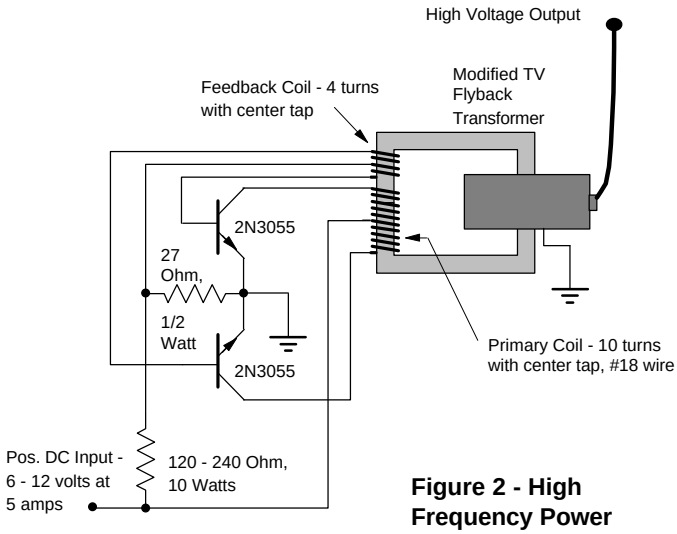


Figure 2 - High Frequency Power Supply

A small collection of plastic tubing, tees and pinchclamps may be used to introduce other gases to the globe. Small quantities of helium may be drawn from party balloons. Balloons may also be used to capture gases commonly used in welding shops (oxygen, argon). **Never introduce explosive gas mixtures into the bulb.**

Other electrodes of various materials and shapes may be affixed to the brass tube. Brass or zinc alloy drawer pulls may be useful. Since the electrode is also

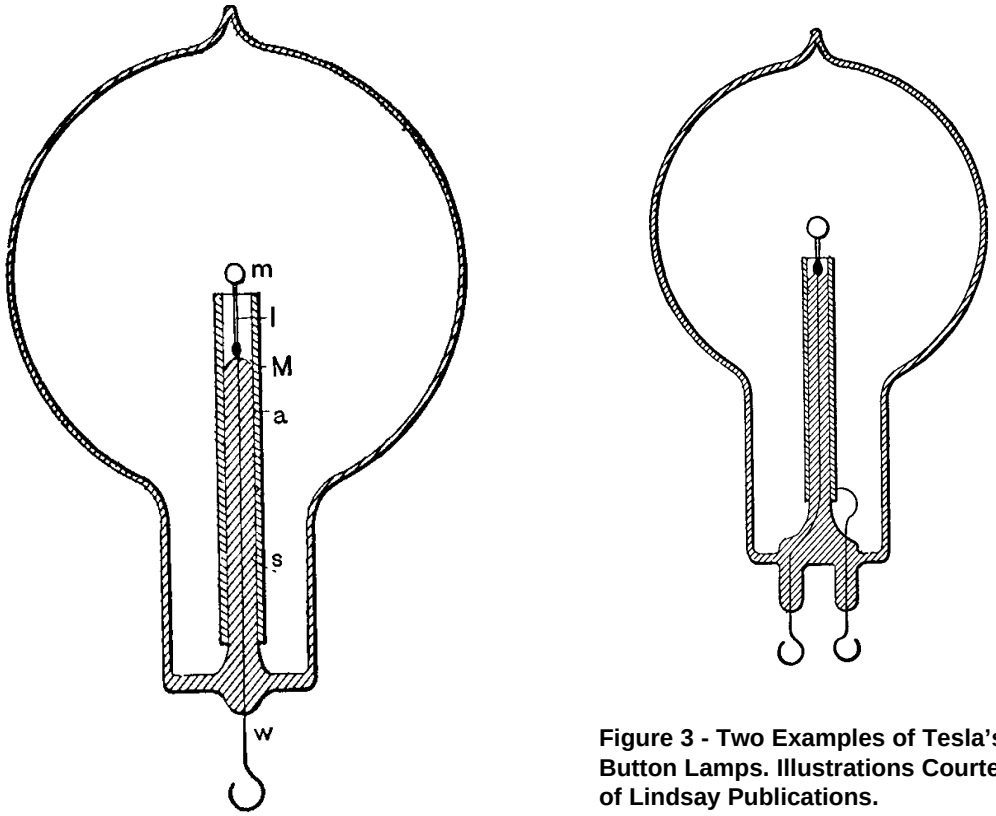


Figure 3 - Two Examples of Tesla's Button Lamps. Illustrations Courtesy of Lindsay Publications.

the evacuation line, take pains to ensure that the passageway is not constricted by alternate discharge electrodes.

III. FURTHER READING

Those who are interested in experiments at high vacuum along the lines of the Tesla 'button lamp' should begin with Tesla's descriptions as contained in "Tesla's Experiments with Alternate Currents of High Potential & High Frequency" as reprinted by Lindsay Publications, Inc., Bradley, IL 60915. This material is also contained in another Lindsay reprint, "The Inventions, Researches and Writings of Nikola Tesla" as compiled and edited by Thomas Comerford Martin. However, the illustrations are more clearly delineated in the former book. Figure 3 shows a couple of Tesla's lamps. Tesla was very unclear as to the level of vacuum that he could achieve in these globes with his modified Sprengel pump. However, based on Crookes' experiments with similar pumps, it is quite likely that Tesla was working in the high vacuum range.

Gordon McComb's "Gadgeteer's Goldmine" (TAB Books, Blue Ridge Summit, PA 17294) describes some experiments with a simple plasma sphere. The magazine *Electronics Now* (formerly *Radio-Electronics*) has had a number of articles on plasma spheres and associated power supplies.

This article was originally presented in Volume 4, Number 2.

Some Notes on Atmospheric Pressure Plasma Displays

Dr. Ed Harris of Ohio State posted a note on the net concerning his experiments with atmospheric pressure discharges. This led to several correspondences and the following is a compilation of the various communications that have occurred over the past few months concerning this work.

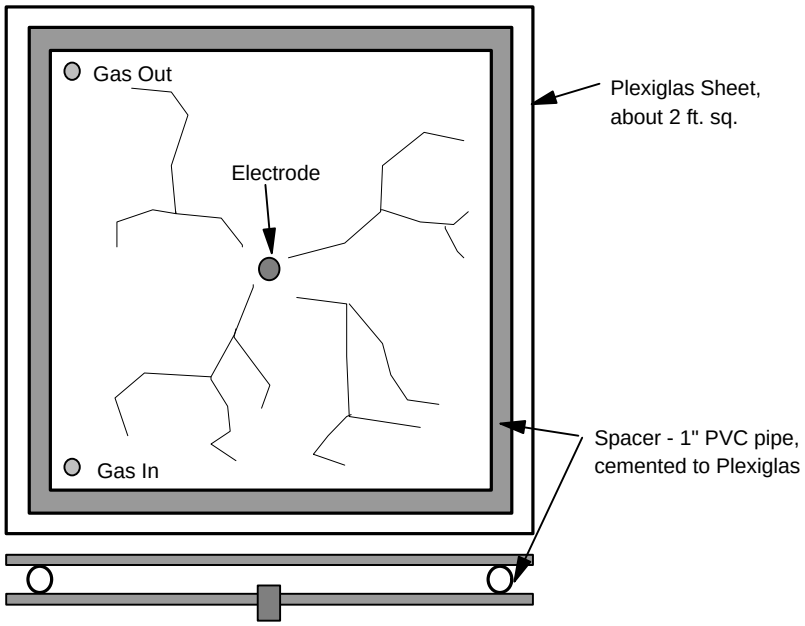
"There was, for a while, some interest on the USA-TESLA mailing list in how plasma globes are built. They generally consist of a sphere of glass through which there is placed a single electrode (mine is a 1/4" copper pipe with a steel ball bearing soldered on top). And some means of pumping out and sealing off the globe. Many people including me have made them by pumping out a glass jar, sphere or flask to a vacuum level of ~1mm Hg or 1/750 of an atmosphere. Some use other gases than air like neon (expensive) or argon but at similar pressures (1/100-1/10000 atm). This works pretty well if you've got a vacuum pump and are not afraid of an implosion. In this form, the "plasma globe" is rather similar to Tesla's own carbon button lamp, although he generally ran much more current into them - often with the effect of heating the discharge terminal to incandescence due to energetic ion bombardment.

"Then someone (can't recall the name) said he had heard that commercial plasma globes were made to work at or near normal atmospheric pressure. Well, with air alone this obviously can't be done with the voltage levels available - 10 kV or so from a flyback oscillator (like in a TV set). But this seemed possible to me if you keep the same partial pressure of air or other gas (1/1000 atm) and fill the rest of the space with helium gas. This is a trick used by gas laser people for 30 years to raise the working pressure of electrical discharge gas lasers (See Scientific American *Amateur Scientist* from 1970-1972 on the N_2 gas laser).

"So, I tried it. It works quite well. You can easily obtain 5" arcs inside a glass globe with only 5 kV or so from the unrectified output of a flyback supply using atmospheric pressure helium and 1/100-1/10000 partial pressure of air or other "dopant" gas. It is the dopant gas which is generally doing most of the glowing. As a natural extension of this I believe it was Bill Beatty (sorry if I got the spelling wrong) who suggested making plasma "displays" from plastic bags filled with helium. I tried using a rubber balloon instead, and found pretty good results if the balloon was well purged with helium to get out most of the air. Again 5" arcs were obtained inside the balloon with under 10kV.

"Interesting semi-stationary forked structures similar to those seen with high power vacuum tube Telsa coils can be seen with much lower voltage levels. I see many different types of discharges depending on voltage and pressure. I still want to get a nice big plastic bag as Bill suggested, but the purging could be much more difficult.

"More recently I have gotten much better results in the sense that they are more visually striking by using argon and argon/helium mixtures. Mixtures containing almost all helium are rather dull red/purple/orange. Argon discharges, on the other hand, are rather brilliant white/blue. Argon works well alone at reduced pressures (say 10" Hg or less) and produces eye-of-the storm like discharges. Helium can be added to raise the working pressure up to atmospheric with some detriment to the argon discharge which can be overcome to an extent by using a higher input power. There is certainly a large parameter space to cover!"



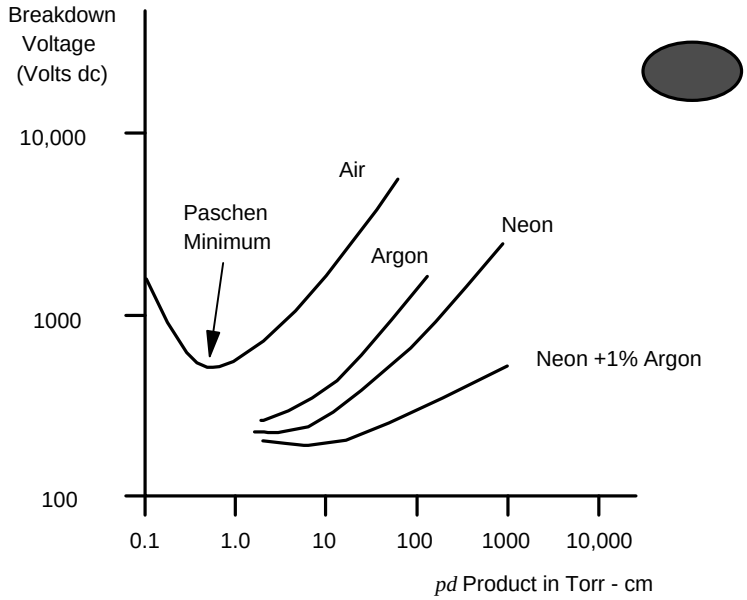
Ed Harris' Atmospheric Pressure Plasma Chamber

At about this point (late August), Ed sent me a video tape showing a larger apparatus. A sketch of his setup is shown in the figure above. Power is provided by a flyback supply capable of delivering up to about 30 kV. Gas flow is set to a level where the gas can be felt when the hand is placed at the outlet port. Regarding the geometry of the device, a wide/thin discharge space:

"As to the question of surface conduction along the plastic, I suppose that surface conduction effects across the plastic could contribute somewhat to the length of the discharge, but I've also done some experiments with long glass and quartz tubes with similar length discharges (actually longer). So from limited evidence, I'd guess that surface conduction isn't noticeably helping to lengthen the discharge.

"I have noticed something which seems to make sense, however. Confining a discharge allows it to be longer. If the discharge is constrained to a tube as opposed to a volume or 2-d plane, it tends to be longer for a given amount of power. This kind of makes sense to me, since the power needed to excite the gas should grow as the volume of the gas excited (or something like that)."

At this point it probably makes sense to talk a bit about the relationship between breakdown voltage, gap length, pressure and gas type. The relationship between all of these is shown in something called the Paschen curve. If sparking voltage for a particular gas is plotted against the pressure (e.g. in Torr) times the gap distance (e.g. in cm) a curve will be developed as is seen in the chart at the right. (The value on the x axis is also called the *pd* product.) The interesting thing is that there is a minimum voltage at a certain *pd* value. This minimum is the *Paschen Minimum*. At either side of the minimum the breakdown voltage is seen to rise. Needless to say, this relationship does not hold at all pressures but it does work through the region where glow discharges exist up through atmospheric pressure. Most glow discharge devices, like neon signs and plasma reactors, work on the right-hand side of the minimum. Other devices, where it is desired to be able to hold off high voltages in environments where glow discharges are easily formed, are designed to work on the left-hand side of the curve. High voltage feedthroughs into sputtering chambers sometimes exploit this effect.



Curves for Sparking Potential

With regard to gas type, different curves exist for each type of gas. As can be seen from the chart, a given voltage will support a longer arc in argon at a given pressure. And, neon with a trace of argon will support a very long arc under the same conditions.

Ed's device operates in the region noted by the circle at the upper right. This appears to be a stretch for argon, but it also must be remembered that his device works with an excitation source that operates at a few 10s of kHz. This imparts more energy to the gas, giving a longer length than would a dc discharge. This effect will have been noticed by anyone who has worked with a Tesla coil.

In working with helium, tendrils are seen that look very much like the arcs in a commercial plasma globe. Currently, Ed is experimenting with discharges in a long glass tube and has been able to produce 2 foot discharges in a 1-1/2 inch tube using a conventional 60 Hz ac neon sign transformer.

For more on the Paschen curve, I'd suggest von Engel's book (*Ionized Gases*, now available through the American Institute of Physics) which has been frequently mentioned in these pages.

Also, I'd like to mention a new book, *Industrial Plasma Engineering* by J. Reece Roth (Institute of Physics Publishing, Philadelphia, 1995). Besides being up to date, Roth, who is a professor at the University of Tennessee at Knoxville, touches (in a mostly understandable way) on just about every topic and device using plasmas. He has also done a fair amount of work on atmospheric pressure plasma reactors and the operating parameters are discussed in some detail in the book. He notes the importance of frequency on the form of the discharge, finding that at either side of an optimum frequency (where a stable glow discharge exists) the discharge breaks into a filamentary form. This book is available in paperback for about $40 and is recommended reading for anyone who is tinkering around with plasma discharges. A second volume is due in a year or so.

This article was originally presented in Volume 4, Number 4.

Infrasound Monitoring with a Microbarograph

How to detect low frequency acoustic waves in the atmosphere

Steve Hansen

This article is a bit off-topic in that it doesn't deal specifically with vacuum. However, I think that this subject will be of interest to many amateurs in addition to providing an example of another application involving the measurement of very low pressure differentials. - Ed.

I. INTRODUCTION

A host of pressure disturbances are created in and are propagated through the environment of the earth. Seismic activity is one of the better known and a significant number of amateurs are engaged in monitoring the waves produced by earthquakes and volcanic eruptions.

At sites that are good for seismic observing even relatively small earthquakes can be registered throughout the world by fairly simple apparatus. However, it is found that it is only the lower frequency components of the earthquake wave that propagate to the greater distances. This is because absorption decreases with decreasing frequency and the dominant losses at low frequencies are geometric. Most wave phenomena share this characteristic [1].

Just as the low frequency components of seismic waves spread through the earth from their points of origin, so can acoustic pressure waves travel long distances through the atmosphere. These far-travelling disturbances occupy the frequency range below that of human hearing and are therefore termed infrasound. Horizontal propagation of these waves occurs in sound ducts created by temperature gradients and is modified by the winds of the upper atmosphere with winds at altitudes of 40 to 60 km having the greatest influence [2].

Infrasound can be generated by natural causes such as severe weather, frontal passages, meteoric fireballs, the aurora, mountain associated waves and avalanches. Human activity also can generate infrasound. Examples include aircraft, large machinery and explosions. Taken together, these have been termed the "atmospheric wave zoo."

While somewhat differing definitions exist, we will consider the infrasound spectrum to extend from about 0.001 Hz to a few Hz. At higher frequencies, propagation distance is limited and there is considerable noise from the din of the local environment. Below this frequency range we have the long timescale, large amplitude atmospheric pressure variations that are due to normal meteorological processes. This is the effect that one observes on a standard barometer.

So, where do these infrasonic signals fit into scientific research? Much of the activity on infrasound began during the Cold War. Nuclear detonations generate infrasonic signatures and arrays of detectors can assess the yield and location of an explosion. Today there are plans to implement a worldwide network of detectors that will be used in conjunction with seismic sensors to test compliance to the Comprehensive Nuclear Test Ban Treaty. Activities of a less political nature include the study of the origins and propagation of acoustic gravity waves (longitudinal pressure waves in the atmosphere, not to be confused with the elusive gravity waves predicted by general relativity), the detection of large meteor-fireballs (bolides) that intersect with the earth, and research into the coupling between the upper and lower parts of our atmosphere.

For the amateur, there are several areas that may be worth pursuing. For example, it might be interesting to include an infrasound observation capability with a seismology program. A challenge would be to try to correlate the passage of acoustic gravity waves with reported meteorological conditions. An observing program might also involve studying possible relationships between acoustic waves in the lower atmosphere and internal gravity waves in the ionosphere. The latter are transverse waves that may be detected by observing the propagation of low to medium frequency radio waves [3]. Both can be produced by disturbances in the atmosphere. Infrasound from the explosive disintegration of bolides could be verified by checking a database of visually observed fireballs such as the one maintained by the International Meteor Organization's Fireball Data Center (FIDAC) [4]. Several interesting papers on meteor infrasound have been published [5, 6, 7]. A number of topics are discussed in a recent broad review of infrasound [8].

II. MICROBAROGRAPHS

The acoustic pressure sensors that are used to detect these low level, low frequency waves are called microbarographs. Basically, a microbarograph is

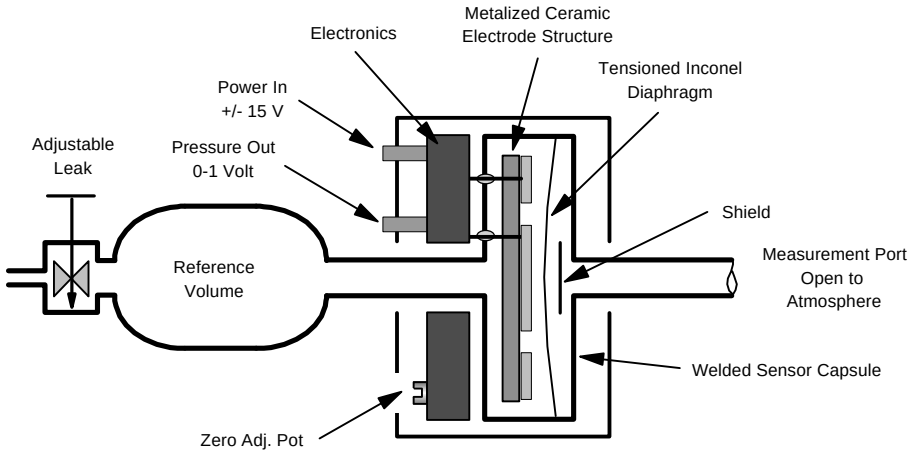


Figure 1 - Differential Capacitance Diaphragm Gauge Configured as a Microbarograph

nothing more than a microphone that has been optimized for high sensitivity at very low frequencies. These sensors also have to be designed to minimize pressure effects that are not of interest.

Seismic sensors have an advantage in that they get bolted to solid earth and they only move when a disturbance hits them. The microbarograph has to detect very low pressure fluctuations of periods ranging from a fraction of a second to minutes that ride on top of the constantly changing absolute pressure (the barometric pressure) of the atmosphere. Barometric pressure variations tend to have much longer periods than those of the infrasound signals so this effect may be removed by incorporating a high pass filter element into the microbarograph.

This is done by using a sensitive differential pressure sensor with one side of the sensor pretty much open to the atmosphere and the other side connected to a finite and stable volume which is connected to the atmosphere by means of a low conductance element: a leak. Thus, while one side of the sensor can respond quickly, the other has a “leaky volume” that gives the device a high pass characteristic. The sizing of the leak and the reference volume will determine the time constant of the device, hence the lower limit of the frequency response. Figure 1 shows the general configuration.

With atmospheric pressure variations out of the way, the next step is to tune the instrument to the area of interest. Infrasound occupies a broad range of frequencies and pressure levels and, as luck would have it, no one instrument will adequately cover the entire spectrum. Dividing the spectrum into two bands is typical. A higher passband, say from about 0.05 to 10 Hz is useful for detecting close and relatively small events. This is the range that is used by the defense establishment to listen for low yield atmospheric nuclear weapons testing. A lower passband, from about 0.003 to 0.03 Hz is generally used for monitoring natural infrasound emissions and acoustic gravity waves. In either case, sensitivities are typically on the order of 0.1 to 1.0 microbar and events may have amplitudes to 10s of microbars. (A microbar is defined as a pressure of 1 dyne per cm^2 and is equivalent to 0.75 milliTorr or 0.75 micron Hg.)

Verification and localization of events is essential in serious work and this requires a network of microbarographs. The network might consist of several microbarographs located on a grid covering a few square kilometers or it might be a complex global network. For any amateur who is familiar with seismology, this will all sound familiar.

For the detection of acoustic gravity waves resulting from distant disruptive events, the specifications of the sensors of a French microbarograph network [9] are instructive. The passband is 0.003 to 0.04 Hz (periods of 333 sec to 26 sec) with a sensitivity of 1 microbar. With instruments deployed in such locations as the Ivory Coast, French Polynesia and, of course, France, this network recorded and localized the explosion of Mount St. Helens from distances as far as 11,500 km. Peak amplitudes were on the order of +/- 50 microbar.

III. EQUIPMENT

A number of sensor technologies have been used for microbarographs. Professional instruments typically use modified high quality capacitance microphones. These have sensors that determine pressure changes by measuring the displacement of a diaphragm by electrical means. However, with price tags in the multikilobuck range, this was not an option for me.

When undertaking this project I looked at two specific types of differential transducer: solid state pressure sensors as exemplified by Honeywell Micro Switch's 163PC01D36 (±3.5 in H_2O [about 6.5 Torr] range), and capacitance diaphragm gauges (CDGs) as exemplified by MKS Instruments' Model 223 Baratron® (±200 milliTorr range). The former costs about $100, the latter about $465.

I found the Honeywell device to have an electronic noise level roughly equivalent to 10 milliTorr peak to peak whereas the CDG's noise was not detectable. Also, the solid state device tended to have a rather high zero drift with nominal ambient temperature changes, nearly 100x the CDG's drift. Finally, the CDG has an accuracy of 0.5% of full scale and a resolution of 0.02 milliTorr, parameters that are not specified for the solid state sensor.

Given the sensitivity and stability differences, plus being a long time vacuum enthusiast, the CDG seemed to be a natural route. Normally used on process equipment, these work something like capacitance microphones but would be lousy for normal audio applications because their response drops off above about 10 Hz. No problem for this application.

This is not to say that the Honeywell device wouldn't produce some interesting results. A few weeks ago I got a call from a Chicagoan who detected the sonic boom of the shuttle entering the atmosphere using such a sensor.

The diagram of Figure 1 shows how the CDG is integrated into the microbarograph. For details on the principles of operation of CDGs please refer to the article on manometers in Vol. 5, No. 3.

As noted before, in the microbarograph configuration the reference port of the CDG is connected to a reference volume. That volume, in turn, is connected to the atmosphere. With the leak adjusted properly, long, slow variations in atmospheric pressure will affect both sides of the CDG's diaphragm equally. Shorter period variations will upset the equilibrium since the reference volume will not respond quickly enough to prevent a differential pressure from developing across the diaphragm. Thus, the time constant that is represented by the interplay between the size of the reference volume and the conductance of the leak is what sets the low frequency response of the microbarograph.

Since we are dealing with very low differential pressures, temperature effects will have a significant effect on the instrument. A very slight warming of the reference volume will cause the air to expand,

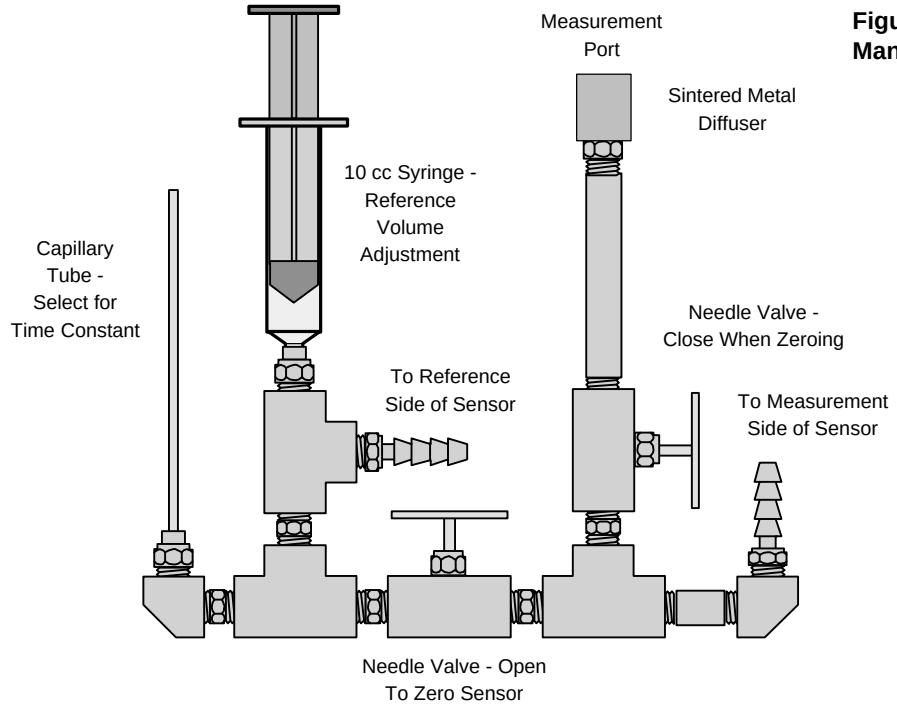


Figure 2 - Microbarograph Manifold Assembly

increasing the pressure and leading to a false pressure indication. In a practical installation, the entire assembly must be housed in an isothermal container where short term temperature variations are minimal.

My implementation is shown in Figure 2. The CDG is connected to this manifold by short lengths of plastic tubing. The manifold is constructed from 1/8-inch brass plumbing fittings. The reference volume consists of this manifold, the reference side of the CDG and the connecting tubing. To permit the volume to be varied, I added a plastic syringe. Once I get the device tuned properly, this will be replaced with a fixed volume made from plastic pipe and fittings. Two cheap brass needle valves (also from the hardware store) are incorporated to allow the CDG to be zeroed without having to disconnect it from the manifold. Whenever adjustments are to be made in the plumbing, the valve between the CDG's ports should be open to prevent overpressure conditions from developing across the diaphragm. (More on this below.) A sintered metal diffuser of the type used on air tools is used to keep dirt and dust out of the manifold and CDG.

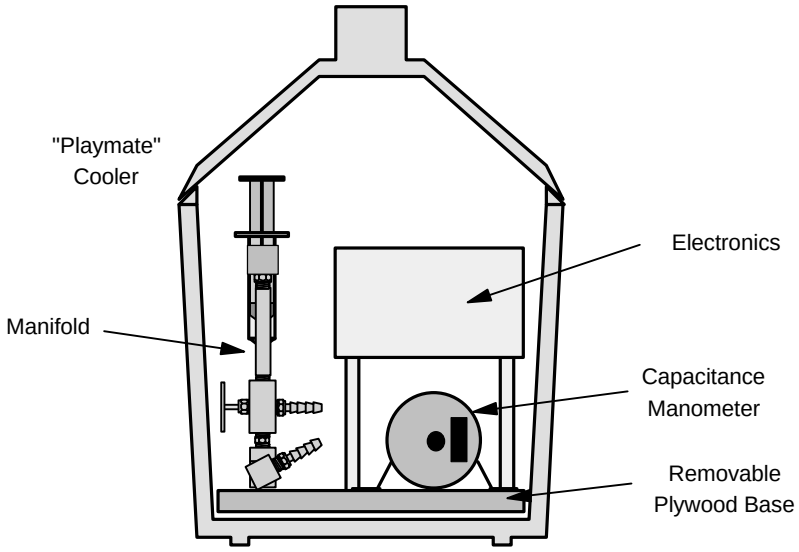
One option for the leak would be another needle valve. However, I found it more convenient to use hypodermic tubing (available in 6-inch lengths from Small Parts Inc., 13980 N.W. 58th Court, P.O. Box 4650, Miami Lakes, FL 33014-0650). Several leaks were made by gluing various diameters and lengths of hypodermic tubing (0.005 to 0.008 inch inside diameter) to threaded brass fittings using epoxy cement. The response of the microbarograph may then be adjusted in a repeatable fashion by varying the volume of the syringe and the size of the hypodermic tubing.

The time constant can be determined with coarse accuracy by connecting a scope to the CDG and then creating a slightly negative pressure on the reference side by slightly withdrawing the plunger of the syringe. The scope will show the characteristic exponential curve as the pressure comes back to equilibrium. The time constant and, therefore, the frequency turnover can then be determined.

Note that I specified a negative pressure on the reference side. It is best to avoid positive overpressures on the reference side of some differential CDGs as the diaphragm can become distorted when it stretches toward the baffle. Usually the measurement side can take positive pressures to 20 psi or more because the diaphragm is not harmed by flattening against the ceramic/metal electrode structure. One configuration of the MKS Type 223 is available with 20 psi overpressure on either side and that is the configuration that should be specified if going this route.

Figure 3 shows the completed microbarograph. I mounted all of the components on a piece of plywood that was sized to fit snugly into a "Playmate" cooler. The cooler helps to minimize fast temperature variations. The lid fits securely enough to keep rain out but, since it is not air tight, the instrument works properly. The electronics package is a simple fixed gain amplifier made from two op-amps. This is used to boost the output of the CDG to a level that is adequate to drive the recording system.

Figure 3 - Complete Microbarograph



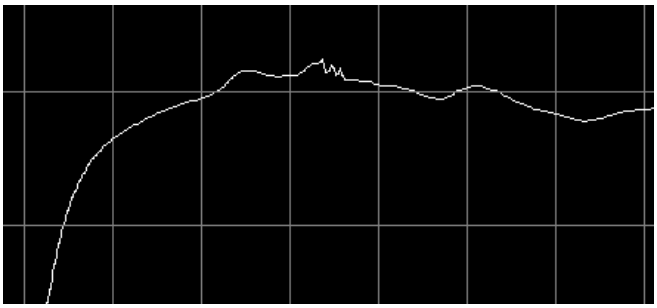


**Figure 4 - Output of Microbarograph with "Longer" System Time Constant.
Pressure relaxation (left) and door swing (middle).
Vertical Scale: 100 mTorr/div.
Horizontal Scale: 20 sec/div.**

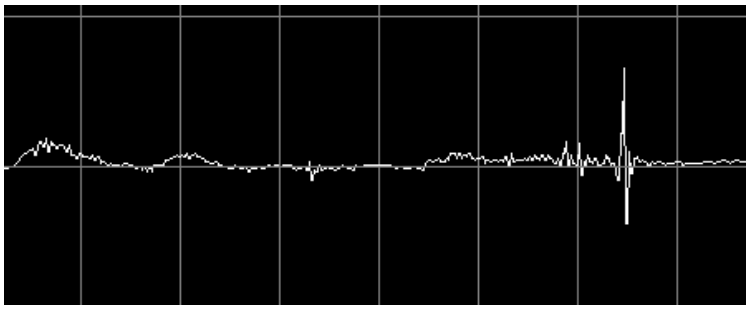


**Figure 5 - Output of Microbarograph with "Shorter" System Time Constant.
Response to passing jet at right.
Vertical Scale: 6 mTorr/div
Horizontal Scale: 20 sec/div.**

Proper siting of the instrument is important. Keep it outdoors, away from heavy traffic, out of the sun and shielded from the wind. Wooded areas are good locations. The instrument and its insulated box may also be located indoors with a length of moderate diameter tube (e.g. a piece of garden hose) leading out of doors to an appropriate area. The French microbarographs of Reference 9 used a 3 meter length of 1.5 cm tubing for this purpose.

At this point, I have completed the basic instrument and have run some tests with the instrument indoors, near an open door. The next steps will be to site the instrument in the woods behind the house and optimize the frequency response.

While I haven't recorded anything of great significance, the device is very sensitive. Moving a door anywhere in the house will show a strong response. Figure 4 shows the instrument with a "slow" response. The left hand side of the trace shows the exponential response of the reference volume as the pressure equalizes. The three oscillations at the center were produced when I slowly swung a closet door at the other end of the house.

Figure 5 shows a recording made at higher sensitivity and with a faster response (time constant about 1 sec). The signal toward the right hand side of the trace corresponds to the passage of a jet airliner as it made its approach to the Manchester, NH airport, about 15 miles distant. I can't say for sure if the trace is due to the jet. However, nothing else seemed to be going on at the moment.

IV. NEXT STEPS

Two items are on the plate as next steps. The first is to set up and configure the computer based data recording and analysis system. The second is to add a noise reduction system to the device.

For data recording I am using two programs that were developed for amateur seismology by Larry Cochrane, the driving force behind the Redwood City Public Seismic Network. The first program, Seismic Data Recorder (SDR), is a DOS program that is used to collect the data. It is used in conjunction with a PC Labs 711s AtoD card (available from Jameco, (800) 831-4242) or a similar (and less expensive) card that Larry produces. The second program, WinQuake, is used to view and analyze the recorded events. Both of these programs are free and are available via the PSN's Web site at http://psn.quake.net/.

With regard to the second item, I will be looking at ways to decrease the effects of local noise. A variety of noise reduction techniques have been developed to minimize the effects of wind noise. Simply baffling the inlet with a piece of foam (like the wind shields seen on microphones) will have some positive effect but the best performance is obtained with arrays of long lengths of perforated or porous hoses that are connected to the inlet of the microbarograph. References 8, 10 and 11 may consulted for further information.

Finally, it must be noted that I only looked at two commercially available pressure transducers. This should not prevent the amateur from evaluating other units or even attempting to build the sensing element.

V. ACKNOWLEDGMENTS

I would like to thank Bruce Kendall of Penn State University who first brought this topic to my attention, Rodney Whitaker of Los Alamos National Laboratory who was kind enough to provide useful comments as this article was being developed, and Peter D. Hingley, Librarian of the Royal Astronomical Society, who provided reprints of several sections from Reference 8. I would also like to extend appreciation to MKS Instruments, Inc. who provided the capacitance manometer that was used in this project.

REFERENCES

1. Rodney W. Whitaker, "Infrasonic Monitoring," Phillips Lab Monitoring Symposium, 1995.
2. Earle E. Gossard and William H. Hooke, "Waves in the Atmosphere," Elsevier Science Publishers, 1975, Chapter 9.
3. Jearl Walker, "Billows in the Ionosphere are tracked with transistor radios" in The Amateur Scientist, Scientific American, September, 1980.
4. The International Meteor Organization maintains a Web site at http://www.tu-chemnitz.de/~smo/imo/. They indicate that their URL is subject to change.
5. Douglas O. ReVelle, "Historical Detection of Atmospheric Impacts by Large Bolides Using Acoustic-Gravity Waves," International Conference on Near-Earth Objects, Sandia National Laboratory, April 24-26, 1995.
6. Douglas O. ReVelle, "Infrasonic Observations of Meteors," 1995 Monitoring Technologies Conference, May 15-18, 1995.
7. Douglas O. ReVelle, "On Meteor Generated Infrasound," Journal of Geophysical Research, Vol. 81, 1217-1237, 1976.
8. The Geophysical Journal of the Royal Astronomical Society, Vol. 26. (This is an issue devoted to infrasound propagation theory, instrumentation and observation.)
9. C. Delclos, E. Blanc, P. Broche, F. Glangeaud and J.L. Lacoume, "Processing and Interpretations of Microbarograph Signals Generated by the Explosion of Mount St. Helens," Journal of Geophysical Research, Vol. 95, D5, 5485 (April 20, 1990).
10. F.B. Daniels, "Noise Reducing Line Microphone for Frequencies Below 1 cps," Journal of the Acoustical Society of America, Vol. 31, 529-531, 1959.
11. S.D. Noel and W.W. Whitaker, "Comparisons of Noise Reduction Systems," Los Alamos report LA-12008-MS, 1991.

Note: References 1, 5 and 6, as well as several other papers, may be ordered through the Web site operated by the DOE's Comprehensive Test Ban Treaty Group: *http://www.ctbt.md.doe.gov/cgi-bin/bibl.cgi.*

This article was originally presented in Volume 5, Number 4. There have been several updates since. I did reach the conclusion that sufficient thermal stability could only be gained by mounting the sensor underground. However, the manifold has remained essentially the same.

A Home-Grown, Sealed Carbon Dioxide Laser

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Abstract: The construction of a sealed, CO_2 gas discharge laser was undertaken as an independent study project. Glass laser tube design, as well as clear acrylic housing, make it an excellent demonstration tool. Sealed operation was characterized in mode, power, warm-up and stability over the period of weeks. Novel design approaches were used for expediency and cost saving. An anomalous turn-on behavior is also discussed.

In reviewing the vacuum related articles in Scientific American's The Amateur Scientist*, a high percentage of them dealt with the construction of lasers of various types. When I undertook t*BJ *I figured that lasers would also play a prominent role. That has not proven to be the case, perhaps due to the ready availability of low-cost HeNe and diode lasers.*

Certainly, if one needs a laser to perform a specific task, then the easiest route is to buy or borrow one. Here the laser is a means to an end, an appliance. But, if one wants to understand lasers, then there is no better way than to go through the experience of building and optimizing one.

I discovered this article, in unfinished form, on the Internet a few months ago. With minimal prodding, David agreed to complete the article for publication in this journal. While this article is fairly technical, it does approach the construction of the laser in the manner that an ambitious amateur would. Anyone undertaking such a project would also be advised to acquire a copy of the CO_2 laser article which appeared in The Amateur Scientist *of September, 1971*. - Ed.

I. INTRODUCTION

The construction of a sealed CO_2 laser was undertaken for several reasons. First, the CO_2 laser provides an impressive and graphic demonstration of quantum mechanical processes. Since the laser is constructed out of glass all of its components can be clearly seen if it is contained in an acrylic case. Finally, the laser produces more than 20 watts of coherent, monochromatic laser radiation, enough to burn paper and wood.

Another reason why this was undertaken was because all of the supplies, equipment and lab space were readily available; it only took time and inclination to exploit these resources to arrive at a useful device. It was also an achievable project as evidenced by the author's demonstration of a smaller, less complicated flowing gas CO_2 laser the previous semester in a junior physics lab course.

Most importantly, the author simply wanted to have a relatively compact, portable laser, which did not require bulky vacuum equipment or gas cylinders. In this manner, a CO_2 laser would be available for future work.

II. THE CO_2 LASER

The Resonator

The resonator used is confocal-confocal. Since the optics used in the this project were donated from a company, the author did not have a choice of mirror curvature. Since it was higher power and not best quality mode that was desirable, particular attention was not paid to resonator optimization. With the high reflectance mirror radius of curvature of 2.685 m, and output coupler radius of 5.072 m, the resonator is in the stable resonator regime at 0.66, as determined by the stability relation:

$$0 \le \left(1 - \frac{L}{R_1}\right) - \left(1 - \frac{L}{R_2}\right) \le 1$$

where L is the cavity length, here 65 cm, and R is the radius of curvature of the mirror.

Energy Transfer in the Discharge

The most commonly observed laser transitions in the CO_2 molecule, barring the use of any frequency tuning mechanisms, are from the CO_2 asymmetric stretch transitions, from the (00°1) to the (10°0) 10.6 micron and (02°0) 9.6 micron states, using the notation $V_1V_2°V_3$, where V_1 refers to the symmetric stretch quantum number, V_2 refers to the asymmetric stretch quantum number and V_3 refers to the asymmetric stretch quantum number.

There are literally dozens of other lasing transitions [6] which can be easily chosen by employing an intracavity grating. In a CO_2 laser, lasing of one

vibrational transition precludes the efficient lasing of another, so that lasing lines 'hop' from one to another depending on instantaneous gain medium and resonator conditions. Any single possible laser line can be forced through the use of an intracavity grating. Rotational structure, having energies clustered very close to one another, may exist at any time.

Nonradiative decay to short-lived lower lying states followed by nonradiative decay to the ground state follows. N_2 is added to the laser gas to more efficiently transfer energy from electron impact to the CO_2 upper vibrational laser level.

The glow discharge is a very effective mechanism for vibrational excitation of nitrogen. Since N_2 is a homonuclear molecule, dipole radiative de-excitation is forbidden. This allows for long-lived vibrational states which makes excited N_2 molecules more readily available for collisional excitation of CO_2. De-excitation is only accomplished collisionally with the wall or other gas constituents, the most beneficial of which is the CO_2 molecule. The N_2 v=2 state is only 18 cm^{-1} (2.2 x 10^{-3} eV) from the upper laser level of the CO_2 molecule. This makes resonant energy transfer between N_2 and CO_2 more likely. This energy is much smaller than the average kinetic energy of the molecules in the surrounding glow, so vibrational energy can easily be supplied to the CO_2 molecules. Energy transfer occurs from vibrational levels up to v=4 in N_2, because the ensuing anharmonicity of these states, due to bond stretching, is still well below the average molecular kinetic energy [7].

CO is isoelectronic with N_2 and also has vibrational levels easily excited in the glow discharge. Figure 1 details the more common energy transfer routes in the CO_2 laser. Excited N_2 and CO transfer vibrational energy through collision to CO_2, exciting any of a number of stretch and vibrational modes.

Thermal poisoning, a build up of lower lasing level populations in CO_2, is a problem that can occur. This results in a reduction in laser output power due to a "clogging" of the path from the upper lasing level to the ground state where the CO_2 upper lasing level is most efficiently populated through collisions with N_2. These lower levels may be cooled by the addition of He to the gas mix.

The helium energy levels are much higher than the molecular energies of N_2 and CO_2: above 20 eV. For typical electron energies in a glow discharge of 1 to 3 eV, the discharge is not significantly affected by the addition of He [7]. Only a small amount of energy is lost from the discharge due to inelastic collisions with He and subsequent collisions with the walls.

At the pressures we are dealing with, the thermal conductivity of any specific gas is independent of pressure. Since the thermal conductivity of He is roughly six times that of CO_2 and N_2, He makes an

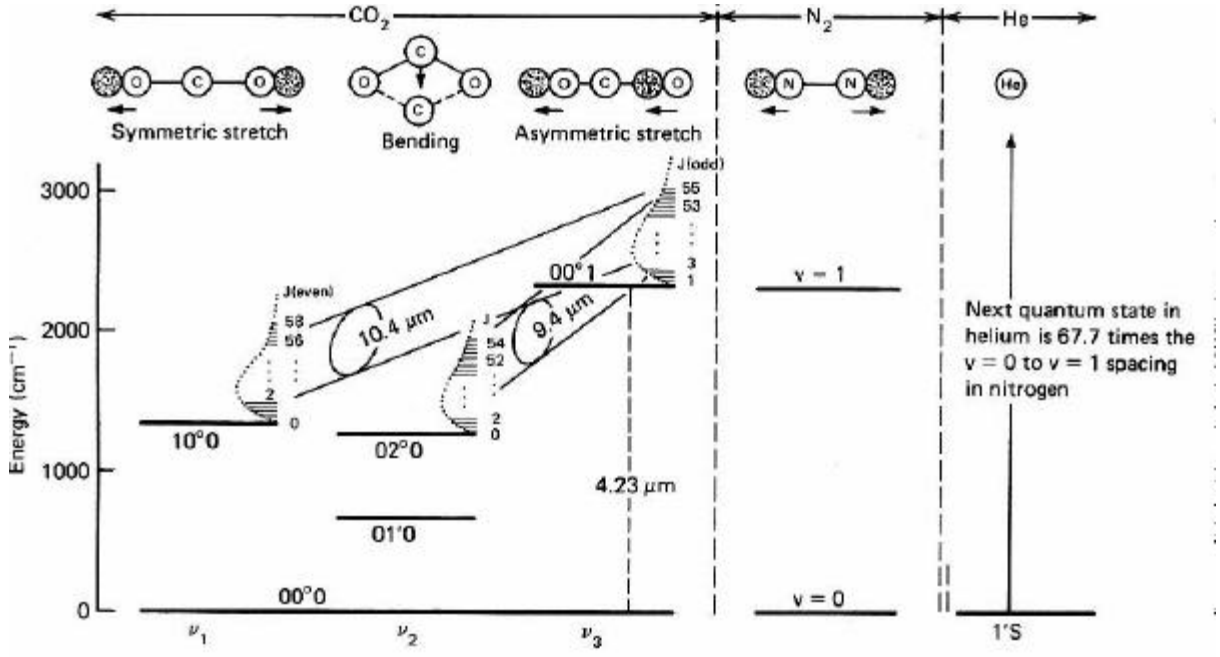

Figure 1 - Energy Level Diagram of the CO_2 Laser

efficient transporter of waste heat to the walls of the discharge tube. The efficiency of heat transfer resulting from the addition of He to the mixture allows for a higher discharge current before radiation saturation [7].

CO may also be added to the laser mix to improve efficiency, but it does not transfer vibrational energy as efficiently as N_2, due to a difference between the CO ν=1 level and the CO_2 upper lasing level of 170 cm^{-1}. CO also has a dipole moment which creates a radiative decay channel to depopulate the electron impact excited CO, thus making CO less available for the job of CO_2 excitation. CO is also a component in the dissociation equilibrium of CO_2, so when using added CO with CO oxidation catalysis, larger concentrations of CO affect the CO_2 concentrations, not always in a predictable manner. Even with these drawbacks, CO still adds to more efficient CO_2 vibrational excitation than electron impact alone.

H_2O can be added as a heat transfer enhancer but is less efficient at cooling than He. H_2O, in small concentrations, additionally has the beneficial side effect of homogeneous catalytic recombination of the dissociated CO_2 products, CO and O [1]. H_2O in larger concentrations overwhelms the beneficial catalytic effects and effectively depopulates the upper lasing levels of CO_2. The optimum concentration of H_2O in the laser gas has been shown to be a function of the laser bore diameter [7].

Xe may also be added to a laser gas mix to effectively cool the electron temperature of the discharge for a given current, thereby reducing the amount of electron impact dissociation of CO_2. The prohibitive cost of laboratory grade Xe prevented this investigator from utilizing it.

III. CONSTRUCTION

The Laser Tube

The laser is a sealed, DC discharge type with attached ballast tank for long gas life. The silver-copper cathode design was used to reduce the amount of gas consumption by sputter pumping through chemisorption and physiosorption. Construction of the laser was kept simple to reduce expense and excessive consumption of time. The laser was constructed of Pyrex, fabricated on the Boulder Campus by the chemistry department's Master Glass Blower. Figure 2 is a dimensional drawing of the laser.

The design incorporates a laser bore nested in a water cooling jacket, with a feed through the water jacket so that the discharge can go to an external cathode and anode. Having the external electrodes lower than the axis of the laser bore reduces the possibility that sputtering or oxidation products at the electrodes will contaminate the optics. This becomes an important consideration when working with CO_2 lasers as intracavity power densities, even in a resonator of this design, can easily exceed 100 W/cm^2, quickly causing thermal damage on the surface of an optical element, should a small piece of contaminant land on it.

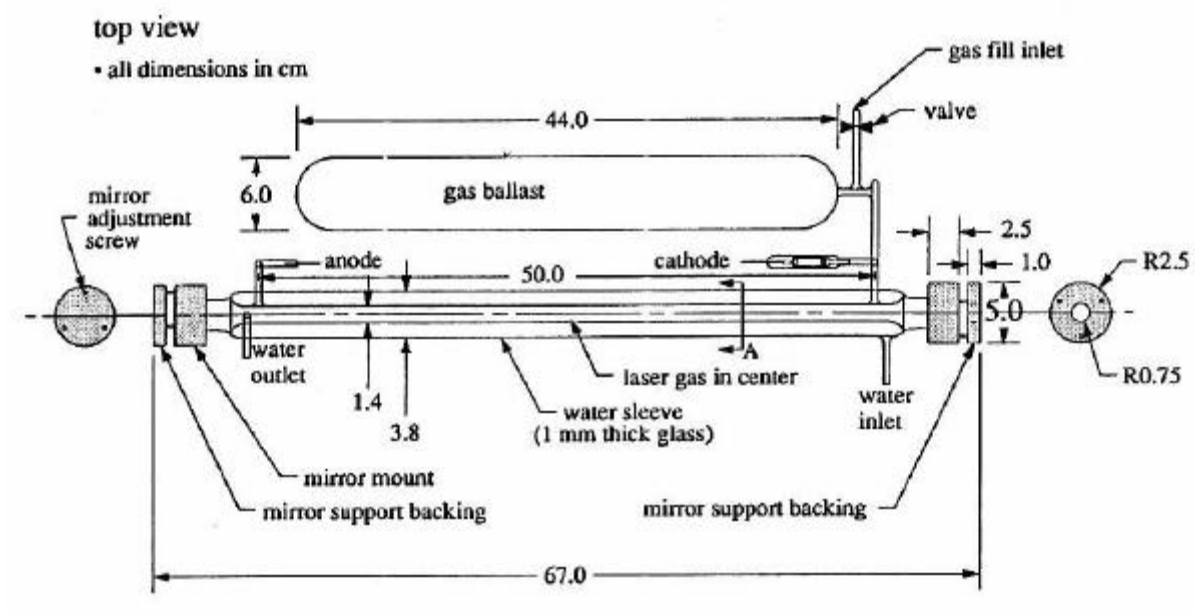

Figure 2 - Schematic of the Sealed CO_2 Laser with Critical Dimensions

A glass vacuum valve is attached for evacuation and filling of the laser gas mixture. The water inlet and outlet are positioned so that air bubbles which form inside the water jacket are ejected as they rise to the top of the tube. Without this design, air buildup inside the jacket would create a radial temperature differential perpendicular to the optical axis, detuning the resonator and/or thermally poisoning the gain medium.

The Cathode

Cathode material selection in a sealed DC laser is crucial. The abundance of free, ionized oxygen in the discharge rules out any materials which readily oxidize, like tungsten, nickel, aluminum or stainless steels. The abundance of CO in the discharge also limits many otherwise suitable materials since many materials form gaseous carbonyls, removing CO_2, and exposing fresh cathode surface for oxidation. These carbonyls can then be transported to the optics and deposited, forming a strong IR absorber. The "Hochuli cathode" was found through a literature search on cathode materials conducted earlier by the author. Professor Urs Hochuli generously donated cathodes for this project.

The Hochuli cathode was designed specifically for long life sealed DC CO_2 lasers, but was only tested at much lower currents, on the order of 5 mA [3]. The Hochuli cathode is made of Ag and Cu in a matrix which is internally oxidized. This oxygen equilibrium within the cathode keeps the Ag and Cu from consuming oxygen from the discharge, thereby eliminating the cathode as a chemical sink of oxygen. The materials in the cathode are not readily transported in the discharge, so mirror contamination is not a problem. The cathode is in the familiar hollow cathode design, so sputter pumping of gas through physisorption is less of a problem, although this may be the ultimate source of gas consumption by the cathode.

A glass plasma limiting sheath is incorporated in the cathode design to prevent the glow from forming on the outside of the cathode. Should that happen, it would rend the hollow cathode design useless, and sputter pump the heavier gas constituents quite efficiently. The discharge is physically channeled to the inside of the cathode, as the sheath acts as a physical plasma limiter. A small rod of tungsten is used as the anode since it is rather easily fed through the glass using a graded uranium glass to metal seal. The anode doesn't suffer from the high momentum impacting ions as the cathode does, so it does not act as a sputtering loss element. The cathode is the only electrode which suffers from sputtering, since it is the source of electrons, which requires a large number of ions crashing into its surface to form secondary electron emission. This is the mechanism whereby the cold cathode provides electrons [4].

Mirror Mounts

The mirror mounts are of a simple yet versatile design, incorporating an O-ring which provides both the vacuum seal and the restoring force for the mirror adjustment. The two mounts are identical and are shown in Figure 3.

The mounts were turned from pieces of scrap rolled aluminum. The finished pieces were sanded with a very fine grained sand paper and then were polished. This served to reduce the surface area exposed to the low pressure environment with the effect of reducing outgassing of adsorbed contaminants which could poison the laser gas. Since the travel needed for optical adjustments is very small, the motion of the mirror on the O-ring won't break the vacuum seal.

Both the high reflectance (HR) mirror, and the output coupler (OC) have mirror mount backings with a depression in them to hold the mirror near the center of the optical axis during assembly. With extra room between the glass end of the laser and the mirror mount shoulder, the mount can be aligned coaxially to the laser bore during assembly and the glass blowing need not obey strict tolerances. The mirror mounts need not be mounted exactly perpendicular to the laser bore since even relatively gross alignments can be accomplished with the O-ring backing plate.

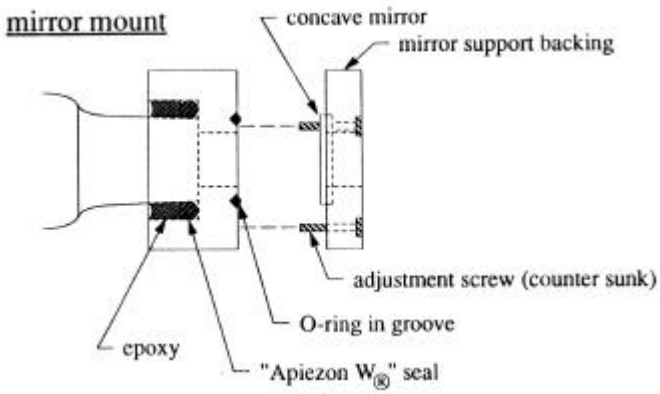

Figure 3 - Mirror Mount Detail

Assembly

The mirror mounts were attached to the glass laser tube with the aid of a mandrel which aligned the mirror mounts coaxially with the optical axis. The mandrel consisted of a piece of Delrin turned so that one end fit snugly into the center of the mirror mount with the other end fitting snugly into the laser bore. This alignment procedure was necessary to compensate for the previously noted relaxed tolerances of the glass work.

To install the mounts, the laser tube was tipped on end and the mirror mount brought up from beneath with a small lab jack, which then met the laser tube end. After each mirror mount was aligned correctly, the mandrel was removed.

Next, some Apiezon W vacuum sealing wax was broken into small chips and dropped along the inside edge of each mirror mount, between the glass and the aluminum shoulder. A heat gun was used to warm the mirror mounts so that the sealing wax melted and flowed to form a positive vacuum seal between the mount and the tube. Apiezon melts at about 80 °C. While Apiezon W makes a suitably clean vacuum seal, it does not have adequate structural strength for this application, so "Five Minute" household epoxy was added on top of the wax, to enhance the mechanical integrity of the mount.

After the mounts were installed, the device had to be aligned. This was done using a HeNe laser. This was facilitated by mounting the laser on an optical rail.

The rail also served to support an acrylic enclosure. This protected the laser from breakage and the user from electrical shock. The laser tube itself was secured in the acrylic box by using acrylic mounts lined with Sorbethane shock absorbing material. Acrylic pieces screwed in the laser mounts secured the laser tube proper while aluminum strips were used to secure the ballast tank to its mounts.

For alignment of the laser resonator, two alignment mandrels were again made, each fitting snugly in the ends of the laser bore, but easily passing through the mirror mount. Each had a 1mm hole drilled in the center so that coaxial alignment of the HeNe alignment laser with the CO_2 resonator was assured.

After the HeNe beam was aligned with the CO_2 laser bore, the mandrels were removed and the rear reflector was mounted and aligned so that the beam was reflected back into the HeNe aperture. The output coupler was then mounted then aligned to the HR by eye, since trying to make sense of the multiple reflections of the HeNe beam was futile. A bright, quartz halogen light was directed into the OC so that the image of the light was visible on the HR when looking down the bore through the OC. The OC was then adjusted so that the light source created "hall of mirrors" effect between the HR and OC. With a concave-concave resonator and such a high gain medium as CO_2, this type of alignment procedure is more than adequate. A simple evacuation and fill system was assembled using Polyflo tubing and Swagelok fittings which used polyethylene ferrules. Industry standard CO_2 laser gas mix consisting of 4.5% CO_2, 14% N_2 and balance He was used for the fill.

Power Supply

The power supply for the laser was designed for low cost and simplicity. A 15 kV, 60 mA neon sign transformer provided the high voltage. The output of this was then connected to a high voltage bridge rectifier. Since high voltage bridges are expensive and often difficult to find, the bridge was assembled out of several HV diodes in series, one set of three diodes on each leg of the bridge. The output of the bridge was connected to a HV filtering capacitor, with a 30 Meg Ohm bleeder resistor across its terminals.

200 k Ohms of high power ballast resistance is placed in series with the discharge to limit tube current. The tube current is varied rather crudely, by simply using a Variac to adjust the input voltage to the transformer.

Since the secondaries of neon transformers are center tapped to the case, neither end of the winding can be tied to ground. Consequently, while in operation, all components of the HV end of the power supply are floating with respect to building ground. For safety, the power supply components were mounted in a separate acrylic enclosure

IV. LASER OPERATION

After the laser was assembled and aligned, it was connected to a sink faucet and drain for cooling water. (Eventually a closed circuit heat exchanger will be built to make the laser self contained.)

An open-ended U-tube mercury manometer was used to measure the pressure of the gas fill. Due to daily fluctuations of atmospheric pressure, the manometer had to be set to the day's pressure. The laser and fill system was roughed out until it settled to its lowest value. This lowest value was taken to be "0" Torr, since a good mechanical pump can pump down to at least 0.1 Torr and usually much lower [5]. Due to this approximation, the laser tube pressure is taken to be within one Torr of measured value.

After evacuation, a leak-proof system was verified to first order by simply closing off the system to the pump, then watching the pressure gauge for a rise in

pressure. The system was deemed to be sufficiently leak free when no detectable rise was observed during a period of several days.

This test over with, the system was filled to a nominal pressure of 30 Torr and lasing was attempted. Lasing was obtained but it lasted for barely a second. This anomalous behavior was a setback, but after careful consideration revealed itself to be a chemical imbalance, as will be discussed later in this paper.

The laser eventually gave consistent performance and output power data were taken at varying pressures and currents.

Mode burns were taken in several materials and with several alignments of the resonator. A burn at the best power alignment was also taken. Due to laser powers exceeding the capabilities of the commercial power meter available, power was measured by a simple thermocouple pair. In this arrangement, the laser light was absorbed by a small piece of black anodized aluminum, which is very effective at the 10 micron wavelength of the laser. This small piece of metal was mounted against a larger heat sink to dissipate the laser power. One junction of a thermocouple pair was placed against the target piece, and another series reference junction was placed against the heat sink. The millivolt output of the junctions was monitored as an indicator of input laser power. This meter was then calibrated using a commercial laser power meter.

V. PERFORMANCE

The data in Table 1 were taken before the laser tube had come to complete equilibrium from the turn-on phenomenon described earlier and without optimizing the resonator each time. Therefore, the peak power obtained in the data does not correspond to the highest power obtained (22 watts). It is quite clear from the data, however, that an optimum pressure and current does exist for maximum power and efficiency. The optimum pressure was arrived at empirically to be 30 Torr. The optimum current was 9.2 mA.

After cycling the laser power and allowing for mixing as described earlier, output power was measured as a function of time. Power fluctuations at turn-on were found to be due to mode changes. It appears that as the laser warms up, the resonator alignment changes. This can be verified by simply tweaking the mirrors for the lowest order mode while it is warming up, then watching as that mode is detuned. The mode can be brought back by simply adjusting the resonator again.

Thermal detuning appears to be a plausible explanation since the glass tubes which feed the discharge out the laser bore go through the walls of the tube which make up the laser resonator. These feeds heat up on the outside since there is no water cooling over them. Because of this they expand and create a wedging in the resonator structure.

Given this, the laser can be tuned for best mode after stabilizing. If the laser is allowed to cool and then is turned back on and allowed to restabilize, the good mode that was achieved previously will come back. The supports the idea of thermal detuning.

Power performance as a function of time after turn on is given in Figure 4. Monitoring the mode periodically during the first few minutes at turn on indicates that the power fluctuations are due to the cavity tuning through different modes. In the data shown, the power fluctuations settle down after about 25 minutes and settle to something other than the best power mode.

Further measurement has shown this settling behavior to consistently be the case. The spikes in the data are artifacts of a noisy chart recorder. The thermal inertia of the meter does not allow for such a fast response so the spikes cannot be real. Initial variation of 22% of the peak power is seen as the cavity tunes through different modes and the highest power attained is 16.5 Watts. The resonator does not settle to the highest power mode.

Typical best power ‘donut’ or ‘bulls eye’ laser modes, (TEM 01* or TEM 10) are shown as contrast-enhanced burns when thermally sensitive fax paper or plain white cardboard are placed in the beam.

Other mode burns in cardboard, taken during cycling of the laser during warm up and over periods of about one minute, show evolving symmetries corresponding to lower power.

VI. DISCUSSION

The anomaly mentioned earlier where, upon a fresh fill, measurable laser power lasts less than a second, is believed to be due to oxygen specie migration between the laser bore and the ballast tank. This mechanism is believed to occur as follows: Upon initial filling, the gas in the laser bore and ballast tank is uniformly a laser mix of 18.7 to 2.4 to 1 ($He:N_2:CO_2$). When the discharge is turned on, many other species are formed due to dissociation of CO_2 and CO and O and the subsequent formation of NO_x and other compounds. At this point, the laser bore has an abundance of NO_x and other species, and the ballast tank does not. Diffusive processes drive these byproducts from the laser bore to the ballast tank. Indeed, the characteristic green glow of N+O combination fluorescence can be seen streaming out of the cathode section toward the ballast tank. Also, a characteristic white discharge in the laser bore indicates a CO rich medium, which would be the

case if free atomic oxygen were leaving the bore. In low current discharges of CO_2 laser mix, the pinkish emission bands of the N_2 fourth positive system are seen which are due to nitrogen first-positive transitions [2].

If a white discharge is seen, dissociation is dominating the discharge. The length and brightness of the exiting fluorescence stream is demonstrated to be proportional to the discharge current. A rapid migration of oxygen containing species out of the laser bore makes for a sink of CO_2 in the ballast tank and the laser then becomes lean in its lasing medium. If this migration hypothesis is correct, we would expect that the laser power should last longer upon initial turn on if dissociation products are allowed to come to an equilibrium between laser bore and ballast tank.

This is indeed the case. After repeatedly turning on the laser then allowing diffusion time, performed over a period of a couple days, power does begin to remain consistent at turn on. The maximum power attainable does improve after several days of turning the laser on and off and allowing it to sit. Ultimately, a laser power of 22 Watts (44 Watts/meter) has been achieved this way, with relatively stable output power lasting for over an hour's worth of operation.

VII. CONCLUSIONS

The original objective of building a sealed, DC CO_2 laser was accomplished with satisfactory laser performance. The anomalous turn-on effect delayed progress since many other possible problems were

Table 1 - Performance Data

	Current (ma)	Voltage (kV)	Power (W)	Efficiency
20 Torr				
	5.3	9.76	1.1	.021
	5.7	9.59	.93	.017
	6.1	9.55	.22	.004
	6.5	9.36	.99	.016
24 Torr				
	6.9	10.46	1.42	. 020
	6.5	10.44	1.75	.026
	7.0	10.29	2.07	.029
	7.6	10.15	1.26	.016
	7.8	10.12	.99	.012
	8.1	10.03	.17	.002
26 Torr				
	7.4	10.57	2.94	.037
	7.7	10.78	3.49	.044
	8.2	10.45	2.62	.031
	8.6	10.35	2.18	.024
	8.9	10.71	1.75	.018
28 Torr				
	8.4	11.13	9.80	.015
	8.8	11.03	7.62	.079
30 Torr				
	9.2	11.40	11.43	.109
	8.9	11.37	10.34	.102
	8.8	11.28	9.53	.096
	9.1	11.25	5.45	.053
	9.4	11.08	.99	.009
32 Torr				
	9.0	11.41	3.75	.037
	9.1	11.31	3.43	.033
	9.3	11.17	1.64	.016

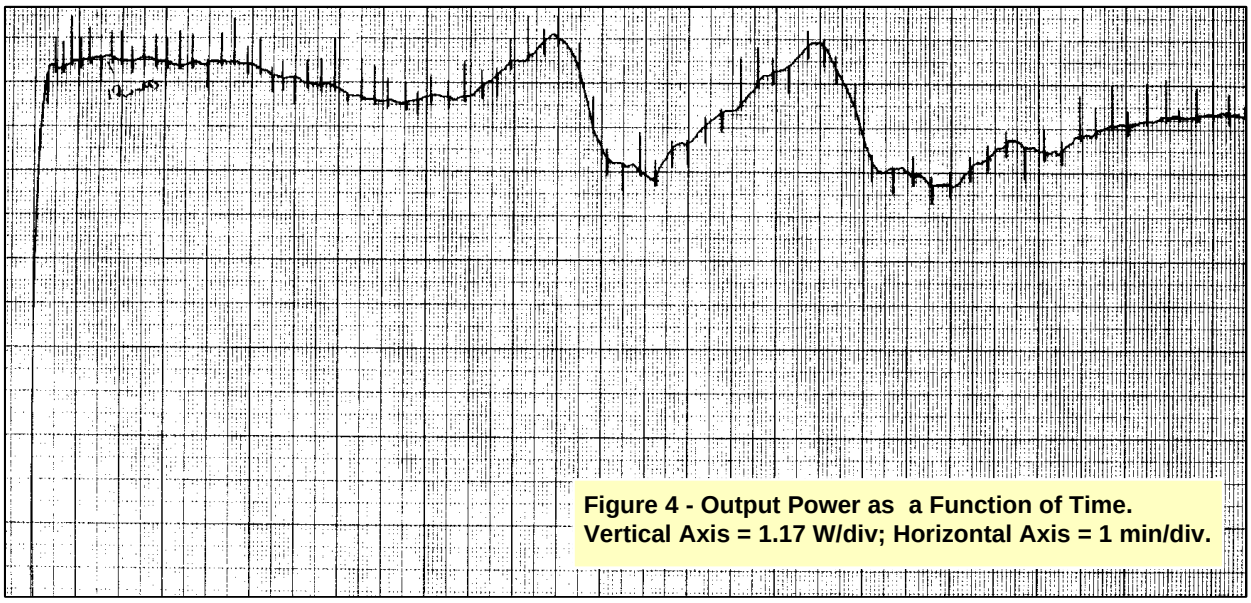

Figure 4 - Output Power as a Function of Time. Vertical Axis = 1.17 W/div; Horizontal Axis = 1 min/div.

investigated first such as a leaky system, water contamination, dirty optics etc., but is now believed to be understood. The laser has yielded powers up to 22 Watts and has repeatable power performance, on a single fill, on the order of several weeks. The best mode attainable for this resonator design is a donut mode (TEM 01* mode).

VIII. FURTHER INVESTIGATIONS

It is recommended that, should further research be possible on this laser design, the oxygen transportation phenomenon be more thoroughly studied. More detailed analysis will most likely require the use of a mass spectrometer for time resolved characterization of the gases in the laser bore and in the ballast tank after initial turn-on. Improvements in the resonator design with respect to thermal stability and the warm-up cycle is desirable.

Recent work in Au intracavity catalysts has been said to improve laser performance to powers in excess of 100W/m. Further studies in intracavity catalysts such as Pt/Pd on SnO_2 or Rh on SnO_2 may prove fruitful.

IX. ACKNOWLEDGMENTS

The author wishes to acknowledge the contributions and support of Professors D. Bartlett, B. Ridley and S. Robertson of the University of Colorado at Boulder Physics Department and Professor U. Hochuli as well as the technical assistance and support of E. Lutter and R. Tyler.

COMMENTS - OCTOBER 1996

Sputtered gold catalyst was added to the bore several years after construction of the tube. This allowed higher tube currents and output powers exceeding 50 watts.

REFERENCES

1. Campbell, I. M., *Catalysis at Surfaces*. Chapman & Hall Ltd., New York, 1988.
2. Gaydon, A. G., & Pearse, R.W.B., *The Identification of Molecular Spectra*. Chapman and Hall, New York, 1976.
3. Hochuli, U., et al., IEEE JQE., **10**, 139-244, 1974.
4. Llewelln-Jones, F., in *The Glow Discharge*. Edited by Worsnop, B. L. John Wiley & Sons Inc., New York, 1966.
5. O'Hanlon, J. F., *A Users Guide to Vacuum Technology*. John Wiley & Sons, New York, 1980.
6. Weber, M.J., *CRC Handbook of Laser Science and Technology, Vol. 2, Gas Lasers*. CRC Press, Boca Raton, FL, 1986.
7. Verdeyen, J.T., *Laser Electronics*. Prentice-Hall, NJ 1981.

Other useful references include Ready, J.F., *Industrial Applications of Lasers* (Academic Press, NY, 1978) and Seigman, A.E., *Lasers* (University Science Books, Mill Valley, CA, 1986).

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Part 4: Musings

Vacuum in *the Amateur Scientist*

As has been mentioned frequently, Scientific American's *Amateur Scientist* column contained a number of vacuum related projects. The compendium "The Scientific American Book of Projects for the Amateur Scientist" (Simon and Schuster, NY, 1960) contained a number of columns from the late 1950's. Two of particular interest are the ones on an electron accelerator and homemade cold cathode x-ray tubes. The former, contributed by Franklin Lee, was the first project to use converted refrigeration compressors and a simple glass mercury diffusion pump (Kurth-Ruggles design) to produce high vacuum in an amateur project. Lee's voltage source was a fairly large van de Graaff generator that could charge its sphere to a potential of 500 kV.

Lee's project prompted the formation of Morris & Lee, a company that supplied van de Graaff kits, vacuum pumps and various accessories. Later columns incorporated many of Lee's techniques. Specific projects detailed in these columns included a He-Ne laser (9/64 with an addendum in the 12/65 issue), a high altitude chamber for biological studies (9/65), Steiner's hand pumped discharge tube (8/66), an argon gas laser (2/69), molecular beam apparatus and mass spectrometer (7/70), a proton & deuteron accelerator along the lines of Lee's machine except with a simple ion source (8/71), a CO_2 laser (9/71), two transmission electron microscopes (9/73), N_2 laser (6/74) and a mercury-vapor ion laser (10/80). The molecular beam apparatus and mass spectrometer are elegant little designs and it is hoped that this journal will have updated versions in the near future. The electron microscopes were built by students at a private high school in Chicago. One used a cold cathode electron source with a simple (one element) lens to produce a maximum magnification of about 100x. The other used a filament to produce the electrons and had three lenses (condenser, objective and projection) to provide magnifications to 10,000x.

A later Scientific American compilation, "Light and Its Uses" (W.H. Freeman, 1980) contained the above mentioned laser articles along with 21 other projects concerning lasers, holograms, interferometers and spectrographic instruments.

Stong's predecessor at Scientific American, Albert Ingalls, produced the classic three volume series "Amateur Telescope Making." While mostly not dealing with vacuum, Book 2 (1944) includes a chapter on aluminizing mirrors by John Strong of Caltech. Strong, as you may remember, was the author of "Procedures in Experimental Physics", now available through Lindsay Publications. This chapter also provides notes on the aluminizing of the 100 inch Mt. Wilson mirror. In an afterward, Ingalls noted "John Strong is not (that is, not yet-1944) yet an amateur telescope maker (ATM) though hopes are perennially entertained by the present writer. He has, however, been so close to Russell Porter (the 'patron saint' of ATMs and the designer of much of the mechanical structure of the 200 inch Palomar telescope)...that the amateur's outlook has rubbed off on him."

Book 3 (1953) offers a chapter on high vacuum equipment by Earle R. Brown of Farrand Optical Company. Here there are some good ideas on feedthroughs, valves and other improvised hardware. Also covered are evaporation techniques for various materials other than aluminum and general information on lens coating, interference filters and the cleaning of optics.

Brown's introduction is interesting and relevant to this day. He says "Every TN (Telescope Nut) has probably had the desire to aluminize his own mirrors and anti-reflection coat his own lenses...but has felt that the equipment for doing this work was so complicated and expensive that it was not worth while... Coating lenses and aluminizing mirrors, however, is only a small part of the work which can be performed by high vacuum equipment, and the processes of which it is capable offer a fascinating field for the TN with his mechanical ingenuity and intellectual curiosity.... Not the least of the appeal of high vacuum to the TN is its natural perversity. Compared to high vacuum systems, the most recalcitrant optical surface is a paragon of meek submissiveness. This sort of thing makes raving maniacs of most people, but TN's are of the peculiar breed of cat which thrive on frustrations."

Pathological Science

This was Irving Langmuir's term for 'sick science' where there is "no dishonesty involved but where people are tricked into false results by a lack of understanding about what human beings can do to themselves in the way of being led astray by subjective effects, wishful thinking or threshold interactions."

Langmuir (1881 - 1957), an industrial researcher and Nobel laureate, is well known in the vacuum physics community for a number of significant contributions. His research areas included surface science, atomic structure, thermionic emission and electrical discharges in gases. He improved Gaede's original diffusion pump by adding chilled walls and termed the resulting device a *condensation* pump. (While Gaede's terminology is what has survived, today's diffusion pumps are all based on Langmuir's practical implementation.) The term *plasma* was coined by Langmuir and he developed the important *Langmuir probe* which is used to measure electron temperature in plasmas.

The study of pathological science occupied Langmuir for many years but he never wrote down his observations. He did, however, give a talk on 18 December, 1953 at General Electric's Knoll Atomic Power Laboratory. This talk was recorded on magnetic tape, a tape that subsequently was lost. But, in 1966 a disk transcription of poor quality was found amongst Langmuir's papers and more recently a written transcription was made by Robert N. Hall, a colleague of Langmuir's. This written transcript, from which the above quote was taken, has been published in *Physics Today* [1].

In his talk, Langmuir covered several specific instances of research which eventually fell into the category of pathological science. Some of these were consigned to that category through the personal efforts of Langmuir. First discussed was the claim by Barnes and Davis, researchers at Columbia University who, in 1929, claimed that electrons would be captured by alpha particles when their velocities were equal. Not quite fitting theory and relying on threshold level observations of scintillations with claimed observation peaks mere millivolts wide, Langmuir showed through a bit of trickery that Barnes was really "counting hallucinations, which is common among people who work with scintillations if they count too long."

Another case was that of Fred Allison of Alabama Polytechnic who, in 1927, discovered an effect which could identify new isotopes. Utilizing the 'Allison effect', a pile of new elements and isotopes were discovered. In this case it was some time before the effect, and all of the new additions to the periodic table, were shown to be imagined. (When was the last time you heard about alabamine or virginium?)

Mitogenic rays, dating from the early 20's, were emanations that were supposedly given off by plant and animal life. Like UV, these seemingly ubiquitous rays could go through quartz but not glass. This was 'proven' by observations of photographic plates exposed to the rays (coming from such things as onion roots), filtered by quartz and glass. The short of this was that it was another threshold effect dependent upon film grain and the observer's interpretation of specks on the film.

Referring to one of the then most celebrated cases of pathological science, Langmuir discussed Blondlot's N-Rays. These mysterious rays were discovered in 1903, at the time when x-rays were a hot item for research. Blondlot's rays, created by a hot filament, could *just* be detected with a phosphor screen and had the unusual property of being refracted by an aluminum prism. Blondlot also found out that almost anything would give off N-rays. This was all blown up when an American, R.W. Wood, was given a demonstration. Unknown to Blondlot, Wood pocketed the prism and Blondlot continued to get the same results. End of N-rays and Blondlot's previously distinguished career.

As a summary to his observations, Langmuir listed five symptoms of pathological science:

- The maximum effect that is observed is produced by a causative agent of barely detectable intensity, and the magnitude of the effect is substantially independent of the cause.
- The effect is of a magnitude that remains close to the limit of detectablity or, many measurements are necessary because of the low statistical significance of the results.
- There are claims of great accuracy.
- Fantastic theories contrary to experience are proposed.
- Criticisms are met by *ad hoc* excuses thought up on the spur of the moment.
- The ratio of supporters to critics rises to somewhere near 50% and then falls gradually to oblivion.

Langmuir concluded his talk with a recollection of some of his investigations into extrasensory perception and flying saucers. While not attributing the bad science he uncovered to outright fraud, ESP and UFOs represent broad areas that begin to move out of the realm of "honest" self-deception and into the shady area of pseudoscience.

Stephen Jay Gould of Harvard's Museum of Comparative Zoology explored this darker side of pathological science in an address which he gave in

1986 [2]. In this address he described four pathologies of science. These may be summarized as follows:

- *Fraud*. This covers the spectrum ranging from the use of manufactured data to outright hoaxes.
- *Finagle*. Minor hoaxes which cause a misrepresentation of phenomena resulting from 'massaged' data or intentional systematic errors in observation, description, and/or recording.
- *Propaganda*. Selective presentation of evidence to support a preestablished point of view.
- *Prejudice*. The passive equivalent of propaganda where a point of view is favored contrary to what objective evaluation would support.

Gould's own study of 'research' that supported racial differences based on brain weight and other 'intelligence differentiators' [3] is an excellent analysis of how the above pathologies played a major role in a significant stream of 'scientific' endeavor.

That these pathologies occur only reflects human nature. The important thing to remember is that the scientific method provides a mechanism to set things right, even if it takes time. However, in some cases (psi, UFOs as examples) the human desire to believe in wonderful things may take the debate beyond the bounds of normal scientific discourse. Furthermore, political beliefs or commercial ends may also introduce intentional or unintentional fraud or finagling or the premature drawing of conclusions from acknowledged (in the scientific community anyway) incomplete or overwhelmingly contrary evidence.

A useful exercise would be to pick one or more recent 'amazing' discoveries, match and track against the criteria of Langmuir, and see how the work fits into Gould's categories. You don't have to look too far to come up with contemporary examples.

References and further reading.

[1] Irving Langmuir, Physics Today, October 1989. This is Langmuir's talk, transcribed by Robert N. Hall.
[2] Stephen J. Gould, Keynote address to the CSICOP (Committee for the Scientific Investigation of Claims of the Paranormal) Conference, Boulder, Colorado, 1986.
[3] Stephen J. Gould, "The Mismeasure of Man" (Norton, 1981). A vivid recounting of the contorted research which has attempted to relate intelligence to racial differences.

- Gould's observations on pathological science are noted in an article by Donald D. Jensen, *Pathologies of Science, Precognition, and Modern Psychophysics* (Skeptical Inquirer, Winter 1989).
- Martin Gardner is the author of a number of books and articles on bad science. Good examples include "The New Age - Notes of a Fringe Watcher" (Prometheus), "Science: Good Bad and Bogus" (Prometheus) and "Fads and Fallacies in the Name of Science" (Dover). Another recommended book is James (the Amazing) Randi's "Flim-Flam! Psychics, ESP, Unicorns and other Delusions" (Prometheus).
- For where to find odd stuff, check out 'Rev.' Ivan Stang's "High Weirdness by Mail - a Directory of the Fringe: Mad Prophets, Crackpots, Kooks & True Visionaries" (Simon & Schuster, 1988).
- The March 1994 issue of *Physics Today* contains some interesting correspondence concerning a favorable review in that magazine's January 1993 issue of John R. Huizenga's book "Cold Fusion: The Scientific Fiasco of the Century". One correspondent, writing to trash the review, provided supporting recent 'evidence' in support of cold fusion. Responding were the book's reviewer, David E. Williams, and Huizenga. Regardless of where you stand on this issue, a couple of items are worth noting. First, electrochemical calorimetry techniques have improved as a result of the need to have more accurate energy measurement methods of sufficient resolution to prove or disprove the threshold effects of the reputed cold fusion phenomenon. Second, the cold fusion proponent noted the 'fantastic' energy storage of Pd as shown by recent tests (e.g. 45.1 megajoules/mole in one test). Williams shows how impressively big power numbers such as these are easily obtained when a small, seemingly positive (again, getting back to the threshold effect) power gain is multiplied by the long time duration of the experiment (maybe days or weeks) and then divided by the small volume or mass of the experimental sample. Doing that, it is easy to make microwatts per milliliter turn into megajoules/liter. Maybe looking at the meter needle at another angle would have changed this to *minus* 45.1 MJ/mole!

The last letter, from Stanley Bashkin, notes some experiments conducted by chemist Fritz Paneth in 1926 which were almost identical to those of Pons and Fleischman (except using hydrogen given that deuterium had not yet been discovered). Finding that helium was being produced by his electrochemical cell, Paneth reported success. He soon after retracted his claims when he found that his apparatus simply had not been properly outgassed. Bashkin concludes that this delayed evolution of deep lying helium in current cold fusion cells could be the real mechanism for helium evolution in these experiments, not some 'magically enhanced' tunneling process.

This article was originally presented in Volume 3, Number 2.

Some Recollections

Franklin Lee

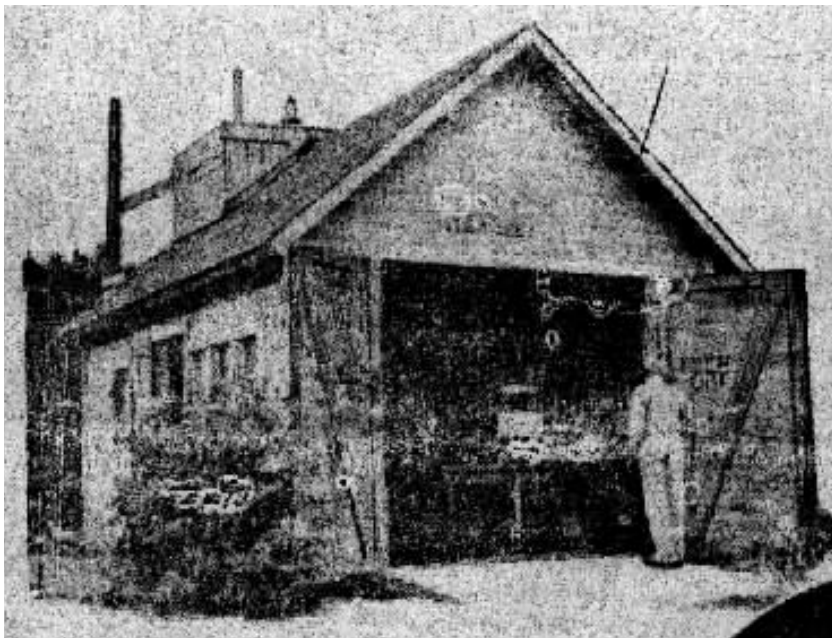
Certainly, in my case, this teacher and advocate of hands-on science was a major factor in my life's interests. Frank recently commented that he felt that he never lived up to his potential, that he could have been another Edison. Well, perhaps the world didn't need another Edison. The inspiration that Frank provided to others to do science, in my opinion, is of similar value, albeit harder to recognize and measure. The following material has been pieced together from several correspondences with Frank. - Ed.

I was born in a small town, Granite Falls, Minnesota, population 1500. For some reason that town was the home of a number of talented or famous people. There was Doctor Wellcome, one of the founders of Burroughs Wellcome Drug Company in England. There was Olai Lende, the inventor of many automobile components such as the differential, screw drive steering wheel, etc. Presidential candidate Hubert Humphrey's father had a drug store in Granite Falls before moving to No. Dakota. Senator Volsted lived across the street from Grandma Lee and Archibald Bush, a founder of 3M Company, came from Granite Falls. His charitable gifts exceeded $1 billion with $5 million a year going to Granite Falls for the schools, etc. Then there was Mildred Lee, my mother, first woman admitted to the Minnesota Bar, recognized poet and historian, and an early feminist.

My first attempt to use vacuum occurred in 1934 or 35. I was trying to make an electronic tube as my allowance was not enough to purchase a ready-made tube. I was 13 at the time and didn't realize what high vacuum was. I tried a water aspirator which got me to 30 mm Hg, the vapor pressure of water. What I got was a Geissler tube with blue light. This ended my childhood involvement with vacuum.

My last year in high school was rather turbulent. I was 15 at the time and running in all directions. I was making fireworks contrary to my parent's views. For a dollar, one could get 5 lbs. of sodium chloride at the hardware store. It was used as a weed killer. When mixed with charcoal or other combustible material it was a nice explosive. With a friend or two, I went outside town and blew up tin cans, split logs, etc. It could also be used to make colored flares. I also made a small cannon out of two pieces of steel pipe - 3/4" for the barrel which was placed within a piece of 1" that served as reinforcement. I would pack the barrel with 2" of "mix", put in a wad of cloth and insert a 3/4" glass marble as the projectile. The marble would go through 2-1/4" of pine. Once it missed the boards and went through one wall of a shed and out the other.

I did have the sense to stand behind the cannon while loading it. Once while loading it, the cannon



A photograph of Franklin Lee's Garage-Laboratory ca. 1934. In a magazine article about him this is described as the place where he "carries on his chemical and electrical research experiments. A network of charged wires keeps out the loiterers."

went off and took one finger and messed up my hand. Dad made me clean house. This occurred again when I was caught bringing tear gas to school.

A more constructive endeavor was the analytical balance I made at about this time. The fulcrums were razor blades, the weights were nickels (about 5 gms), short lengths of rolled-up wire (1/10 to 2 gms) and pieces of foil (1 to 50 mg). I got interested in electronics at about this time.

The next project that involved vacuum was when I became employed by Union Carbide in the research department. This was around 1943. The project involved some work with a vacuum furnaces. The furnace consisted of a 1-inch diameter ceramic tube with a boat of high carbon ferrochrome, chromium oxide and calcium silicate flux. The idea was to get the carbon in the ferrichrome to react with the oxygen in the chromium oxide. The vacuum atmosphere was to improve the equilibrium. While the project resulted in a production process, it was soon displaced by a better method.

It wasn't until rotary compressors became commonplace that I realized there could be an opportunity for an amateur system. When I started teaching chemistry at Erie Community College in 1953 I had lots of opportunity to make apparatus for the school lab. One item was a molecular still which would fractionate mixtures like vegetable oil at temperatures low enough to avoid decomposition. I didn't spend much time on this as it was sort of dull.

When C.L. Stong took over the *Amateur Scientist* column he suggested an article on a particle accelerator. I don't know how he got the idea, although Albert Ingalls, the previous editor of the column, had produced an article on an amateur-built cyclotron. This used commercial vacuum apparatus. Trying to make due with something simpler, I got a lot of used refrigeration compressors and tried them out. A local glassblower made me a McLeod Gauge (based on information in "Review of Scientific Instruments" - there are lots of interesting articles in that journal). The high vacuum mercury diffusion pump was also based on material from "Review of Scientific Instruments." The resulting article, "A Homemade Atom Smasher," was published in May of 1957. The machine consisted of a 350 kV van de Graaff electrostatic generator with an accelerating column that would produce a beam of electrons.

Later in 1957 I started supplying Van de Graaff terminals to hobbyists who had read my article in Scientific American and one thing led to another. After the accelerator article came out I was ready for requests for information and supplies. My wife did not take kindly to my hobby as it cluttered up the cellar, drew on my time and involved a lot of mail and phone calls.

My friend Morris Most had a sewing machine company that was not doing well and he was willing to share my responsibilities. He took care of the mail, bookkeeping and shipping & receiving. I recognized a need for science education equipment and started adding this to the line. There was a big market then and I went after what Science Kit wanted but couldn't get from the other suppliers. This piece of the business soon eclipsed the amateur market which remained more of a hobby than an enterprise.

When it came to having a turret lathe, punch press, two drill presses and seven assistants in the cellar two nights a week my wife again suggested that I clear it out. By 1975 Morris and Lee had a part time crew of 10 and a turn-over of about $1M in current dollars.

Morris died in 1977 and I quit teaching to take over. The business was a mess by this time and it took some 2 years to get back to profitability. In 1984 my older daughter and her husband took the plunge and came into the business. Ray and Nancy proceeded to cull out all the products of marginal profit and reduced the product line from about 200 items to about 50. The company changed its name in 1992 to Science First and is fairly sound with only 12 employees and a $1M turnover. Competition is coming mainly from the Far East and I see a real challenge ahead. Fortunately we have a large investment in high tech tooling which, for now, allows us to compete.

Returning to the amateur products, the Morris and Lee customers were a mixed lot. Some were experimenting with gravity devices - I tried to talk them out of making purchases. Some were very talented with lots of money to spend. One ambitious project was a full scale particle accelerator that must have cost $5000 to build. All were people who wanted to see things happen.

All of the amateur business was dropped in 1984. I did continue to sell the 500,000 volt Van de Graaff kit but stopped advertising around 1990 and I only sell 5 or 6 a year by word of mouth. We do sell 200,000 and 500,000 volt generators through dealers like Edmund Scientific and some hobby shops.

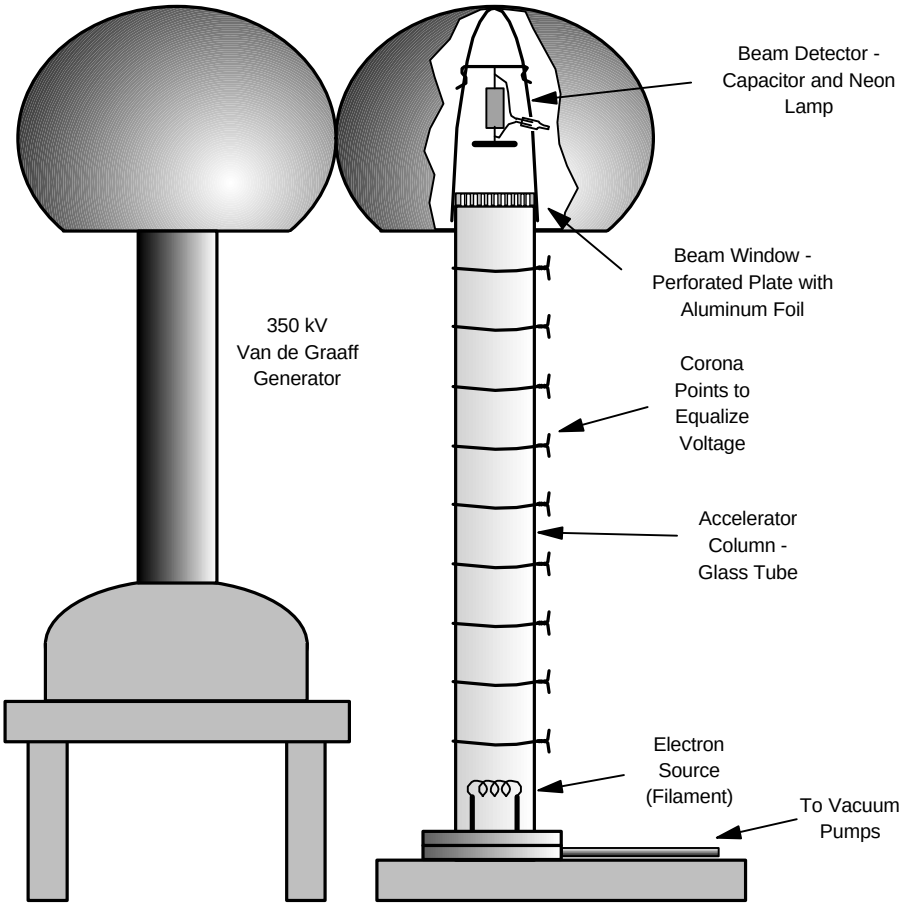
Scientific American pretty much dropped the Amateur Scientist column around 1973 after Stong died. Virtually all the amateur business I had came from his articles since he had included my name and address. I later made gas chromatographs, electronic balances, spectrophotometers, air liquifiers, etc. but just for my own entertainment.

One of the big differences today is a lack of supplies. Fear of litigation has made suppliers unwilling to deal with amateurs. The fear of law suits has also diverted some nice stuff away from people into the dump. Its simply hard to buy something that you could possibly

get hurt on. Surplus dealers are one remaining good resource - they get the original supplier off the hook to some degree. Scientific American once published an article on making amateur rockets using zinc powder and sulfur as fuel. I think they got chewed out by some group for that one.

Also, we have become a nation of button pushers. I feel that one reason for a lack of interest in such projects is the availability of computers - many people prefer pushing buttons to using tools. (*Certainly, the Morris and Lee kits required a good degree of competence on the part of the purchaser to put something together that worked. It took me a couple or three years to really get my Hickman diffusion pump to actually do something...but I learned a lot in the process. There seem to be fewer people now who are willing to really work to make a project go, but they are there and they seem to feel like the group that business forgot. If they didn't exist, 'the Bell Jar' would have no readership.* - Ed.)

This article was originally presented in Volume 4, Number 4. It really hit a chord as the following correspondence indicates:



Frank Lee's Electron Accelerator (1957). The beam tube was a glass cylinder. The pump-out port and filament were located at the bottom, grounded end of the tube. The beam exited through a window of 1 mil aluminum foil, supported by a perforated aluminum plate. Beam detection was accomplished by means of a metal plate that was connected to a neon bulb and capacitor, arranged in parallel. The charge collection rate was indicated by the flashing frequency of the bulb. The pumping system consisted of a glass mercury diffusion pump backed by a pair of refrigeration compressors operated in reverse. Pressure measurement was accomplished with a McLeod manometer. An alcohol/dry-ice trap prevented mercury from backstreaming into the accelerator.

Feedback:

Norman Stanley of Rockland, ME writes "It was most interesting to read Franklin Lee's reminiscences as I remember well that article in *Modern Mechanics* about the youthful electrical genius. As an impecunious 18-year-old electrical and chemical experimenter back then, I was impressed enough to save that issue (and still have it somewhere in my attic). However, I never realized that he grew up to be the Lee of Morris and Lee, from whom I bought one of those 500 kV Van de Graaff kits. I still have the kit; what with marriage and other distractions I never got to put it together. I'll have to dig it out and do that real soon now!"

Bill Powell of Manhattan Beach, CA writes "I particularly enjoyed the Franklin Lee recollections. When I was young I would dream over articles about science projects a boy could build for "under $25". The trouble was I had never had that much money all at once. Later in life I was too busy working to do much. But now I am retired and have managed to keep back the $25 from the government so this is my chance to relive the "Morris and Lee" days. I fool around with computers and all that but it's not the same. I need the "roar of the forepump and the smell of the Octoil!" I may not be alone in all of this as the content of "our" magazine seems to hit all the right buttons."

Richard Hull (Tesla Coil Builders of Richmond) wrote "I was in high school when (Frank Lee's) atom smasher article appeared in the Amateur Scientist. I cut my teeth on the wonderful articles in the back of those old Scientific Americans. Alas, those days are gone. I pay hell trying to get a few chemicals for experiments. I had to agree to pick up a gallon of benzene the day it arrived at the distributor. They refused to keep it on site and really didn't want order it for me....my mom used to clean the rugs with the stuff and she's still full of life at 91. ....With the government and local agencies just wanting to protect the hell out of us, thank goodness there are Hamfests."

Home-Made Geissler Tubes

Or - A Simple and Highly Dangerous Way from Yesteryear to Make Vacuum

F. Castro

This article is from The Experimenter, *December 1925. It is now contained in an interesting little compilation of old articles that is available from Lindsay Publications under the title of* Double Tesla-Oudin Coil - Selected Articles from The Experimenter Magazine (Catalog #817). *The article is reprinted with Lindsay's permission.*

Today, we vacuum experimenters are spoiled by the broad availability of good and reasonably priced vacuum pumps. This, in large measure, is due to the prevalence of vacuum in industrial applications. Volume production of pumps translates to price competition and a healthy used market. In 1925 vacuum pumps were very rare. This continued until fairly recently, hence the incentive for Franklin Lee to outline ways of converting refrigeration compressors to usable vacuum pumps for technical applications.

I guess the following is somewhat similar to the classic vacuum demonstration where water is boiled in a metal can. When the container is filled with steam it is removed from the heat source and the lid is screwed on the can. The condensing water then creates a vacuum which collapses the can. In this article, a glass tube is filled with hydrogen and oxygen, in the proper proportions to create water when a chemical reaction is induced. This will indeed produce a moderate vacuum, limited by the vapor pressure of the contained water vapor. However, the process is (to say the least) a bit on the dangerous side. Hence, this article is offered as a piece of history, not as a practical method to make vacuum. Please, don't try it. - Ed.

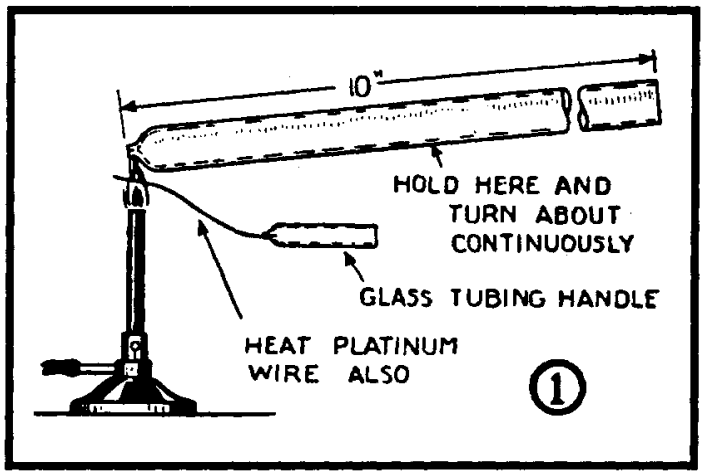
A novel and simple method for constructing at home and at small cost some of these beautiful tubes is illustrated. The apparatus required is that owned by any amateur chemist, and the materials reduce to thick glass tubing, some common and cheap chemicals, and about three cubic inches of mercury. The platinum wires required can easily be made out of the plate and grid of a discarded radio tube.

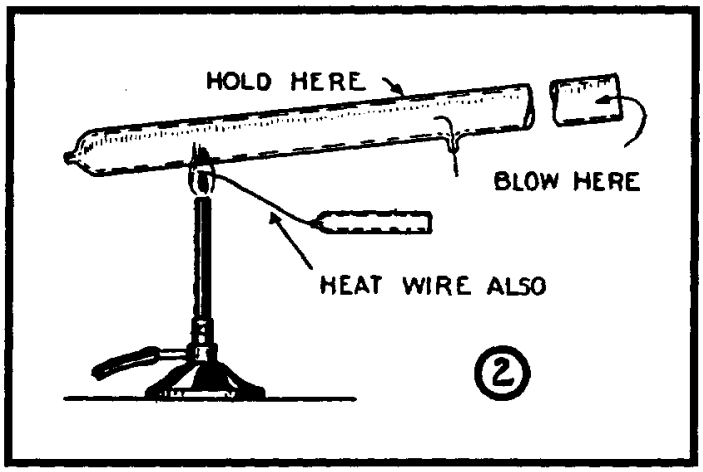
Figs. 1 and 2 show how to seal the platinum wires through the glass; in both cases the heating should continue until it is firmly sealed thereto. In Fig. 3 a simple and effective form which can be given to the tube is depicted.; the bulbs are easily made by blowing and turning the tube about, while heat is being applied. Other forms can be easily given, if only a little care and ingenuity is exercised. All depends on the manipulator. The next step (Fig. 4) is to fill the tube with mercury and note the volume required to completely fill it. To do this take the capacity of a small bottle or beaker as a unit and calibrate the mercury dish by pouring that known volume of water in it, and making a scratch

a file where the level of the water reaches each time. Then displace exactly two-thirds of the mercury with hydrogen, and the rest with oxygen. Both of these gases can be generated as shown.

Now the tube is sealed as shown in Fig. 5; the flame is directed with a blowpipe or a piece of narrow glass tubing connecting with the large tube; while applying heat it is slowly twisted until the sealing is complete.

Last, clamp the completed tube as shown in Fig. 6 and connect the two upper platinum wires to the secondary terminals of a spark coil; get at a safe distance (as the slight explosion may break the tube), and close the primary circuit. The spark will cause the rapid combination of the two gases, leaving only a very small quantity of one of them (it is never a mixture of both) and a tiny speck of water. This produces the high rarification necessary in the Geissler tubes. If now one of the upper wires and the lower on are connected to the secondary of an induction coil or to an influence machine, beautiful rings of light will appear in the tube.





If the tube is of sufficiently thick glass to stand a rather violent explosion, a better plan is to fill it completely with mercury, invert it open end downward in a cistern of mercury and introduce electrolytic gas evolved by the decomposition of water by a battery until the mercury is all displaced. The electrolytic gas should be dried before being introduced, which is readily done by passing it over soda lime or other drying agent. When the tube is full and sealed off the gas may be exploded, as described in the preceding.

There is some danger that the hot glass may bring about a premature explosion, so as much moderation as possible is to be exercised in the application of the blow-pipe flame.

Other gases were experimented with but the above seem most satisfactory.

This article was originally presented in Volume 5, Number 2.

Appendix: Some Suppliers

Suppliers come and go, especially the smaller, more specialized ones. A more current list of suppliers may be found on the tBJ web site.

Vacuum Equipment and Components

Duniway Stockroom Corp., 1305 Space Park Way, Mountain View, CA 94043
A good supplier of new and rebuilt vacuum equipment, fittings, components and supplies. Catalog.

Kurt J. Lesker Company, 1515 Worthington Ave., Clairton, PA 15025
Mentioned frequently in "the Bell Jar", Kurt J. Lesker's catalog is comprehensive and is chock full of useful tables and charts. Of note is a new line of evaporation sources in addition to the broad selection of fluids, pumps, and hardware.

E. McGrath, Inc., 35 Osborne Street, Salem, MA 01970-2599
Specializing in used and rebuilt vacuum equipment and components, this company has supplied much of the author's equipment base. A catalog is available.

MKS Instruments, Inc., Six Shattuck Rd., Andover, MA 01810, (978) 975-2350
Process control instruments, gauges, flow controllers, vacuum components and fittings. MKS also has many reprints of technical articles relating to vacuum instruments and vacuum processes.

Related Materials and Supplies

Eurocom Imports, Inc., 1937 Westridge, Irving, TX 75038, (214) 753-1110
Supplier of quality European equipment and components for the neon sign trade. A request will bring an informative catalog and some sample electrodes.

OE Technologies, P.O. Box 703, La Madera, NM 87539
Quality surplus supplier of optical, nuclear, vacuum and high voltage components and equipment. The prices are good and some of the items (scintillators, ignitrons, laser mirrors) are quite unique. A listing is available.

Glassware

ACE Glass Incorporated, P.O. Box 688, Vineland, NJ 08360
Complete line of fabricated scientific glassware including the "Ace-Thred" line which was featured in the Spring 1993 issue of "the Bell Jar". While not specifically a supplier of vacuum hardware, some very useful adaptations of ACE's stock items are possible. ACE will do custom fabrication.

Wale Apparatus Co., 400 Front Street, P.O. Box D, Hellertown, PA 18055-0201
Wale has several catalogs which cover glassware, glassworking apparatus, and supplies. The two most useful catalogs for the amateur are *Glassworking & Laboratory Products* and *Standard and Special Glass Products*. A wide selection of stuff and you don't have to buy an entire crate of a given item.

Radiation Detectors and Monitors

Aware Electronics, P.O. Box 4299, Wilmington, DE 19807, (302) 655-3800
Aware's RM-60 Geiger counter is a small device that interfaces directly to an IBM compatible computer via a phone type cable to the serial or printer port. A dedicated PC is not required as the software gathers the data and stores it to disk even while the computer is running other applications. The software displays the data in a scrolling bar chart format with date and time for each bar. Also provided is the cumulative average dosage. Cost for the RM-60 package is about $150. Aware's catalog also describes several other radiation monitoring items.

Landauer, 2 Science Road, Glenwood, IL 60425-1586, (708) 755-7000
This company provides film and thermoluminescent (TLD) dosimeters as part of their service. These are provided as either wearable badges or as room monitors. To sign up for the service you select a monitoring frequency (weekly, monthly, quarterly) and pay a small set-up fee plus a year's payment in advance. Before the end of each monitoring period, you receive a new badge. At the end of each period you send in the current badge and within 5 days you receive a report giving dosage for the period plus cumulative dosage. For a TLD dosimeter sensitive to x-ray, gamma, and beta radiation with a quarterly schedule, the cost is under $100 for a year.

Technical Book Dealers - Rare and Reprints

Lindsay Publications, Inc., P.O. Box 538, Bradley, IL 60915-0538
Specializing in technical books on mechanical and electrical topics, Lindsay's catalogs are treasure-troves of good material. This company has a wide selection of reprints of old and obscure books as well as newer material. On top of this, Mr. Lindsay's humor is truly sick.

New Wireless Pioneers, P.O. Box 398, Elma, NY 14059
New Wireless maintains a stock of old electrical and radio books. They also have an interesting video "In Quest of the Light; Visible and Invisible" that shows a wide range of antique discharge and x-ray tubes. Some apparatus is also available for sale including vintage Geissler tubes.

Rainy Day Books, P.O. Box 775, Route 119, Fitzwilliam, NH 03447, (603) 585-3448
Frank Bequaert's used and rare book store in rural New Hampshire specializes in used and rare technical books. While much of the stock is associated with electronics and amateur radio, a conscious effort is made to find and stock vacuum related books. Catalogs are available for a 32¢ stamp. The shop is seasonal (April - November) but the phone always works.

Electronics and Electromechanical Components, Misc. Surplus, Materials

American Science and Surplus, P.O. Box 48838, Niles, IL 60714-0838
Mostly small items are contained in this company's catalog. However the diversity is tremendous and frequently you will find that 'critical item' that's needed to finish off a project. From sticky tape to optics to small pumps and rubber chickens, if it's odd-ball surplus they will have it at one time or another.

C&H Sales Company, P.O. Box 5356, Pasadena, CA 91117-9988
Lots of electro-mechanical and optical surplus, much of which is in like-new condition. C&H also has a good supply of miscellaneous high voltage energy storage capacitors. The inventory on these tends to vary rapidly so a note or phone call is advisable.

Fair Radio Sales, Inc., P.O. Box 1105, 1016 E. Eureka St. Lima, OH 45802
A long time supplier of real military electronic surplus, Fair Radio has a good selection of transformers, chokes, capacitors, insulators, etc.

Herbach & Rademan, 18 Canal St., P.O. Box 122, Bristol, PA 19007-0122
H&R's catalog has a great collection of electrical and electromechanical surplus including laser and robotics components and a wide selection of motors.

Shapiro Supply Co., 1259 Delaware, St. Louis, MO 63133, (800) 833-1259
Small friendly company that sells metals (aluminum, steel, brass, titanium) in various forms.

Small Parts Inc., 13980 N.W. 58th Court, P.O. Box 4650, Miami Lakes, FL 33014-0650, (305) 557-8222
Everything in precision hardware, materials and tools. Go here if you can't find it anywhere else but don't expect any bargains.

United States Plastic Corp., 1390 Neubrecht Road, Lima, OH 45801, (800) 537-9724
All forms of plastic materials and products. Of particular interest are the sheets, rods and tubes of polyethylene (regular, high density and UHMW), Teflon, Delrin, Nylon, PVC, styrene and polycarbonate. They also carry lipless stainless steel beakers that are useful for making small vacuum chambers. Ask for the full catalog.