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Papers

A Method for the Preparation of Rubidium Absorption Cells

by
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Abstract:
Absorption cells, using primarily elements from the alkali or halide groups, are a staple in today's laser labs. Researchers rely on them as reference cavities for frequency locks, for creating and maintaining optical frequency standards, and even as the science cells themselves, where researchers make precision spectral measurements and characterize spectral lines.

This paper describes a method suited to the preparation of borosilicate glass cells filled with alkali metals, in this case, rubidium. In order to obtain a useful cell, the glassblower must observe sound ultra-high vacuum practices. Care must also be taken to maintain the optical quality of the cell's windows, in order to minimize distortion of the laser beam.

Introduction

The glassblowing techniques required for the construction of borosilicate absorption cells are rudimentary. However, the subsequent distillation of an alkali metal into the cell is not a straightforward procedure. The cleanliness of the cell, the quality of the vacuum, and the ultimate purity of the rubidium are all-important aspects in the success of the final product.

Consider the case of the reference cell used, for example, to lock a laser to the specific frequency of 780 nm. If the cell is not clean, or if the vacuum is poor at the time the cell is filled and tipped off, the resulting frequency absorption peak, although centered on 780 nm, will be too broad to be of use. The modern glass shop should be equipped with a vacuum system capable of obtaining ultra-high vacuum and so afford the glassblower the capability to make and fill high quality absorption cells.

Vacuum equipment need not be prohibitively expensive. Good, used equipment can be had from a variety of sources. There is, however, no substitute for sound vacuum practice. Careful planning in the beginning will ensure a versatile and serviceable system. Vacuum fundamentals and system design are beyond the scope of this paper. However, O'Hanlon[1] and Roth[2] are two of many good references.

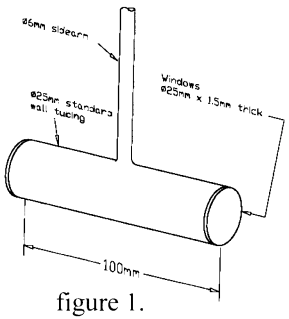


figure 1.

Cells

Researchers typically specify the form and dimensions of the cells based on their requirements. The cell described below is only one possible configuration, albeit a common one. Figure 1 shows the cell configuration researchers favor for rubidium and cesium absorption cells. Although cells may be constructed of anything from quartz to aluminosilicate and even sapphire, this particular cell is made of Corning's 7740 (Pyrex®), as are the windows.

The cells are constructed of 25 mm standard wall tubing, 100 mm long, to which is attached a 6 mm side-arm. The windows are fused to the ends of the cell in the following manner:

• Cork one end of the cell, chuck it up in the left (stationary) spindle, and use the side arm for blow tube access.

• Mount a 20 mm diameter carbon vacuum fixture in the right (traveling) spindle chuck, and, with the pump running, stick a window to it.

• Bring the window close to the tube to facilitate centering. Move it off and fire polish the end of the tube.

• Bring the window back to within a few millimeters of the tube, and, being careful not to splash the flat surfaces with the flame, fire polish the edge of the window.

• Direct the flame back on to the end of the tube and let it melt until it starts to thicken at the very edge.

• Run the window into the melted glass and carefully work the seal between the tube and the window, directing the flame in such a way as to avoid flame splash on the window. The carbon vacuum fixture will protect the window to a point, but if it gets too hot, it will also compromise the optical quality of the window.

• A slight puff to the blow tube, as required to maintain the tube diameter, is all that is necessary. In many cases, the joint can be made without blowing, but I usually have the blow tube connected, just in case.

• Turn off the vacuum pump and move the tailstock spindle away from the window now attached to the cell.

• Carefully flame anneal the window and about 20-25 mm along the length of the tube.

• When it has cooled, turn the cell around, remove the cork and attach the second window in the same manner.

• Furnace anneal cells upon completion.

Wheeler[3] also describes a similar technique in his classic volume on the subject of scientific glassblowing.

Keys to Success

In the case of the cells, the most important aspect is the windows. A robust cell with good flat, but dirty, windows is of little use. Clean the windows with distilled methanol, or by any other means that assures cleanliness, and handle them thereafter with gloves. Use a desiccant for the blow tube, and make sure the vacuum line to the pump is properly trapped to prevent any pump oil from back-streaming up the line and onto the windows.

Use hydrogen as a fuel gas. It is easier to get a more intense flame to work the corner, and it can quickly be converted to an annealing flame by simply turning off the oxygen. However, a hydrogen/oxygen flame will boil the glass much more readily, and so some pre-heating is advisable.

The most robust cells will have a slight fillet in the inside corner. An acute re-entrant will severely threaten the integrity of the cell.

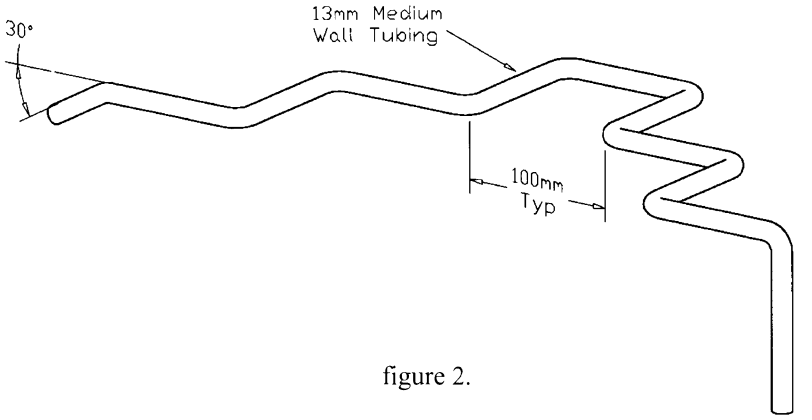


figure 2.

Manifold

The manifold is a means to fill cells, ampoules and break-off tubes with various amounts of the alkali metal using simple vacuum distillation techniques. In this case, the manifold itself is used once and then discarded. It is constructed from a single length of 13 mm medium wall tubing. After many different configurations, I have settled on this variation, shown in figure 2. A manifold of these dimensions works well for ampoules, break-offs and cells 100 mm or shorter in length. The manifold dimensions can be adjusted to meet other sizes. The many 90° bends afford sites to attach cells etc. to be filled. The 30° downward bend, to which a stainless steel glass/metal seal is attached, assures that the rubidium source ampoule remains toward the bottom of the tube. This is a help when it comes time to crush the ampoule. The final 90° downward bend attaches to the vacuum system.

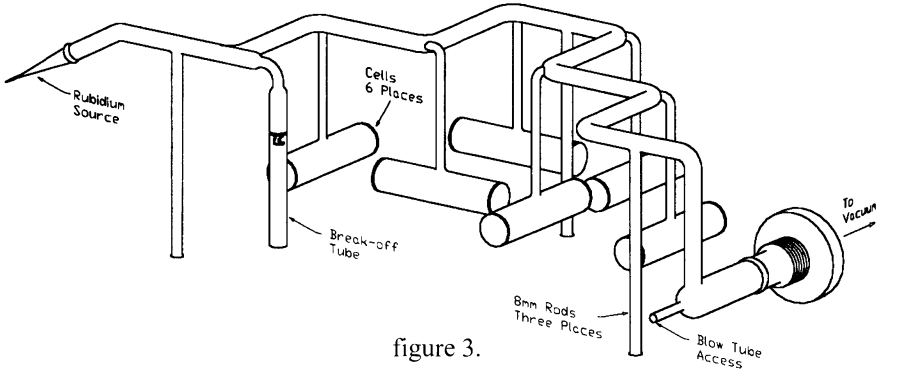


figure 3.

Figure 3 shows the manifold with its complete complement of cells, support rods, break-off tube and glass/metal tube containing the rubidium ampoule, all attached to the vacuum system, ready to have the oven built around it in preparation for evacuation and bake-out.

The easiest sequence I have come up with runs as follows:

• Mount the manifold in a rack in such a way as to give easy access to the sites where the cells etc. will be attached. A couple of ring stands with clamps work well.

• Plug one end and attach the blow tube to the other. Again, I recommend the use of a drying tube to prevent moisture from your breath from entering the manifold.

• Determine the length of the 8 mm rods, and attach them as shown.

• Attach the cells and break-off tube at the 90° bends as shown. Leave the cells sticking straight out, i.e. do not put the bends in the 6 mm side-arms.

• Remove the manifold and set it on its support rods on a flat surface. Using a bushy annealing flame, heat the 6 mm side-arms near the junction with the manifold and allow them to gently take a bend until the axis of the cell is parallel with the surface. You may have to twist them or otherwise adjust them to coax them into the orientation shown. The orientation shown allows for the largest number of cells and the smallest size oven.

• Furnace anneal the manifold with rods, cells and break-off attached.

• Attach the blow tube to the blow tube access at the vacuum side. Blow out a bubble and prepare it as you would to make a tee seal.

• Situate the manifold on an insulating layer. The one I use is 18"x36"x2". Bring the vertical member of the manifold into position over the spot prepared for the tee seal.

• Plug the end of the manifold with the 30° bend, and complete the tee seal to the vacuum side.

• Score a 1 gm rubidium ampoule with a glass knife to facilitate crushing. Take care not to break it. Rubidium will spontaneously combust when it comes in contact with air.

• Unplug the manifold, insert the ampoule into the welded up glass/metal seal, and attach it to the end of the manifold.

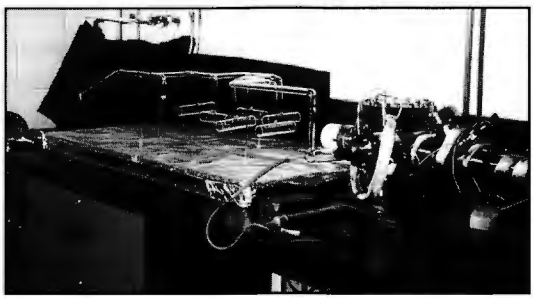


Photo 1.

Photo 1 shows the manifold with the blow tube still attached, ready for tip-off and evacuation.

• Remove the blow tube and seal up the access tube.
• Begin pump down.
• Spark check for leaks.

Keys to Success

Cleanliness is paramount. Use a desiccant tube somewhere in the blow tube.
Warm the tubing around the area of the seals to prevent water vapor from forming inside; this aids in pump out and denies contaminants a place to accumulate.

Rubidium Sources

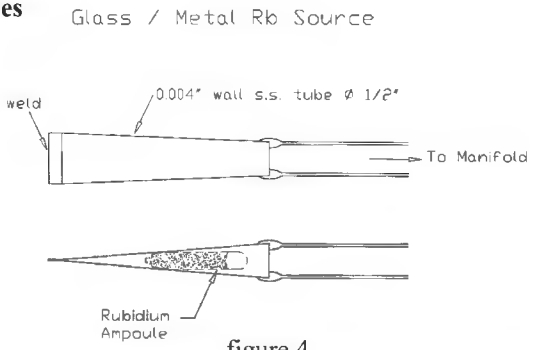


figure 4.

Figure 4 shows the schematic of a glass/metal rubidium source for a one gram ampoule. The ampoule is crushed by means of a C-clamp or vice grips in order to liberate the rubidium for distillation.

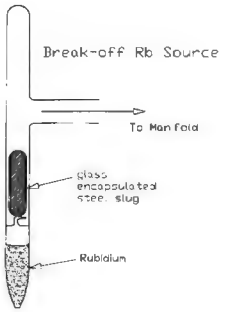


figure 5.

Figure 5 shows the schematic of one possible configuration of a break-off type source. I always fill one of these when I start with a one gram ampoule in the above-mentioned source. A one gram ampoule provides enough metal to fill all the cells, leaving sufficient metal for at least one more run.

Attach either of these to the end of the manifold furthest from the vacuum system. The rubidium source must remain outside the oven during bake-out.

Heater and Oven

The heater is a 16 ohm ceramic plate element set on 10-15 mm lengths of quartz tube. The quartz spacers keep the element off the surface of the insulating layer. Photo 2 shows the position of the heater. Make sure the electrical leads will reach outside the oven.

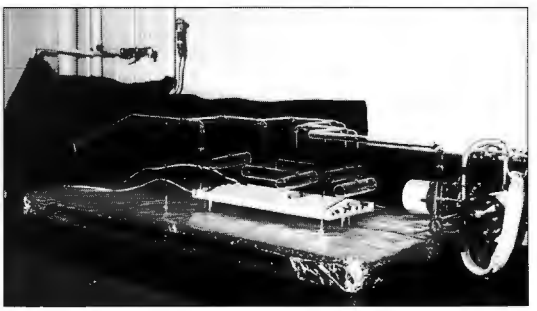


photo 2.

The oven is constructed of fire brick built up around the manifold. Wrapping the fire brick in aluminum foil is helpful for controlling the dust from the fire brick. Fire brick can be sawed or easily fashioned with a rasp to obtain the occasional odd size or shape necessary to complete the enclosure. Notches are required for the vacuum side as well as the source side. Photo 3 shows the first course of bricks in place. Photo 4 shows the start of the second course. Notice that the rubidium source is outside the oven.

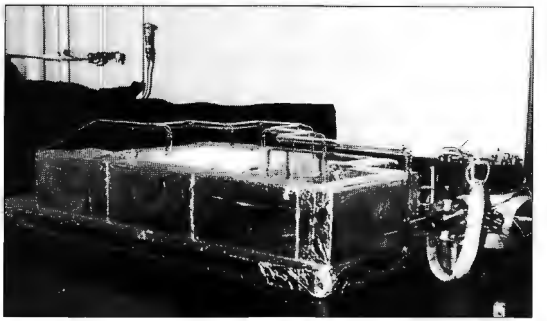


Photo 3.

Photo 4.

Situate thermal couples in the oven, one at the center and one at a corner. The temperature will vary between the two, and it is useful to have some idea of the gradient from center to corner.

Once the second course of bricks is in place, cover the oven with aluminum foil and then with a 1 mm thick aluminum sheet. Finally, place fire bricks on the aluminum sheet to provide an insulating layer on top. Photo 5 shows the completed oven.

Photo 5.

Vacuum Bake-Out

Once the vacuum pressure reaches the 10^{-6} torr region, you can begin bake-out. Connect the heater leads to a variac and start with 40-50% power. While monitoring the pressure, slowly bring the oven temperature to 350° Centigrade. The pressure will rise as the temperature increases, but it will level off and then slowly begin to drop again. A drastic rise in pressure (greater than 10^{-4}) probably indicates a leak. While it is unlikely that a leak or crack will develop during bake-out, it is not unheard of. In this unfortunate event, the bake-out must be immediately aborted, the system vented, and the leak found and fixed.

Pyrex and its equivalents can safely be baked to 450° Centigrade. It is important that the thermal couple recording the lower reading (usually the one in the corner) reach at least 300°. Water vapor is the largest constituent and the last molecules do not leave the walls of the glass until the 300° threshold is reached.

A Residual Gas Analyzer (RGA) is helpful in ascertaining the vacuum quality and identifying its constituents. However, it is not necessary. A vacuum bake at 300°- 400° for five days, in most cases, assures a clean manifold and cells. In any case, the pressure at the end of the bake-out should drop to no higher than the low 10^{-8} torr region. After the heater is turned off and the system is allowed to cool, the pressure should drop to 10^{-9} torr or better.

When the oven is again at room temperature, it can be dismantled and the preparations for the distillation and subsequent filling of the break-off and cells can begin.

Keys for Success

It is a good idea to bake the rest of the system at the same time the manifold is baked. Make sure the manifold remains at a higher temperature than the rest of the system. This assures that no residuals will deposit on the manifold or cell interior surfaces.

Include as much of the manifold in the oven as possible. This assures that the greatest amount of water vapor will be baked and pumped off before distillation.

Keep a notebook. Having a record of each run will help document problems and successes. Date, time, temperature, pressure, variac setting, etc. are useful to record, in order to duplicate a successful run.

The juncture to the vacuum system should be a glass/stainless steel bellows assembly. This affords the glass system a place to flex, thereby decreasing the threat of breakage.

Distillation and Fill

In order to begin the distillation and filling process, it is necessary to liberate the rubidium. In the case of the glass/metal source, crush the rubidium ampoule; in the case of the break-off source, using a magnet, lift the glass encapsulated steel slug and drop it on the tip. There will be a quick pressure rise as whatever gas remaining in the ampoule is liberated. This should be quickly pumped away, and the pressure should return to its previous level.

I have found that a C-clamp works best to crush an ampoule in the stainless steel tube type source. There is always some vibration, and I find I have more control than with a pair of vice grips. This is where the addition of the stainless steel bellows proves its worth. In any case, use heavy paper or cardboard to protect the thin-wall tubing from puncture.

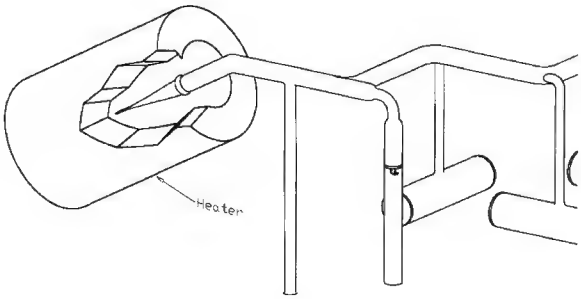


figure 6.

A cylindrical heater drives the rubidium out of the source. It also keeps it warm, thereby preventing the rubidium from re-condensing there. Figure 6 shows the schematic of the heater in place around a glass/metal source.

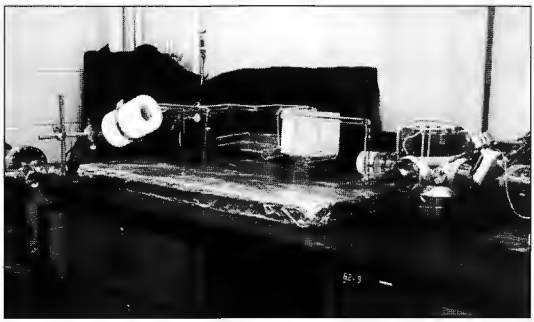


Photo 6.

Photo 6 shows the system at the onset of distillation. Notice the two ring stands: one holds the heater in place, while the other holds a thermal couple probe to measure the temperature inside the heater. Once the temperature rises sufficiently to drive the rubidium out of the trap, about 190°, it begins to condense on the nearest room temperature surfaces, as seen in Photo 7.

Photo 7.

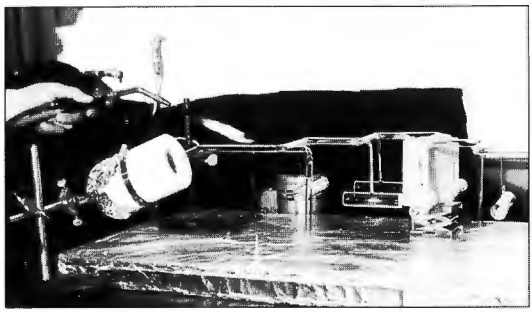
Elements with lower vapor pressures will be pumped away, and those with higher left behind. This occurs as the source is emptied, as well as when the metal is moved when filling the cells.

Move the rubidium along the manifold by means of a bushy annealing flame. Since the rubidium condenses on the nearest room temperature surfaces regardless of the pumping

direction, it is most convenient to fill the break-off first and then the cells, one by one, along the manifold toward the pump. Photo 8 shows the break-off and all the cells filled, as well as the flame used to do so.

Finally, the break-off and cells are tipped off. It is useful to have someone to record the pressure of each tip-off, and mark it on the cell. The lower the pressure maintained during tip-off, the better.

With the last cell tipped off, the last thing to do is tip off the manifold from the vacuum system. The spent manifold contains hazardous waste, and should be disposed of accordingly.



Keys to Success

Moving the rubidium along the manifold is akin to trying to bathe a cat: there seems no end to the ways it can elude you. Keep in mind that the rubidium will move to the nearest cool surface, regardless of pumping direction. This means keeping the site cool where you want the metal to condense, while keeping everything around it warm. Chasing the metal by running the flame parallel to the axis of the manifold tube, while taking care to avoid the bend in the cell side-arms, will allow the rubidium to condense there. It will then either run into the cell on its own, or can then be encouraged to do so by gentle warming. A bead of metal the size of the head of a hat pin, is more than sufficient for each cell.

Do not put a cell at the last possible 90° bend site. Leave the last horizontal 90° bend free. Not trying to chase rubidium toward the last bend helps ensure that you keep your pumping system as free from alkali metal contamination as possible.

Thickening the wall of the manifold above the tee seal will aid in tipping the manifold off the system.

Acknowledgments

A special thanks is due Seth Wieman for his expert help with the drawings, and Kurt Vogel, for his helpful discussions.

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3. E.L. Wheeler, *Scientific Glassblowing* (New York: Interscience Publishers, Inc., 1958), pp. 135-138.

Construction of Quartz Flanges Via Lamination

by
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Abstract

The intent of this paper is to present a detailed explanation of the process of fabricating a quartz flange using a lamination process. Tubing sizes, carbon tools and burner selection will also be discussed as well as optional O'ring grooves.

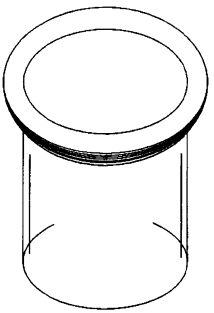

figure 1.

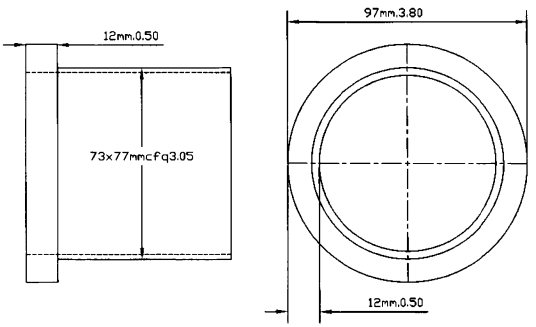

figure 2.

Introduction

Over the years, glassblowers have been able to purchase over-the-counter components to quicken the fabricating process of certain pieces of glassware, e.g., molded spiral surfaces for the inside of Friedricks condensers, O'ring joints, flanges, etc. However, from time to time for one reason or another, we find ourselves in a position where either time is of the essence and we cannot wait for delivery or we need an "off" size not available or, if so, at great cost to the consumer. It is at these moments that the scientific glassblower has an opportunity to show his expertise and value for that one-of-a-kind item.

Method of Construction

To make this particular flange, three different sizes of quartz tubing are used: 73 x 77mm, 80 x 85mm and 90 x 95mm (Fig. 3). Each piece of tubing should be at least one foot in

length; this will allow a comfortable working area as well as reduce any overheating of your chucks.

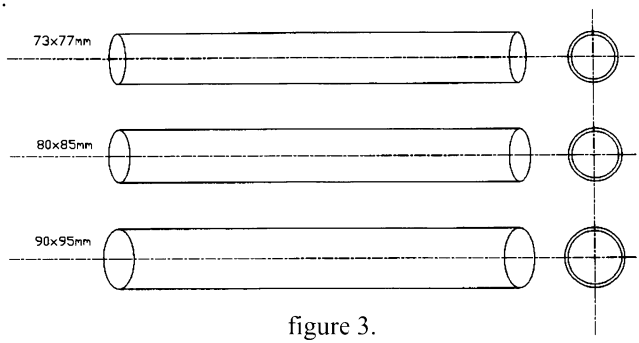

figure 3.

The first step is to chuck up the 73 x 77mm tube in the head stock where it will remain during the entire operation. The next larger size of tubing, 80 x 85mm, will be secured in the tail stock. Making sure that both tubes are running as concentric as possible, roll in the tail stock so that the 80 x 85mm tube overlaps the 73 x 77mm tube by at least 10 to 15mm (Fig. 4). Using hydrogen and oxygen as fuel and a six-fire single-jet cradle burner with 13mm O.D. burner, begin the first lamination. Starting from the left side, or rear, of the flange, heat the 80 x 85mm tubing to the point where you can paddle it down into the 73 x 77mm tube, being careful not to distort or reduce the inner diameter of the 73 x 77mm tube. As the larger tube begins wetting onto the smaller tube, advance the cradle burner slowly to the right to continue this process using a paddle when necessary to insure contact between the two pieces of quartz. It is important to move the cradle burner and carbon paddle slowly across the tube so as not to encapsulate any air pockets between the two pieces of tubing. If at any time during this process the inner diameter starts to collapse, increase the speed of the lathe to spin out the tubing to its original diameter.

After a satisfactory lamination has been attained, the excess 80 x 85mm tubing at the outer edge of the flange may be either fire cut, or, after cooling, cut off on a wet saw. The use of a diamond blade in this part of the operation is preferable. If the fire cutting method is used, do not worry about the semi-ragged edge; simply heat this area and paddle down into the face of the flange as this will be cut away later on the wet saw.

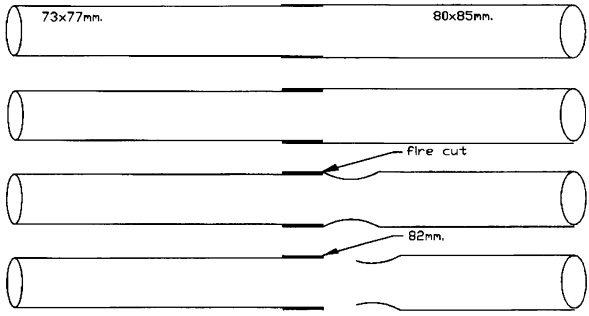

figure 4.

Before proceeding to the next lamination, change over to a six-fire seven-jet cradle burner with 22mm O.D. tips. This will provide the necessary heat to thoroughly bond the

remaining laminations. Next take the piece of 80 x 85mm tube, which you have first cut off, and, making sure you have a square end, re-chuck this in the tailstock. Using the cradle burner, heat a section of 1" to 1 1/2" of this tubing and, with a carbon rod, lift this section up so that it just fits over the flange you have started in the headstock (Fig. 5). This process can be eliminated if the proper size tubing is already on hand. Again, using the cradle burner repeat the process as in the first lamination. Once a good lamination has been attained, remove the excess tubing in front of the flange.

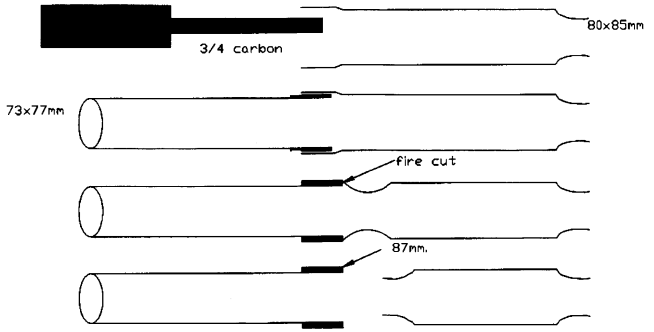

figure 5.

The 90 x 95mm tubing will be used for the last two laminations (Figs. 6 and 7). This should fit nicely over the just-completed portion of the flange (Fig. 6). After completing the third lamination, remove the 90 x 95mm from the flange and use the opposite end for the last seal.

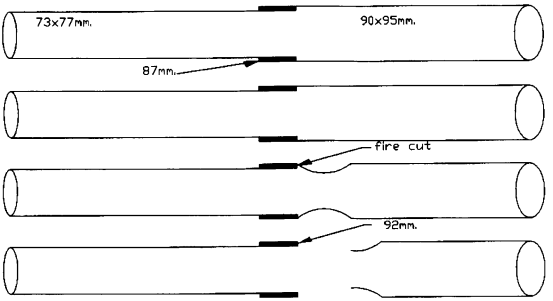

figure 6.

Because the flange is now larger than 90 mm, it is necessary to heat 1" to 1 1/2" of tubing and lift it up with a carbon rod as previously done in the second step of the 80mm x 85m tubing (Fig. 7). Duplicating that procedure completes the final lamination.

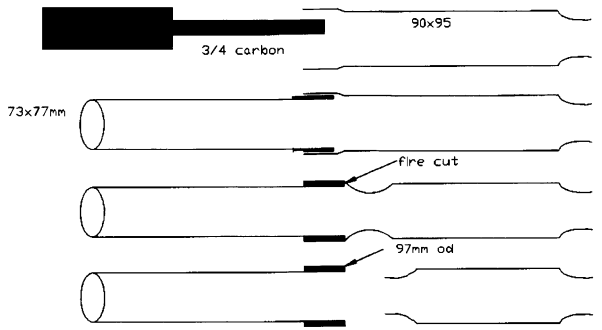

figure 7.

If all has gone well, there will now be a piece of 73mm x 77mm quartz tubing in the headstock with a rough-looking flange on the end approximately 97mm to 100mm in diameter and 10mm to 15mm in width.

The last step is to trim the flange to the desired size using a wet saw with a diamond blade and a belt grinder (Fig. 8). When cutting the front and back sides of the flange, be sure to use a stop on the wet saw table as this will produce a uniform surface when the operation is complete. To start the cut, push the portion of flange to be trimmed into the saw blade approximately 3mm and rotate the tube until a full revolution is completed. Repeat this process to within a few millimeters of the original 73 x 77mm tube. At this point, continue with caution so as not to cut into this tube, but flush with it. The result should be a very good square surface upon completion. The front portion of the flange can be cut in the same manner or by slowly cutting straight through. This will determine the desired thickness of the flange. If the diameter of the flange is too large, simply grind to size on a belt grinder. Finally, soak the flange in dilute hydrofluoric acid to remove impurities, rinse and fire polish. The face of flange can be put on a lap wheel to insure flatness.

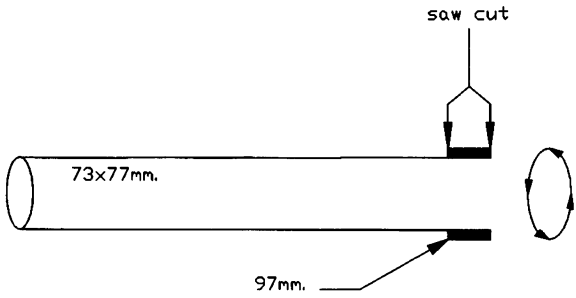

figure 8.

Optional O'Ring Groove (Fig. 9)

If an O'ring flange is desired, securely clamp the flange in a drill press and, either using a diamond core drill or a brass cylinder with notches and 220 grit, "slowly" drill out desired groove. If the drill press is brought down too hard on the flange, it will chatter and jump track. Slow and easy does it every time.

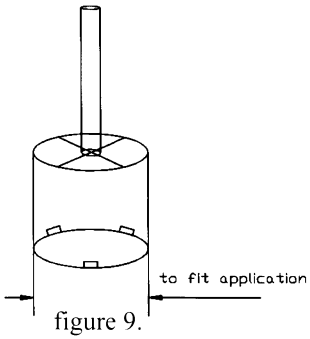

figure 9.

Acknowledgments

I would like to thank Ray Garcia for the illustrations in this paper and Ludwig Veit, a glassblower of 35 years with Hughes Research in Malibu, CA, for introducing me to this technique.

Everyday Glass Technology

by
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The history of glass is well-documented: it dates back to ancient Egyptian glass beads. So I will attempt to tell you a little about everyday modern types of glass.

What is glass?
"It is a product of fusion which will solidify without crystallization."

Most materials, upon cooling, crystallize i.e., metals, water. Apart from applying to oxide glasses, this definition also covers glassy metals which solidify by rapid splash cooling and materials such as glassy carbon fibers used in tennis rackets, bicycle frames, and aircraft parts because of their light weight and their strength.

OXIDE GLASS
The oxide glasses we use can now be formed without fusing by a chemical synthesis from solution using the Sol-Gel process. During this process, metalo-organics soluble in alcohol are mixed, gellified, dried into a sponge, and then shrunk. This method was developed when Corning artists, wanting to make large sculptures, found that the annealing of large masses took too long (the annealing process removes internal stresses due to uneven cooling).

Other applications of the Sol Gel process exist:

• Transparent insulation between windows: since the pores are smaller than the wave length of light, as the solvent evaporates out of the pores, the gel becomes transparent.

• Special glasses such as I-R for transparent fiber optics which cannot be produced by fusion as some of the ingredients are too volatile.

• Very clear small optical parts made by Gel-Tech (Sandia Labs in Albuquerque pioneered this process).

PYREX: A borosilicate, heat resistant glass, with a low expansion due to its boron oxide composition.
Borosilicate glass was invented in Germany to be used in the lamps of railroad conductors which were being cracked by the rain. A group attached to the Leipzig University was asked to make red and green glass for railroad signals. At that time, gold was used to make ornamental red glass. Analyzing the ancient Egyptian beads, the researchers found that Egyptians used copper for red – apparently, copper in a reducing atmosphere colors the glass red. That was the beginning of glass chemistry.

PYREX is the primary glass used for labs and kitchens. The telescope mirror on Mount Palomar was made in the thirties and casting and annealing that 200 inch plate was a serious undertaking. The NrI cast is now in the Corning museum.

A new process for the centrifugal casting of glass mirror blanks was developed at Arizona U. At first, this process was used for casting the funnels for TV tubes.
Pyrex has a high resistance to corrosion. In this sense, it differs from the old stained glass windows in the European cathedrals which are destroyed by sulfur dioxide from car

exhaust and the use of fossil fuels in industry and home heating.

QUARTZ, fused Brazilian.
Fused silica from silicon tetrachloride is the purest man-made material. It is used in large amounts for the diffusion tubes in micro-processor fabrication. In Albuquerque, there is the large micro-processor, Intel. Quartz is also used in high transmissions in UV and IR optical applications.

SOLDER GLASSES, to join glass to glass, metal to glass or ceramic.
This development made color TV possible as there is a low melting and matching expansion.

GLASS CERAMICS
The glass composition includes a nucleating agent, such as titania or circonia. This will precipitate from the glass at high temperatures and start the crystal growing until all the glass is crystallized. This process started at Corning labs when Dr. Don Stookey was working on UV light-absorbing compositions. By mistake, his technician set the furnace temperature too high for heat treatment. When he came the next morning to remove the sample from the cold furnace, he found a white glob of mass. In a rage, he threw it on the floor, but it did not break; and that is how the Pyroceram was invented.

Glass ceramic dishes are formed by automatic glass-forming machines and then converted by heat treatment into practically unbreakable white dishes. The main problem is low heat conductivity.

By including beta eucriptate crystals in the lithium-aluminosilicate glass composition, a 0-expansion glass is produced which is useful for the construction of mirror blanks for telescopes.

Another useful property of glass ceramic is its use as a solder glass. Once the material is fully crystallized it takes a much higher temperature to melt it; color TV tubes are made that way.

Once quartz instruments were commonly fused together with torches and used to measure the pressure inside a jet engine; they are now being CO^2 laser welded as well. In addition, lasers are commonly used to cut quartz throughout the industry.

In the last century, engineers were faced with the problem of making glass strong enough for steam boiler gauges. They made three layers of glass with the outside layers of a higher expansion than the inside layer. After cooling, the inside layer was in strong compression.

Corolla dishes are unbreakable because they are made with a higher expansion glass on the outside and a lower white layer inside in the form of continuous sheet. Then, the dishes are cut by a cookie cutter, formed, and, when cooled down, the outside layers will put the inside in compression thus making it very strong.

The same effect can be achieved by chemical strengthening with "Ion Stuffing." Larger ions of potassium replace smaller ones of sodium in a chemical bath; after cooling, the outside layer grips the inside in compression. Corning has a film in which a coffee cup was dropped from the 8th floor onto a steel plate without breaking. Cemcor glass used in aircraft windshields forms an unbreakable protection when backed by thick layers of aluminosilicate plates.

My Papa in the twenties experimented with glass composition trying to improve its strength; he had no luck, at that time, the physics of glass were not known so well. Being then a small boy helping around the labs, I saw his frustration and said that when I grew up I would make glass that would never break. Papa said, "Do not do that my son, it is bad for business."

Ordinary tempered glass in a patio door is tempered thermally. This is accomplished by heating the plate close to the softening point and then rapidly cooling it, thus leaving the plate in compression. It should be added, that after tempering, the plate cannot be cut.

Why does glass break? The surface is damaged by handling thus forming invisible microcracks; when pressure is applied in tension, the cracks will propagate, sometimes catastrophically. Putting glass in compression prevents crack propagation. A fiber drawn in pristine condition can have the strength of one million PSI. The ceramics, glass-ceramics also, stop the propagation of the cracks on the crystals' boundaries.

FIBER GLASS.

Thin fibers of glass-like wool trap the air, and act as an insulation. Fiber optics depend on the property that, at a small angle, the light is totally reflected with no loss and can be piped into small fibers.

Medical applications include the endoscope, used to look inside the body. But, since the signals can be piped through the fiber faster than electrons through a metal wire and since the signal is not influenced by a magnetic field, the passing of the light does not generate heat like in the wire.

We now have fiber telephones and TV communication, thanks to glassblowers who developed perfectly transparent fibers which can pipe the optical signals many miles without loss. Fiber optic cables are now going around the world and soon our telephones and computers will be linked by optical fibers.

As plastics are replacing glass in our daily life more and more, glass is still used in high technology. It is an essential component of chemical lasers, optical informational storing devices which are faster than electronic ones, and glassy carbon for ultra strong structures.

Sand, the basic ingredient of glass, is the major component of the earth's crust. As we use up the metals, not to mention the hydrocarbons for plastics, glass and glass ceramics will become the material of the future.

"Hyperpolarized Gas Imaging Cells for MRI"

by
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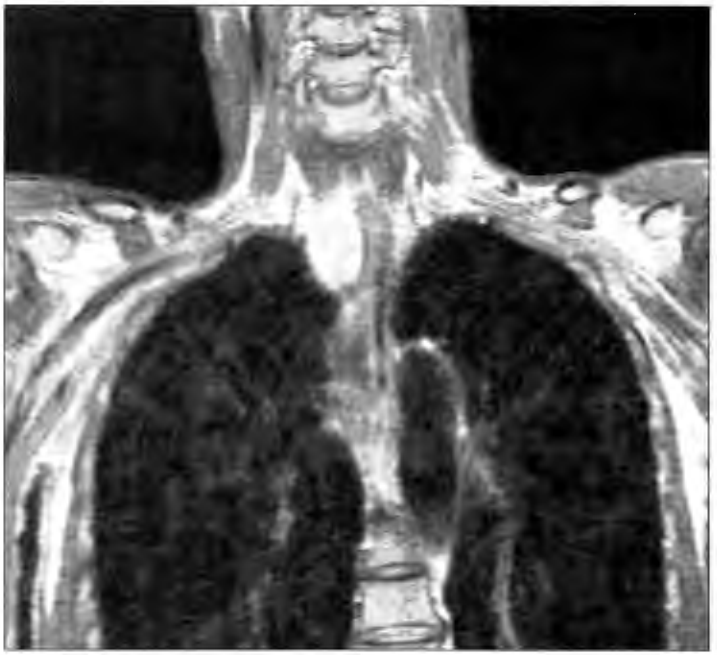
Introduction

In the Spring of 1993, Princeton University, in collaboration with SUNY at Stonybrook, produced the first high resonance image (MRI) of an excised mouse lung using a hyper polarized gas,[1] (a gas in which the magnetization is greatly increased over thermal magnetization). This technique utilized laser polarized ^{129}xenon, and has since been extended to produce laser polarized ^{3}He to give the first high resolution, in vivo images of both animal and human lung spaces. Conventional MRI is based on observing nuclear magnetic resonance signals from the protons in water and fat contained in soft body tissue. However, certain organs, in particular the lungs, have been very difficult to image due to the low concentration of water or tissue. Several examples of current diagnostic images illustrate the difficulty when it comes to imaging lungs.

MRI Image of Chest



In a conventional MRI image the lungs appear to be dark cavities. MRI depends on signals given from areas of the body such as soft tissue where water or fat are present (Photo1).
Photo Courtesy of Univ. of Fla. College of Medicine

Chemistry Department
** Physics Department*

[1]*Nature* 370 (1994):190.

CAT Scan

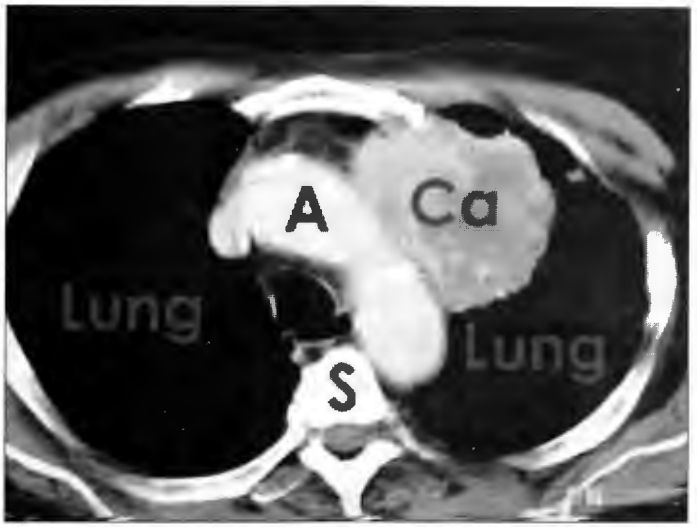
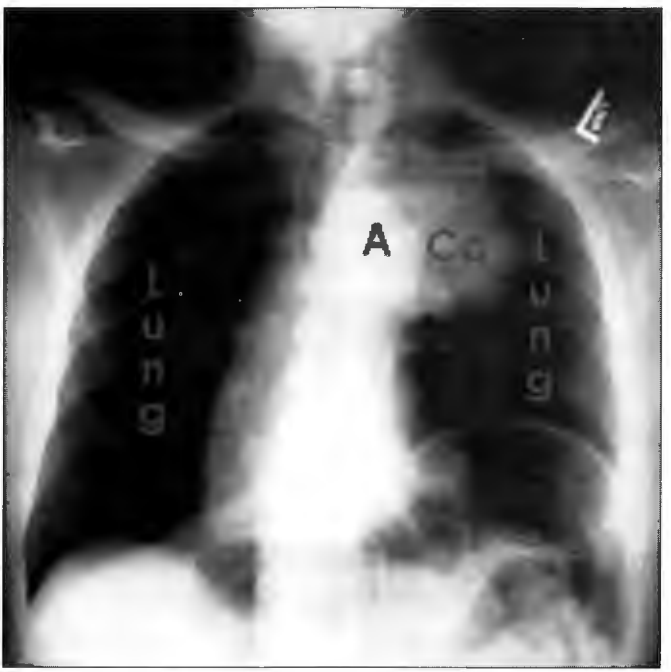


Photo 2 above is a coronial Computerized Axial Tomography (CAT scan) image of a person with lung cancer. Ca denotes the cancers, A the heart, and S the spine (Photo2).

Photo Courtesy of Harvard Medical School Archives

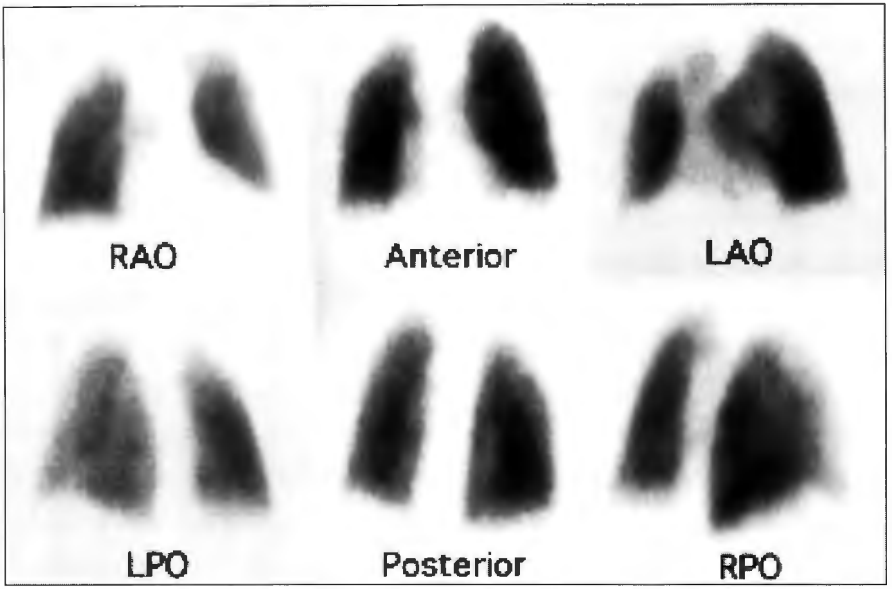
A Chest X-ray



The typical chest X-ray can detect mass, in this case cancer, but x-ray does not detect all soft tissue areas well (Photo 3).

Photo Courtesy of Harvard Medical School Archives

Perfusion Ventilation Scan w/ Radioactive Xenon



Currently one of the most widely used diagnostic tools for imaging lungs is perfusion scans of radioactive xenon. In this technique the subject receives a dosage equivalent to nearly 500 chest X-rays

Photo Courtesy of Harvard Medical School Archives

Typical Doses to Exposed Tissue Received in Routine X-Ray Diagnosis

Courtesy of Encyclopaedia Britannica

examination	dose per exposure in milligray (mGy)*
X-ray photograph	
Chest	0.4-10
Abdominal	10
Extremities	2.5-10
Fluoroscopy	100-200 per minute
X-ray movies	250 per examination
CAT scan	50-100 per examination

*Milligray is a unit of absorbed radiation dose; it corresponds to 1/1,000 joule of radiation energy absorbed per kilogram of tissue.

Magnetic resonance imaging of lungs inflated with hyperpolarized noble gases, such as ^{129}Xe and ^{3}He, overcomes many of these difficulties. In this technique nuclear magnetic resonance signals from the hyperpolarized gas instead of from water are observed. Normally, the low density of gases do not allow for ready visualization. Laser optical pumping, together with spin-exchange, creates a very large nuclear magnetization in the gases.[2] This large magnetization in hyperpolarized ^{129}Xe and ^{3}He allows for the ready visualization of tissue such as lungs. More importantly, the signal produced from MRI imaging with ^{129}Xe allows for differentiation of chemical environments due to the fact that xenon is soluble with hemoglobins.

Inhalation of both these noble gases is benign. Hence, hyperpolarized gas imaging offers numerous advantages over other such imaging techniques such as x-ray computerized tomography, perfusion ventilation scans using radioactive xenon, and other nuclear medicines which utilize ionizing radiation for imaging.

In this contribution, a brief discussion of the fundamentals of magnetic resonance imaging with hyperpolarized xenon and helium will be presented. A glass cell based, gas polarizer is used to produce the hyperpolarized gases, and its fabrication and use will be described. This paper will provide a brief description of the process and its end results.

What is MRI

MRI is the application of Nuclear Magnetic Resonance (NMR) to medicine. The technique relies on the response of magnetic particles to short bursts of radio-frequency waves to produce computer images that provide structural and biochemical information. MRI's are widely considered to be one of the safest diagnostic tools available. They are non-invasive; they do not depend on ionizing rays or dyes and can be performed numerous times in the course of a short period with no short or long term side effects from this process. Some of the other advantages of MRI diagnostics is its ability to provide three dimensional images or holograms from digital data obtained by conventional MRI scanners which can then be reconstructed by computers. These holograms can be useful in providing exact locations of lesions or coronary arteries for bypass surgery.

The disadvantage of MRI is that it relies solely on the abundance of protons available from water (H_2O) and fat. This problem lies in the fact that in certain areas of the body, specifically in the lungs, there are very few hydrogen nuclei to detect. Thus, the magnetic signal is far too weak to measure.

To overcome this problem two isotopes of noble gases, ^{3}helium and ^{129}xenon, can be made highly magnetic, in fact, 10 million times more magnetic, by incorporating a technique known as Optical Spin Exchange Pumping.

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[2] *Chemical & Engineering News* 72.30: 7-8

Estimates of Average Annual Dose Equivalent to the Whole Body from Various Sources of Irradiation Received by Members of the U.S. Population

Courtesy of Encyclopaedia Britannica

SOURCE OF RADIATION	Average dose rates (mSv/year)
Natural	
Environmental	
Cosmic radiation	0.27 (0.27-1.30)*
Terrestrial radiation	0.28 (0.30-1.15)**
Internal radioactive isotopes	0.36
Subtotal	0.91
Man-made	
Environmental	
Technologically enhanced	0.04
Global fallout	0.04
Nuclear power	0.002
Medical	
Diagnostic ***	0.78
Radiopharmaceuticals	0.14
Occupational	0.01
Miscellaneous	0.05
Subtotal	1.06
Total	1.97

*Values in parentheses indicate range over which average levels for different states vary with elevation.

**Range of variation (shown in parentheses) attributable largely to geographic differences in the content of potassium-40, radium, thorium, and uranium in the Earth's crust.

******In an average lifetime nearly 40% of the radiation a person will receive will come from Diagnostic Medicine***

Materials & Methods

Typically ^{129}Xe and ^{3}He are hyperpolarized by means of spin-exchange collisions with optically pumped rubidium atoms,[3] as has been described in detail previously.[4,5] Briefly, the noble gases are densely filled into cells made of aluminosilicate glass for the ^{3}He and from normal borosilicate glass for the ^{129}Xe. Aluminosilicate is preferred when using helium due to its low permeation characteristics. Since the cells have to maintain pressures that can exceed 200 PSI the glass tubing stock is reblown to create thicker walls and to remove hidden flaws. Prior to filling, the cells are evacuated and approximately 0.1 gram of rubidium is deposited inside the cell.

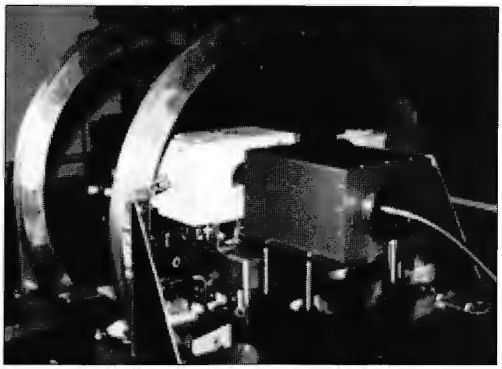
[3] W. Happer, F. Miron, et al., "Polarization of the nuclear spins of noble gas atoms by spin-exchange with optically pumped alkali metal atoms," *Physical Review* 29.Suppl.A (1984): 3092-3110.

[4] H. Middleton, R.D. Black, B. Saam, et al., "MR Imaging with hyperpolarized ^{3}He gas" *Magnetic Resonance in Medicine* 33 (1995): 271-275.

[5] J.R. MacFall, G.D. Cates, B.Driehuys, et al., "Human lung air spaces: Potential for MR Imaging with hyperpolarized He-3" *Radiology* 200.2 (Aug. 1996): 553-558.

Optical Pumping occurs when the ^{3}He cell is heated to 180°C (in order to create rubidium vapor) and is exposed to 100 W of circularly polarized irradiation from a diode laser array aligned to illuminate the entire volume along the axis of the cylinder. The laser is tuned to the resonance of rubidium and induces electronic spin polarization of the rubidium. This polarization is transferred to the ^{3}He by means of collisional spin exchange.

Optical Pumping



A xenon cell is being optically pumped. The white box contains the cell and acts as an oven to heat the rubidium metal inside the cell into a vapor. The diode laser is in front and polarizes the rubidium atoms which then exchange their polarized state with the xenon gas

Photo Courtesy of MITI

In the case of a helium cell, after 10-12 hours of optical pumping, polarization builds up to levels as high as 50%. Once the cell is cooled, the rubidium is deposited as a solid along the inner surface of the cell and residual rubidium vapors are almost non-existent. Under optimal conditions, the ^{3}He remains polarized for several hours after the optical polarization. [6]

The polarized gas is released into a conventional plastic bag (approximately 1.2 liters) to which a plastic straw and a hand operated valve is attached. The bag is given to the subject inside the MRI's magnet and the subject is instructed to open the valve, inhale through the straw and hold their breath as long as possible. The subject is also given a squeeze bulb that actuates a sound to signal the MRI operator as the gas is inhaled. Roughly 0.75 liters is actually inhaled by the subject and imaging starts the moment the operator hears the signal from the subject.

In a short span of time, the polarized gases rapidly fill the trachea and all of the lung spaces in the same manner as breathing. As the subject holds their breath, the gases move by convection and diffusion. ^{3}He diffuses more rapidly than ^{129}Xe and does not lend signal intensity to blood or the surrounding tissues of muscle or fat. Within 20 seconds, the signal dissipates as the helium quickly depolarizes in the body. Nevertheless, the images that are taken during that time prove that a high degree of resolution is obtained.

[6] ^{3}He sustains its T1 value noticeably longer than ^{129}Xe. In principle, cells using ^{3}He can last as long as 30 to 60 hours while ^{129}Xe may only last as long as 6 to 12 hours. However, ^{129}Xe can be accumulated and stored for hundreds of hours in its frozen state.

MRI imaging of lungs utilizing ^{129}Xe shows that xenon moves quickly from the lungs into the bloodstream and from there to the rest of the body.[7] Moreover, xenon, is currently used as a safe anesthetic. Conceivably, hyperpolarized xenon may be used in the future to enhance imaging techniques in an assortment of other areas of the body.[8] Xenon's anesthetic qualities present themselves as its abundant electrons attach to the hemoglobins in the body's blood streams. In addition, xenon can be obtained from our atmosphere and polarized in bulk quantities which can be frozen to 77^K (liquid nitrogen) for a short term of two to three hours or stored for hundreds of hours at 4^K (liquid helium) without substantial loss to polarization.[9]

FIGURE 1 is a basic schematic of a Xe Flowmaster. The cell is encased in an oven with glass windows and heated to 130c–150c

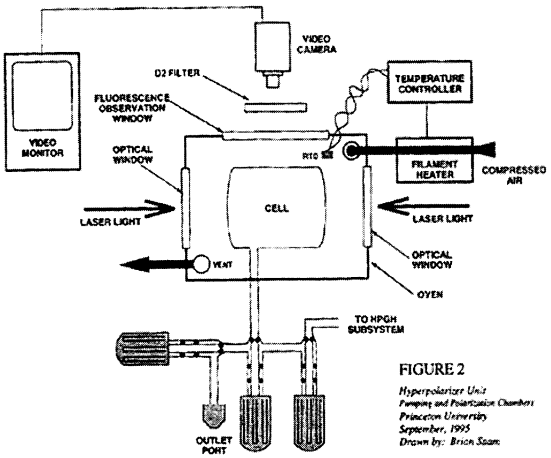


FIGURE 2
Hyperpolarizer Unit
Pumping and Polarization Chambers
Princeton University
September, 1995
Drawn by: Brian Saam

Polarizer Cells

Polarizer cells that utilize ^3He are typically made of aluminosilicate glass. Borosilicate glasses can be used, but the effects of the rubidium at elevated temperatures tend to break down the glass and darken it, eventually making the cell unusable. Ideally, a cell will be refilled endlessly once it is installed into the polarizer.[10] Additionally, unless the cell is made from an aluminosilicate glass, helium permeation[11] occurs rapidly at elevated temperatures during optical pumping, creating less than optimum conditions for polarization.

Figures 1 & 2 show that the cell design for the ^{129}Xe polarizer[12] is noticeably different than the ^3He polarizer. The ^{129}Xe polarizer as designed has the added capability of being an accumulator. It will generate the gas and then freeze it for storage while the ^3He polarizer will generate, store, and deliver the gas. Thus the ^3He cell is larger and made wholly from aluminosilicate glass. In practice, this cell will be filled to 10 atmospheres of ^3He, and,

[7] "MR Imaging and Spectroscopy Using Hyperpolarized Xe-129 Gas Preliminary Human Results", submitted to *Magnetic Resonance in Medicine*.

[8] "Temporal Dynamics of Hyperpolarized Xenon-129 Resonance's in Living Rats," *Journal of Magnetic Resonance* (1996): 300.

[9] M. Gatzke, G.D. Cates, B. Driehuys, D. Fox, W. Happer and B. Saam, "Extraordinarily slow Nuclear spin relaxation in frozen laser polorized ^{129}Xe," *Phy. Rev. Lett* 70, 690 (1993).

[10] As of December 1997, the University of Virginia has been using a cell in their IGI unit that has been continuously used and refilled over 100 times without degradation.

[11] V.O. Altemose, "Gas Permeation Through Glass," *A.S.G.S Symposium Proceedings* 7 (1962): 61-70.

[12] "High-volume production of laser-polarized Xe-129," *Applied Physics Letters* (16 September, 1996): 1668.

next, heated to 180°C. This increased temperature raises the internal pressure of the cell to nearly 200psi. To assure the integrity of the glass, the walls of the cell are 5 mm thick. To achieve such a wall thickness, 90 mm od by 3 mm wall 1720 aluminosilicate tubing is shrunk down to 64 mm od. This process also achieves the added benefit of producing an inner surface that is more optimal for polarization than would be from normal stock tubing.[13] Moreover, manufacturing defects such as cords, air lines, stones and bubbles can be more readily detected and removed by this process, thereby making it safer to withstand high pressure.

Helium Polarization Cell

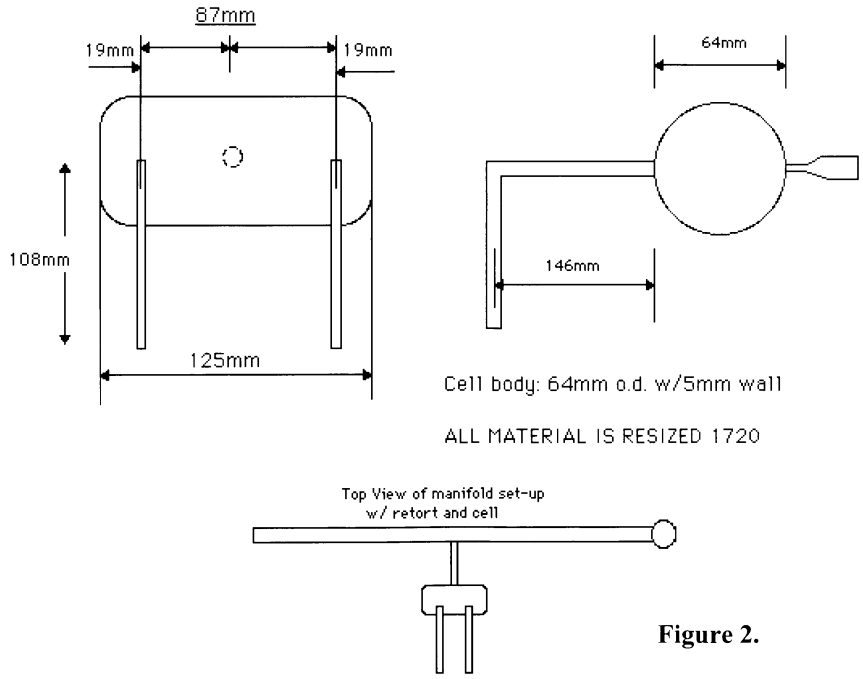


Figure 2.

An outlet tube on the cell is graded to borosilicate for connection to a vacuum manifold that incorporates a retort for the Rb. At the opposite side are reblown capillaries of 1mm id x 6 mm od. The capillaries are connected to glass stem valves that are graded with uranium glass just below their seats. The glass piston on the valves utilizes EP (Ethylene Propylene) o-rings due to their suitability to alkali vapors. The cell is sealed into a demountable fixture called a cradle. The cradle is part of the instrument panel that contains the furnace. In this modular fashion, the cradle can be used to evacuate and later fill the cell.

[13] M.J. Souza, "Super thin windows for high density ^{3}He target cells," *Fusion* 42.4 (Nov.1995): 20-27

Cradle for Helium Cell

A helium cell made of 1720 aluminosilicate glass is mounted to a cradle and is being sealed to a manifold for evacuation

Photo Courtesy of MITI

After the cell is evacuated to 5 x 10^{-8} it is installed onto the gas delivery system of the polarizer unit. Once the cell is in place, the polarizer is a fully transportable device that can generate polarized gases and deliver them at various MRI facilities. Currently these units are sold as IGI (Inert Gas Imaging) devices by Magnetic Imaging Technologies Incorporated[14] (MITI) of Durham, NC.

An IGI (Inert Gas Imaging) Unit

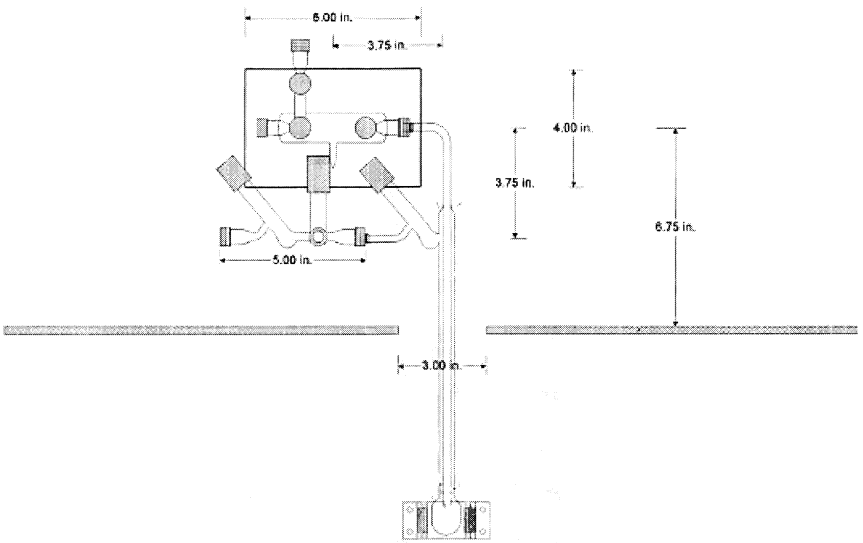
The IGI is the mainframe unit where the gases are polarized.

Photo Courtesy of MITI

[14] MITI has obtained an exclusive license on the basic patent (pending) describing how inert gases are prepared for use in MRI. Researchers from Princeton and Duke have demonstrated the viability of the IGI technique in studies on the lungs of live guinea pigs and in studies on human volunteers. Duke and the University of Virginia are the first IGI sites at which clinical trials will start. IGI is being regulated by the device group (CDRH) of the FDA.

The ^{129}Xe polarizers can be fabricated from a borosilicate glass and, as stated previously, they are smaller. Glass piston valves are once again used to regulate the flow of gases. The outlets to these valves utilize #7 Chem -Threads[15] which in turn hold 1/4" capillary tubing. The capillary tubes have a groove cut near their ends to help secure the compression seal of the o-ring on the #7 Chem-Thread. This type of connecting makes the polarizer easy to assemble and provides hermetic seals that can take pressures reaching upwards to 200 psi. The connections are used to facilitate the addition of a collector trap. The polarized ^{129}Xe streams into a cold trap and is cooled in a liquid N^2 dewar. As the process continues, chunks of hyperpolarized gas in the form of solid ice can be collected. In this solid state, the polarized ^{129}Xe could, in principle, be accumulated at a rate of 20 liters a day[16] sustaining its $T^{1'}$ s for hundreds of hours.[17]

Xe Trap Collector



The ^{129}Xe polarizer is a flowing gas system in which ^{129}Xe is polarized in the optical pumping chamber. As the gas flows through the cold trap, the Xenon is frozen out in a holding field of at least 525 Gauss[9] in the cold trap to maintain polarization.

Invivo Images

Nearly four years have passed since the first hyperpolarized image was produced. Since that time, dramatic improvements have been made and will continually be made in the use of hyperpolarized gases for diagnostics. Presently, Princeton University, MITI, Duke School of Medicine, and the University of Virginia's Department of Radiology are collaborating in trial studies to gain FDA approval. Moreover, dozens of other research groups throughout the world have undertaken the task of extending and improving the development of polarized gases.

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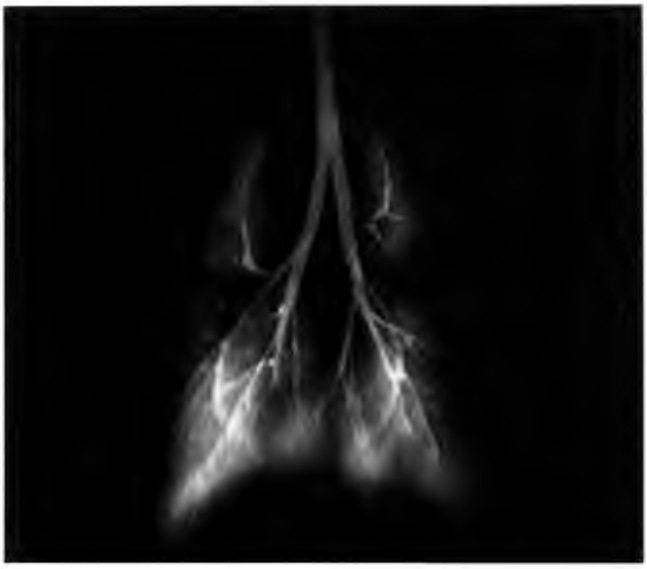
[15] #7 Chem-Threads from ChemGlass, Vineland, NJ.

[16] "High-volume production of laser-polarized Xe-129," *Applied Physics Letters* (16 September, 1996): 1668.

[17] Provided the cold trap was maintained at 4K.

IMAGE ONE

Guinea Pig Lung Image Using Polarized ^3He

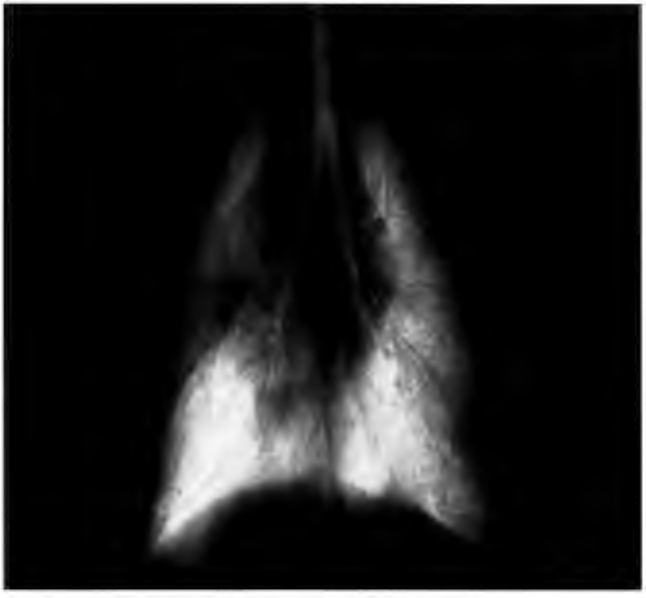


In this first image a live guinea pig aspirates polarized helium. Immediately, the gas lends its signal to the respiratory system. The detail is such that the trachea, which is only 1mm in diameter, is clearly defined.

Photo Courtesy of Duke University Medical Center
Center for In Vivo Microscopy

IMAGE TWO

Guinea Pig Lung Image Using Polarized ^3He



In a matter of seconds the helium diffuses throughout the lung

Benefits from this research

The two most immediate benefits of MRI lung imaging will most likely deal with people suffering from Chronic Obstructive Pulmonary Disease (COPD) and Pulmonary Embolisms (PE). COPD's include Emphysema, Alpha1 Antitripsin Deficiency - related Emphysema (AAD), which is also known as "early onset emphysema," and Chronic Bronchitis. Persons suffering from PE's are at immediate life-threatening risks from blood clots or vascular obstructions lodged in the lungs. They are inherently dangerous in this area of the body because they are so difficult to detect. The following facts speak for themselves:[18]

- There are approximately 630,000 cases of pulmonary embolisms in the US each year, with an 11% mortality rate in the first hour
- The diagnosis is missed in 71% (400,000) of the survivors
- A vast majority of subsequent deaths from PE occur within two weeks of the initial episode with a 23.8% mortality at one year.
- Almost all of these later deaths are attributable to underlying cardiovascular, pulmonary, or malignant disease.

Emphysema is chronic and causes irreversible lung damage. The walls between the lung and air sacs lose their elasticity causing air to be trapped in the air sacs and impairing the exchange of oxygen and carbon dioxide. According to the American Lung Association:

- An estimated two million Americans suffer from emphysema
- COPD's are the fourth leading cause of death, claiming the lives of nearly 96,000 Americans every year.

Change of habits and lifestyle can slow the effects of this disease. However, once the process begins, its effects are irreversible. Subjects slowly lose their capacity to breathe and become dependent on artificial ventilation to sustain their lives. Until recently, the only cure for chronic emphysema was a lung transplant; bottles of oxygen provided sufferers their only relief.

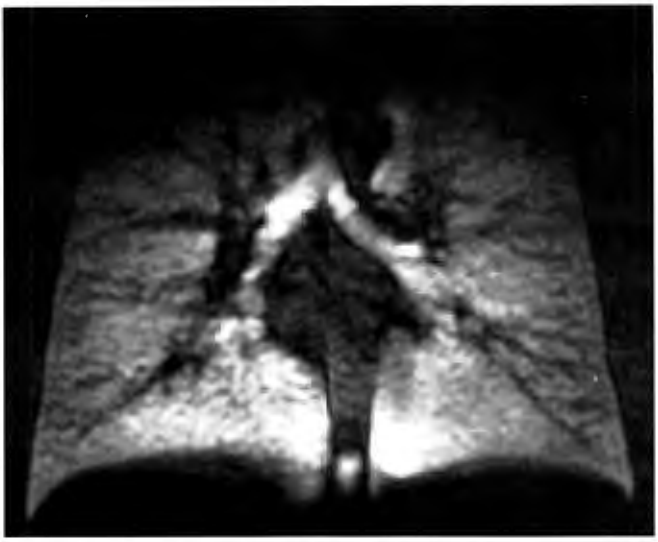
However, a new technique termed "lung shaving" has been shown to give relief to emphysema sufferers.[19] This technique involves excising the deteriorated sections of the lung that have stiffened and continue to restrict the viable portion of the lung's ability to function. In spite of the various case studies that have shown increased comfort and improved quality of life, this procedure is presently deemed "experimental" by Medicare. This action was largely due to the fact that surgeons have only a faint notion of where the damaged parts of the lung exist until the chest is opened. By having a pinpoint map of where such areas exist prior to surgery, surgeons can determine the easiest way to excise the stiff sections of the lung and to do so with the least amount of collateral damage. Polarized gas imaging by MRI offers the best hope for these chronic sufferers.

---

[18] J.E. Juni, A. Abass, "Lung scanning in the diagnosis of pulmonary embolism: The emperor redressed", *Seminars in Nuclear Medicine*, 21 (1991): 282 -296.

[19] According to the National Jewish Hospital's Center for Immunology and Respiratory Medicine in the *Journal of Respiratory and Critical Care Medicine* issued in conjunction with the 1995 meeting of the American Thoracic Society.

Human Lung Image



The image above is one of the first MRI's ever taken utilizing a hyperpolarized gas

Conclusion

The ability of polarized ^{129}Xe to become soluble with the blood lends hope that sometime in the near future the entire vascular system of the body can be scanned. Polarized gases will enable existing MRI equipment to scan new sections of body. They could also, in the very near future, drastically reduce the cost of new MRI equipment, since the function of the superconducting magnets and cryrogenic material (two very costly components) will become far less dependent on producing a magnetized field in a patient. The end result may be that all MRI's will be done in an open room and not in a claustrophobic tunnel. Moreover, smaller medical centers and rural communities will be able to afford the use of more mobile and less expensive equipment for MRI. Consequently, hospitals will become less dependent on radioactive agents for diagnostics and produce less toxic waste.

The benefits of this new technique to Diagnostic Medicine cannot be over stated. MRI is the safest and most benign way to image the human body. By extending its capabilities to new areas such as the lungs, physicians will be able to detect damage to lung tissue at the earliest possible stage, and thereby, prevent or slow chronic and irreversible lung disease.

Acknowledgements

In no particular order I would like to thank several people for their help and guidance. Professors Will Happer and Gordon Cates from the Physics Department of Princeton and my co-author Karen Sauer.

Much of this material would not have been possible were it not for the folks at MITI (Magnetic Imaging Technologies Inc.) in Durham, NC.

The drawings and schematics from Dr. Brian Saam now at Washington University, St. Louis, MO.

The extraordinary images provided by Duke University Medical School's Center for In Vivo Microscopy and their grant **NIH NCRR P41 RRO5959**

Finally, my boss Professor Tom Spiro of the Chemistry Department, Princeton University

WWW Sites

For the latest information and images regarding this subject you can visit the following www sites:

Duke University's Center for In Vivo Microscopy Web Site at
http://wwwcivm.mc.duke.edu

MITI's homepage at http://www.miti2.com/

University of Virginia: Hyperpolarized Xenon-129 and He-3 Work at
http://avery.med.virginia.edu/~tm6a/

Harvard's Medical Web at http://count51.med.harvard.edu/

Miniscale, Glass-Miniplants for Pilot Processing

by
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Apparatus, plants and pipelines from borosilicate glass for chemical processes are usually advanced in laboratories and technical work. The growth of this market is very extensive since the development of petrochemical products, polymers, organic and synthetic chemicals, vitamins, life-sciences... The role of pilot plants in laboratory and industry is increasing; they are the key in the process development in the sense of scale-up experimental work. With demonstration units made out of glass, new technologies in chemistry are usually created for which no analogous experience is available.

The use of the miniplant concept and technique which were developed within BASF AG is a combination of process simulation by computer and experimental data search in practice. Miniplants are a miniaturization or a scale-down of good commercial plant technology or great pilot plants to the smallest scale. This is effectuated in combination with careful engineering and selection of equipment and facilities. The challenge of the market to minimize costs and resources is one aspect; the other is competition and the need for shorter research times and quicker introduction of the product to the market.

Some types of glass-apparatus and plants for chemical processes like absorption, adsorption, distillation or rectification, extraction and reaction are available. These include laboratory and testing systems for chemical research ranging from micro apparatus to full pilot plant and small scale production units.

For micro and semi-micro distillation, the "Fischer Spaltrohr™" columns for universal distillation purposes are useful. The particular advantages of Spaltrohr-columns are: all-glass construction, extremely low pressure loss, high separation efficiency over a wide load range, and gentle heating; in addition, inside Spaltrohr™ there are no mechanical installations, no abrasion and no wear (Figures 1, 2, 3; Table 1).

Fraction Collector ( )

An easily operated unit. The revolving insert holding up to 22 collecting tubes can be removed by opening the quick release spring together with the lower part of the collector and then lifted off. The top part of the collector remains in position. The change of tubes is effected by turning the wheel at the base part. A sample can be taken from any of the fractions through an injection part, also under vacuum.

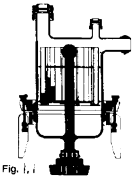


Fig. 1.1

Sample Changer ( )

This practical device enables the operator to change single collecting tubes also under vacuum. The distillate is collected via a greaseless PTFE-valve.

By using a multi-way valve the vacuum in the unit can be released and the sample removed. After replacing the collecting tube the unit can be again evacuated without interfering with the vacuum in the column.

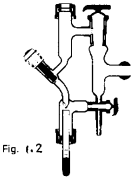


Fig. 1.2

Spider Collector ( )

A simple device for the collection of several fractions. The changing of the tubes is done by turning the lower part of the collector. Various numbers and sizes of collecting vessels can be supplied.

Particularly useful when working with substances of low evaporating temperatures as the collecting vessels can be dipped into a Dewar flask.

It is also possible to take a sample with a syringe from the drop-tip.

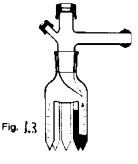


Fig. 1.3

Figure 1.

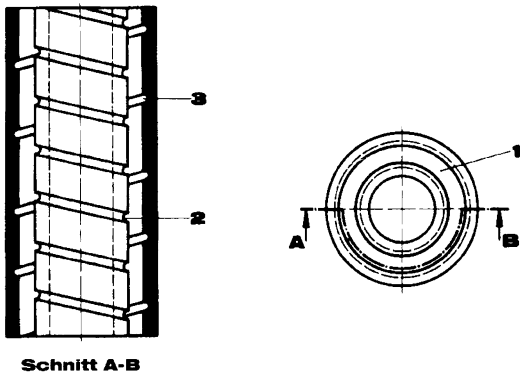
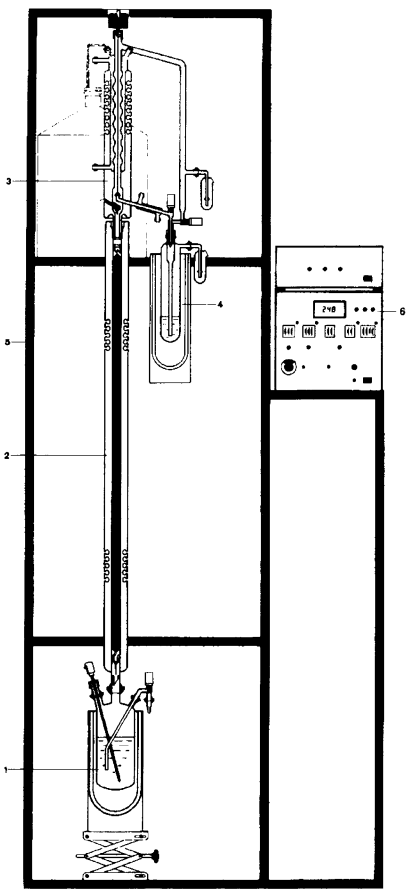


Figure 2. Spaltrohr™ - column. Axial Section of external tube.



Low Temperature SPALTROHR™-Column HMS 1000 T

Distillative separation of mixtures of low boiling hydrocarbons and other gases, e. g. methane to heptane, olefines, mixtures of saturated and unsaturated hydrocarbons, can easily be effected with SPALTROHR™-columns in laboratory scale in the petrochemical industry and for scientific researches. SPALTROHR™-low temperature distillation units enable on one side to run comparing distillations for technical processes in the laboratory, and on the other side also gases can be gained in the laboratory. Furthermore, distillations for analytical purposes are possible as well.

Scheme of a low temperature distillation apparatus
1 Distillation flask 2 l with cooling bath
2 SPALTROHR™-column
3 Column head for automatic reflux division or partial condensation
4 Distillate cooling receiver
5 Mounting frame
6 Control devices

Table I **Technical Data**

Type		HMS 1000 T
nominal charge volume	l	0,5–1,5
working temperature	°C	–190-200
theoretical plates approx.		80

Figure 3, Table 1.

Another type of laboratory and pilot plant for distillation, especially for crude oil distillation, is the "Fischer Labodest 254 AC." This type of automatic distilling unit, which has been developed within the last few years, allows the analysis results to be evaluated on a PC and recorded on a printer. This crude oil pilot and test plant is used for the study or simulation of the operation procedures in industrial scale refineries. In the petrochemical research and development laboratories, such units are successfully in operation for the design and optimization of full scale processes. The test unit heart consists of bubble cap tray columns of borosilicate glass, seen in the schematic of the Fischer Crude Oil Pilot Plant (Figures 4, 5; Table 2).

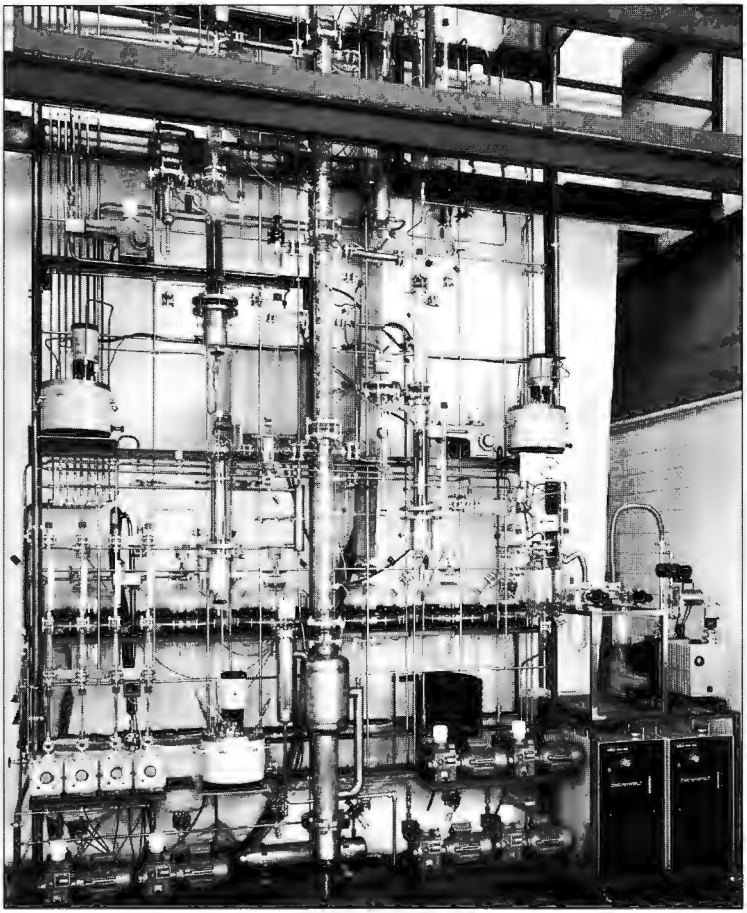


Figure 4. Labodest 254 AC. Crude oil plant for study purposes or for simulation of operating conditions in industrial-scale crude oil refineries.

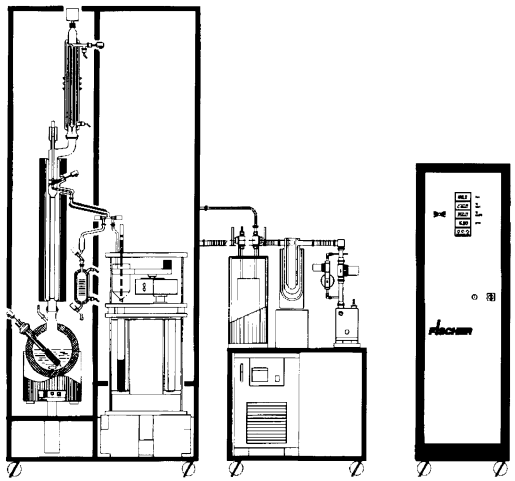


Figure 5. Autodest Model 800 C.
Processor controlled crude oil distillation system.

Operating temperature	°C	up to 350
Temperature range	°C AET	up to 420
Vacuum	Torr	down to 1
Flask size	ml	6,000
Receiver	ml	9 x 250
Dimensions (h x w x d)	m	3.5 x 2.5 x 0.8
Mains supply	V, Hz, kW	220, 50–60, 4.5

Table 2

The newly developed automatic distillation units have proven themselves particularly suitable for research purposes and industrial quality control, both in areas of education and of training. Calculation processes are either pre-selected by the operator or are stored electronically.

For ecological and economical reasons, the recovery of solvents from exhaust air flows is becoming increasingly important. An absorption distillation unit has been developed for exhaust air purification and solvent recovery based on thermal separation procedures like absorption, desorption and rectification.

This type of pilot plant is made of glass for laboratory equipment for studying absorption/ desorption. The washing medium, which is regenerated in the desorption column, is led back into the absorption column and is thus kept in a closed circuit. With these preliminary experiments, it is possible to avoid risks and surprises during scale-up to industrial plant systems.

Currently the miniplant technique is increasing in process development and the role of big pilot plants is changing. For the glassblower, it is a great challenge to manufacture these small and precise glass apparatus and plants. As glassblowers' work becomes more sophisticated, the skill and scientific knowledge for the profession is also increasing. Some miniplant glass-apparatus and equipment can be seen in the following figures, schematics and pictures.

Especially useful for a continuously-operated distillation unit with packed column, diameter 50 mm, is a circulation evaporator, upright form, with 2 quartz rod immersion heaters, 2x1 kW heating capacity, with continuous bottom take-off and/or bottom receiver, and a capacity of 1ℓ (Figure 6, 7 a&b).

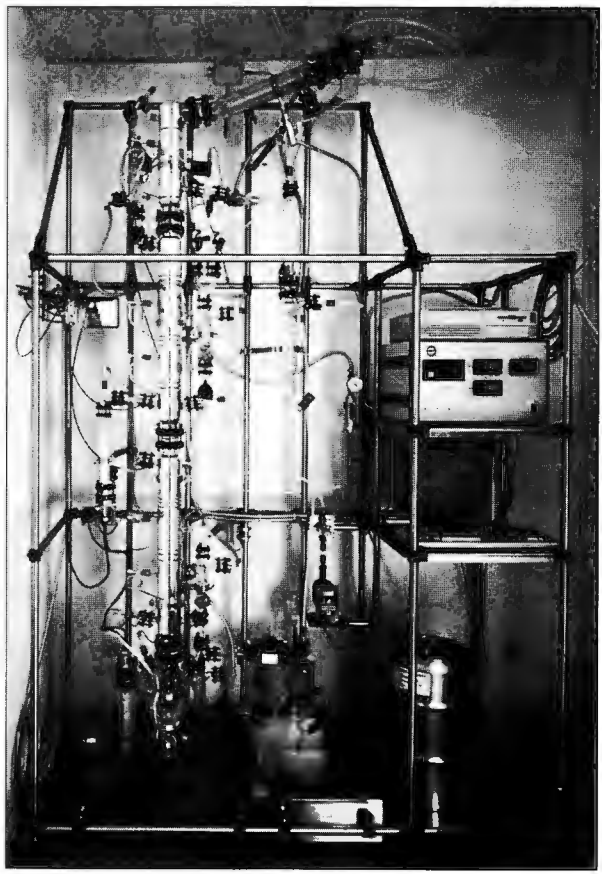


Figure 6. Continuously operated distillation unit with jacketed bubble-cap tray column or packed column DN 50, electronically controlled, semi-automatic.

Figure 7a. Continuously operated distillation unit with packed column DN 50, section of a miniplant unit installed at the University of Karlsruhe.

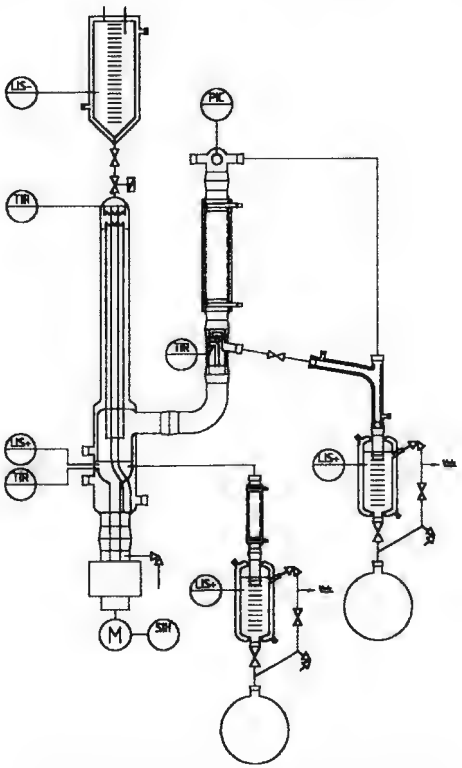


Figure 7b. Falling-film evaporator unit.

For good adiabatic conditions, heated flanges and valves, with double jackets, and without cold places at the flange connections for equal and complete tempering are necessary. For distillation columns for a high temperature range up to 300°C, the heating loss must be compensated for by electric heating and insulating collars with integral Pt 100 resistance thermometers for temperature regulation.

Generally, for temperatures up to 180°C, vacuum jacketed and insulated columns are used. Usually these columns are vacuum jacketed with silvered sight strips and inside expansion bellows; nominal widths for these columns are 30 and 50 mm (Figures 6, 7, 8, 9a & 9b).

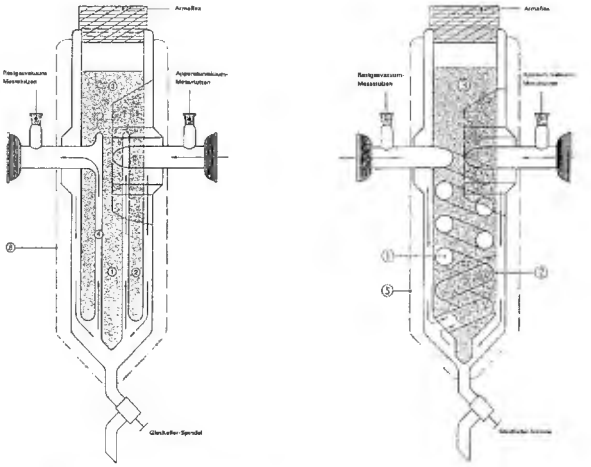


Figure 8. Intensive cold trap system by Trefzer, System CIBA, Ernst Keller, GLAS Keller, CH-4002 Basel, Switzerland.

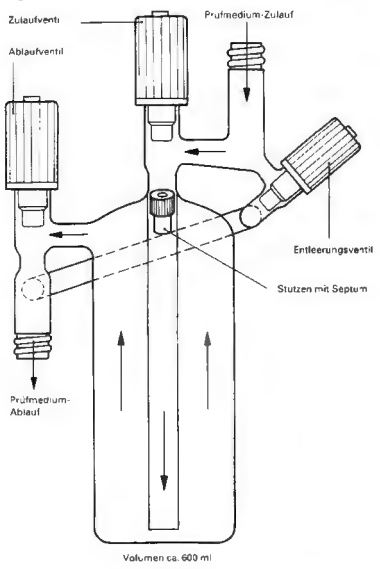


Figure 9a. Sampler by Trefzer, System CIBA, for continuously operating pilot plants, sampling in process linecontrolling.

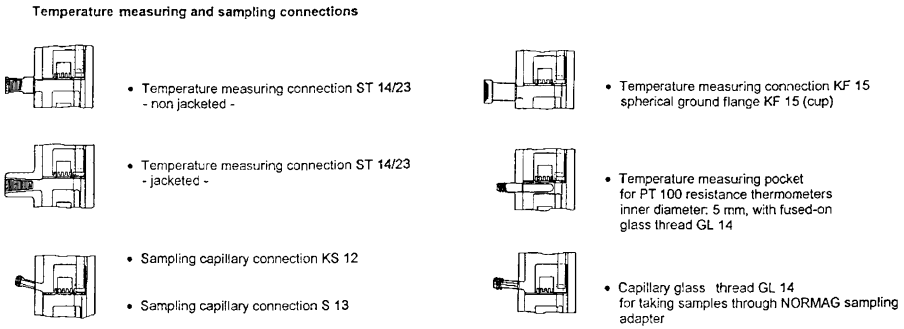


Figure 9b. Columns side connections.

Other miniscale processing plants include the following:

• **FEED DOSING FUNNEL** with glass needle valve for constant dosing at same pressure and equal tempering: this is used for viscous and readily crystallizable substances, for reactions with long feed time, for thin-film evaporation and short-path evaporation (Fig. 10).

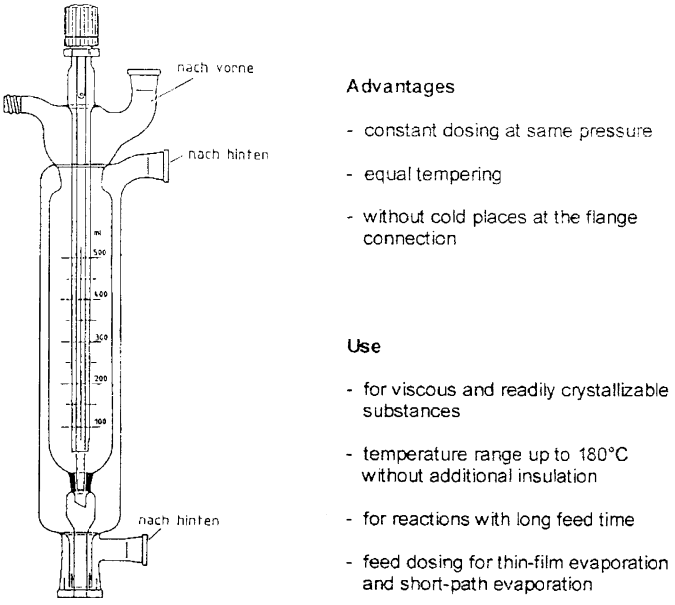


Figure 10. Feed dosing funnel with glass needle valve.

• **SPINDLE VALVE** made of glass/PTFE-Copolymeres, nominal widths 3, 6, 10, 15, and 25 mm in different construction forms and angle forms: this has a universal use for glass apparatus with ON/OFF function. With a tempering jacket for liquid heating medium, the advantages are grease-free operation, safety locking device, high vacuum tight, and adjustable spindle. It also has a bottom outlet valve with dead volume, nominal widths DN 6, 10, 15, and 25 (Figure 11).

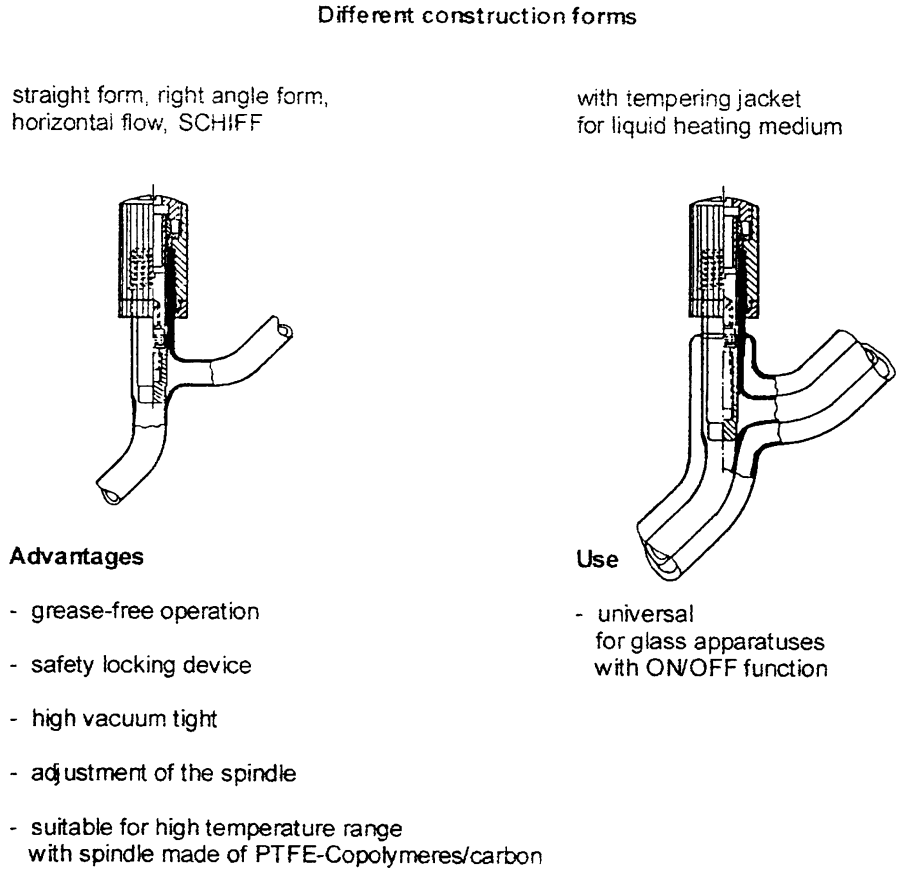
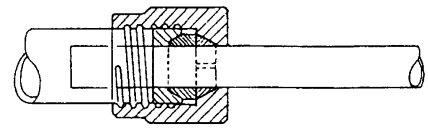


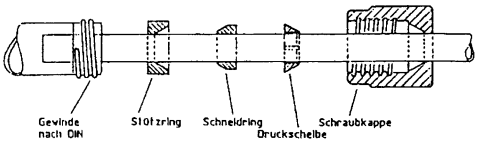
Figure 11. Spindle valve made of glass/PTFE-Copolymeres nominal widths DN 3, 6, 10, 15 and 25.

• **GLASS THREAD** with plastics screwing and product touched PTFE inserts according to the cutting ring principle. Advantages are as follows: flexible and easy to handle, easy connection to tubes, pipes and rods of different materials, vacuum tight, and safe under overpressure up to 5 bar. Suitable uses include: in product line, for sensors, and sampling liquid and vapour state (Fig. 12).



Advantages

- flexible and easy to handle
- connection with tubes, pipes and rods of different materials
- vacuum-tight
- safe under overpressure up to 5 bar
- product touched PTFE inserts, PTFE screwing cap (glass fibre reinforced)



Use

- product line made of PTFE tube, glass and metal pipes
- sensors made of glass, metal and plastics
- sampling liquid and vapour state

Figure 12. Glass thread with plastics screwing and product touched PTFE inserts according to the cutting ring principle.

The Miniplant concept and technique for experimental studies combined with PC process simulation belongs to the future in the laboratories with pilot plant operations. Miniplants are necessary for developing new products at low costs and working with standardized glass apparatus and equipment.

Optical Cooling and Trapping of Radioactive Atoms

by
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At Los Alamos National Laboratory, light pressure is used to slow atoms in a gas and hold them at the intersection of six laser beams. The trapped atoms are levitated by the lasers, pinned in the middle of the glass vacuum cell.

There are several reasons why laser traps are a useful tool for studying radioactive atoms.

1. We want to study man-made radioactive atoms, which are difficult and expensive to make. Traps cool atoms down so they move very slowly. The atoms are moving so slowly that they have one millionth the speed of room temperature atoms. This means we can recycle atoms in a trap, testing and re-testing them a million times, whereas room temperature atoms could escape after just one measurement.

2. Radioactive atoms disintegrate with time. With an atom trap, we can do experiments with radioactive atoms within 10 seconds of making them. There are many isotopes with lifetimes of a few minutes or hours that would disintegrate before they could be studied. By using an atom trap, we can study many isotopes that could not be studied before.

3. You can detect a single atom in a trap. An atom is held in a trap for such a long time, that it emits enough light during this time for us to see even a single atom. We can set up a trap to look for exotic radioactive atoms that are by-products of nuclear weapon production. This application may be important for nuclear treaty verification.

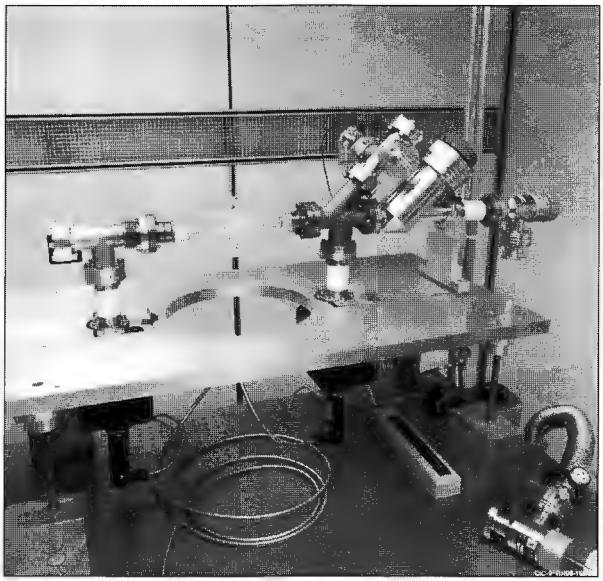

photo 1

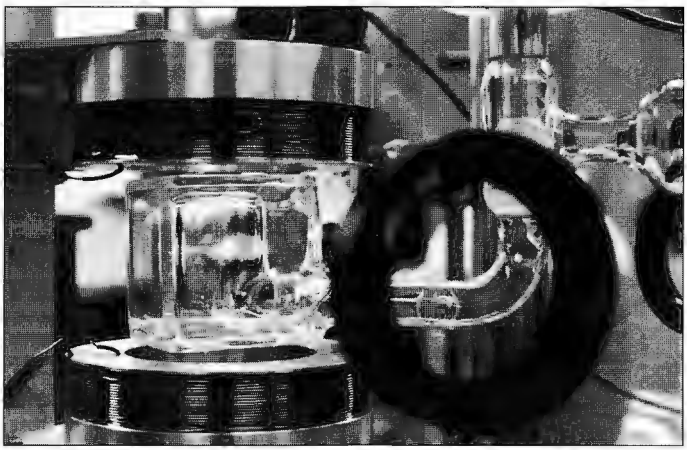

photo 2

Acknowledgments: John Flower and Gary Warren-Photography and Jacob Bartos-MST-7 Machine Shop.

Oxygen Displacement and Encapsulation of Temperature Sensitive Materials

by John L.A. French
M.P.B. Technologies, Inc.
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H9R 1E9

Procedures for encapsulation of materials within a suitable ampoule followed by baking and pumping down to low pressure is commonplace in most glass blowing facilities. The familiar heat treatment for borosilicate glassware, pumping at 450°C for 30 minutes followed by overnight pumping at 400°C, only applies to vacuum vessels. The encapsulation of materials, either as specimens or for subsequent treatment, demands that the physical and chemical properties of the sample be taken into careful consideration. In the case of encapsulating specimens, it is usually sufficient to evacuate to fore-vacuum levels and seal off.

Those materials destined for further study frequently require the removal of as much oxygen and other atmospheric gases as possible. Some materials may tolerate fusion which allows for a reasonably elevated pumping temperature. The same does not hold true for materials which would interact prematurely, or, as is often the case, one of the components will sublime at a relatively low temperature.

The lower the temperature tolerance the more difficult it is to extract absorbed and adsorbed gases. A partial solution to the problem depends on extended pumping times associated with purging with an inert gas. Argon has been found to give the best results because of its atomic radius which is significantly larger than helium which has a tendency to slip into small spaces and be pumped out again without significantly displacing other molecules.

Annealing ovens are normally used in conjunction with a pumping system to evacuate vessels. However, the extended heating cycles required by the demands of low temperature render the use of the oven impractical. An alternative to the problem lies in the adoption of an ABDERHALDEN DRYING APPARATUS - more familiarly referred to as a drying pistol.

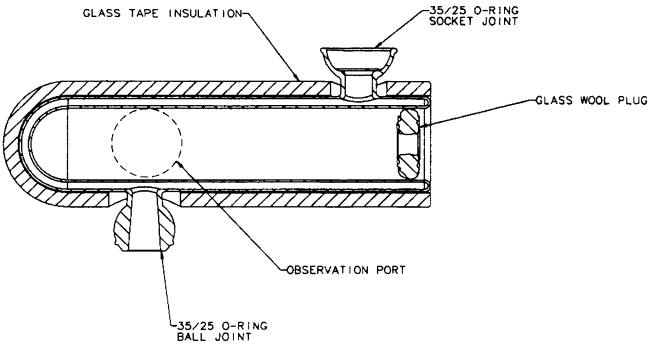

Figure 1.

Basically, a tube containing material to be dried is surrounded by steam while being subjected to a partial vacuum, often a water-operated aspirator pump capable of reaching as low as 15mm-Hg. The beauty of the device lies in the fact that temperature control is automatic and accurate.

The ampoules used in encapsulation have long tubulations which can either be sealed off short as a second step, or kept long to permit the contents to be inserted into a furnace to be subjected to other treatments. It is important to take into account the overall dimensions of the ampoule when determining the length of the inner chamber of the apparatus, bearing in mind the need to heat as much of the tubulated ampoule as possible. No specific dimensions have been set out since these will be determined by the requirements of the project. The spherical O-Ring joints are 35/25, while the boiler for temperatures up to 150°C can be a 1 litre flask. For more elevated temperatures, a 2 litre flask is desirable in order to supply sufficient vapour. When using the maximum temperature range on a long stemmed ampoule, the temperature gradient will be such that the greater part of vapour condensation will occur before reaching the condenser outlet. Nevertheless, gradients aside, the material within the ampoule will have received the maximum desired temperature. Due to the flammable nature of the solvents, it is vital to use a heating mantel. Furthermore, the choice of spherical joints over tapered joints allows for a reduction of stress within the system. It is important to select O-Ring material which is compatible with the solvent of choice. KALREZ, with a temperature tolerance up to 320°, is an ideal choice because of its inertness to the solvents. A list of suitable solvents with their boiling points is set out in Table 1.

Table I

Boiling Points of Organic Solvents
^{o}C

ETHYL ETHER	34.6
ACETONE	56.5
METHYL ALCOHOL	64.7
CARBON TETRACHLORIDE	76.8
ISO PROPYL ALCOHOL	82.5
ISO PROPYL ACETATE	101.6
ISO BUTYL ALCOHOL	108.0
n-BUTYL ALCOHOL	117.4
METHYL CELLOSOLVE	124.5
CELLOSOLVE	134.8
n-AMYL ALCOHOL	137.9
n-AMYL ACETATE	148.0
ETHYL LACTATE	155.0
FURFURAL	161.0
BUTYL CELLOSOLVE	170.6
ETHYL ACETO ACETATE	180.0
DIETHYL OXALATE	186.0
n-BUTYL LACTATE	195.0
CARBITOL	198.0
BENZYL ALCOHOL	205.0
NITROBEZENE	211.0
n-BUTYL STEARATE	220.0
DIETHYLENE GLYCOL	250.0
DIACETIN	261.0
DIMETHYL PHTHALATE	282.0
DIETHYL PHTHALATE	298.0
DIBUTYL PHTHALATE	340.0
DIAMYL PHTHALATE	344.0

In addition to providing adequate evaporation from the boiler, in order to maintain good overall temperature gradients, thermal insulation of the jacket is paramount. At least three layers of woven glass tape should be wrapped around the outer jacket, and an observation port adjacent to the vapour inlet tube is an asset.

The modus operandi is controlled by sealing the tubulated end of the ampoule to a powder trap which is in turn sealed to a T-piece having two valves. One valve is connected to the vacuum pump, while the second one connects to the argon source and incorporates a manometer in order to control the purging cycle pressure. A convenient arrangement is illustrated in Fig. II.

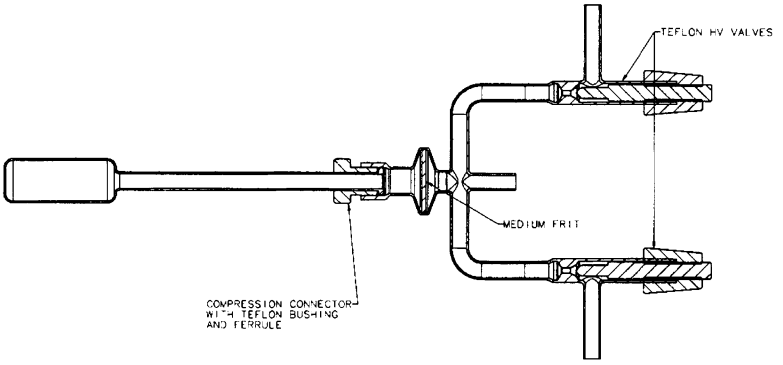

Figure II a.

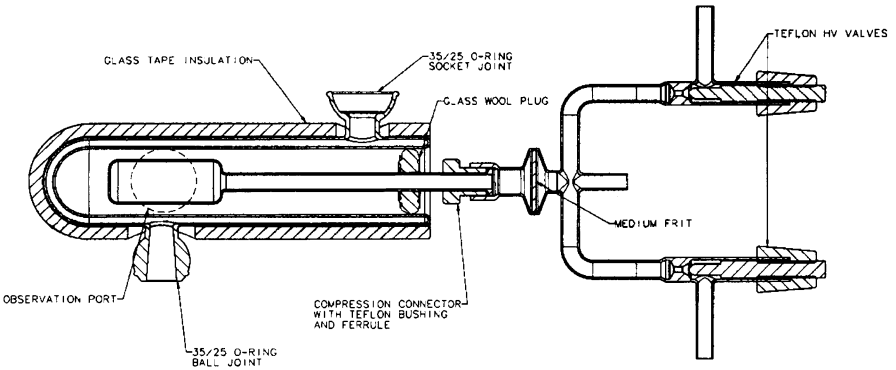

Figure II b.

A threaded compression connector with a teflon ferrule[1] couples the ampoule to the system allowing for the use of quartz as well as glass ampoules. The powder trap is important when dealing with lightweight materials present within the mixture as a fine powder. When faced with this type of problem it is very important to gently open the system to vacuum. A sudden drop in pressure will cause lightweight materials to be drawn towards the vacuum pump. In the absence of a physical barrier (in this case a medium fritted disc 40 to 50mm diameter), the first point of entrapment will be the oil in either the mechanical pump or the diffusion pump. This results in a very costly clean up.

After the initial pump down, argon is admitted until the pressure indicated by the manometer reaches a few millimeters of Hg. Allowing the purging gas pressure to rise to high levels recreates the problem of transporting lightweight material where it is not wanted. Although the fritted disc does slow down the pumping speed, its effect is negligible because of the extended time period of the process which may take several days. The purging process only takes a few minutes repeated at two or three hourly intervals throughout the day. There are no guide lines which can provide a general overview because each procedure is controlled by the nature of the material. The procedure outlined above is far from ideal but it does offer an acceptable solution to the dilemma of reducing oxygen concentrations in temperature intolerant materials.

I would like to express my appreciation for the excellent illustrations prepared by Sander Boelen of the Robotics Division of MPBT.

[1] Ace connectors 7644, teflon bushing 5029, ferruel teflon 11710.

Practical Calculations for Glassware Design

by
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As Scientific Glassblowers, we are often called upon to aid in the design of laboratory glassware which is a specific size, shape, and/or volume, made for a particular purpose. This paper will cover simple calculations for volume and surface area of varying shapes and sizes of glassware, including cylinders, coils (helix), spheres, cones, and some other shapes.

CYLINDERS:

The cylinder or tube is our most commonly used starting material. Refer to handout page 11 for the basic formulas concerning cylinders. The two main factors to consider with cylinders are Volume and Lateral Area, or Surface Area. Refer again to your handout page 1 for the Standard Wall U.S. made tubing. There are 9 other pages with this format which includes: Special, Medium, Heavy Wall, and Duran tubing. These give you a quick easy reference for volume information. An example of how to use this chart follows: Dr. Wheezus in the analytical lab needs you to cut a cylinder of standard wall tubing to contain 2000ml (cc) which is 3-31/2" diameter and between 12-18" length. There are two sizes of standard wall in this size range - 80 and 85. Look at the column for Internal Volume in cc/cm of length and you will find the values of 44.4 for 80 and 50.5 for 85. To determine the length needed for 2000ml, simply divide 2000 by the cc/cm figure.

$$\frac{2000}{44.4} = 45\text{cm}(17.7") \qquad \frac{45\text{cm}}{2.54\text{cm/in}} = 17.72"$$

$$\frac{2000}{50.5} = 39.6\text{cm}(15.6") \qquad \frac{39.6\text{cm}}{2.54\text{cm/in}} = 15.6"$$

So, Dr. Wheezus has two choices: one unit is a little shorter than the other. Be certain to cut the cylinder accurately, because when he seals the ends with duct tape, he wants exactly 2000ml.

I did not include a column on the charts for the surface area of each size of tubing because it is used far less often than volume.

Surface Area is most important when designing a device which is used as a heat exchanger or condenser. Engineers and/or chemists may come to you with a request for an exchanger of a specific Surface Area. You must then calculate the surface areas of the various shapes and sizes of glass surfaces in the device and add them up to see what you have. You can then adjust the size and length of the device and arrive at the correct surface area. I would suggest calculating your areas in sq. cm. and convert to sq. in. if desired (1 sq.in. = 6.4516 sq.cm.). Formulas for Surface Areas of various shapes may be found in the handout pages 11, 12, 14 & 15. An important modified form of cylinders is the coil.

COILS

Coils are used to greatly increase the Surface Area in a given space. In order to calculate the Surface Area or Volume of a coil, we must convert it mathematically into a cylinder. The following is a simple technique which is relatively accurate and errs on the small side concerning length. Most designers of heat exchange equipment include a "fudge factor" for some extra capacity. Let's say we have a coil made by winding 10mm od tubing on a 1" mandrel. What is the length of the coil? You take the diameter of the mandrel (25.4mm) and add the diameter of the tubing to get the centerline diameter of the coil: 25.4 + 10mm = 35.4 mean diameter. To get the approximate length, count the number of complete coils, and multiply by the mean diameter in cm; π x 3.54 = 11.12cm length per coil. The Surface Area of 10mm tubing is π times the od of the tubing in cm or π x 1.0 = 3.1416 sq. cm/cm length. So, for a set of 4.75 coils made in this way,

4.75 x 3.1416 sq. cm x 11.12 = about 166sq.cm of Surface Area

If you need this in sq. inches divide by 6.4516 cm^2/in^2

$$\frac{166}{6.4516} = 25.73 \text{ sq.in.}$$

The Surface Area of a tube varies directly as the diameter. If you increase the size of tubing used for a coil from 8mm od to 10mm od and use the same length, your surface area will increase 25%.

SPHERES

The second most common shape encountered in our work is the sphere. Refer to handout page 14 for basic formulas concerning spheres.

$$\text{VOLUME} = \frac{\pi \times d^3}{6}$$

This is used to calculate the volume of a sphere of a known diameter. If you know the volume desired, use $D = \sqrt[3]{\frac{6 \times v}{\pi}}$ to calculate the diameter of the spheres.

Example: Design a gas bomb with hemispherical ends using 70mm standard wall to have a volume of 500ml. The id of 70mm standard is 6.52cm.

$$V = \frac{\pi \times d^3}{6} \quad \text{or} \quad \frac{3.1416 \times 6.52^3}{6} = 145.12\text{ml volume - sphere of 70mm standard wall}$$

The volume of a 70cm standard wall tube is 33.39ml/cm.
Subtract the volume of the sphere from 500 (500 - 145 = 355ml). You then need 355ml @ 33.39ml/cm length or 355 x 33.39) = 10.6cm length of straight wall 70 standard. The overall length = 1 sphere or 70mm plus 106mm straight wall

$$70 + 106 = \text{oa length}$$

You will find values for the volume of most spheres or hemispherical ends on each tubing specification sheet.

CONES

Two other important shapes are the cone and the frustrum of a cone - see handout page 15. These are useful in determining designing values when these shapes are incorporated in equipment designs. Let's design a gas bomb using conical ends instead of hemispherical ends and see if there is a significant difference in the length. We will use a design where the internal length, h, of the cone is equal to the od of the tubing. So if

$V = \frac{\pi \times r^2 h}{3}$ or $\frac{3.1416(3.26 \times 3.26)7}{3}$ 77.9cc vol. of cone

2 x 77.9cc= 155.8cc 500cc - 155.8cc= 344.2 $\frac{344.2}{33.39}$ = 10.31 OR

103mm of straight wall 70 standard needed 103 + 70 + 70 = 243mm overall length.

Hopefully, this collection of formulas, data, and examples of their use will help you impress your local professor with your design prowess and ability to give him/her just what they need.

Standard Wall PYREX®

Size O.D. mm.	ID in mm	Total Var. O.D. ± mm.	Wall mm.	Total Var. Wall ± mm.	Internal Volume cc/cm of length	Internal Volume Sphere cc	Approx. Kilos Per Meter	Approx. Lbs. Per Ft.	Approx. Pieces Per Case	Approx. Weight Per Case Lbs.	Approx. Weight Per Case Kilos
2	1.0	.15	.5	.1	--	--	.0052	.0035	357	5	2.27
3	1.8	.2	.6	.1	--	--	.0101	.0068	551	15	6.81
4	2.4	.3	.8	.1	--	--	.018	.012	520	25	11.35
5	3.4	.3	.8	.1	--	--	.024	.016	390	25	11.35
6	4.0	.3	1.0	.1	.126	--	.034	.023	271	25	11.35
7	5.0	.3	1.0	.1	.20	--	.042	.028	223	25	11.35
8	6.0	.3	1.0	.1	.28	--	.049	.033	189	25	11.35
9	7.0	.3	1.0	.1	.38	--	.057	.038	164	25	11.35
10	8.0	.3	1.0	.1	.50	--	.062	.042	148	25	11.35
11	9.0	.3	1.0	.1	.64	--	.070	.047	132	25	11.35
12	10.0	.3	1.0	.1	.79	--	.077	.052	120	25	11.35
13	10.6	.3	1.2	.1	.88	--	.100	.067	93	25	11.35
14	11.6	.3	1.2	.1	1.06	.82	.107	.072	86	25	11.35
15	12.6	.3	1.2	.1	1.25	1.05	.116	.078	80	25	11.35
16	13.6	.3	1.2	.1	1.45	1.32	.124	.083	75	25	11.35
17	14.6	.3	1.2	.1	1.67	1.63	.132	.089	70	25	11.35
18	15.6	.3	1.2	.1	1.9	1.99	.141	.095	65	25	11.35
19	16.6	.3	1.2	.1	2.16	2.40	.149	.100	62	25	11.35
20	17.6	.4	1.2	.1	2.43	2.85	.158	.106	58	25	11.35
22	19.0	.4	1.5	.2	2.84	3.59	.216	.145	43	25	11.35
25	22.0	.4	1.5	.2	3.8	5.58	.247	.166	37	25	11.35
28	25.0	.4	1.5	.2	4.9	8.18	.278	.187	33	25	11.35
30	26.4	.7	1.8	.2	5.47	9.63	.354	.238	26	25	11.35
32	28.4	.7	1.8	.2	6.33	12.0	.379	.255	24	25	11.35
35	31.0	.7	2.0	.2	7.55	15.6	.461	.31	20	25	11.35
38	34.0	.7	2.0	.2	9.08	20.58	.506	.34	18	25	11.35
41	37.0	.9	2.0	.2	10.75	26.5	.551	.37	16	25	11.35
45	41.0	1.0	2.0	.2	13.2	36.1	.610	.41	15	25	11.35
48	44.0	1.0	2.0	.2	15.2	44.6	.640	.43	14	25	11.35
51	47.0	1.0	2.0	.2	17.35	54.4	.684	.46	13	25	11.35
54	49.2	1.0	2.4	.3	19.01	62.4	.863	.58	4	9	4.09
57	52.2	1.0	2.4	.3	21.4	74.8	.908	.61	4	10	4.54
60	55.2	1.1	2.4	.3	23.9	88.0	.982	.66	4	10	4.54
64	59.2	1.2	2.4	.3	27.5	108.6	1.027	.69	4	11	4.99
70	65.2	1.2	2.4	.3	33.39	145.0	1.116	.75	4	12	5.45
75	70.2	1.3	2.4	.3	38.7	181.0	1.205	.81	4	13	5.90
80	75.2	1.3	2.4	.3	44.4	222.0	1.309	.88	4	14	6.36
85	80.2	1.3	2.4	.3	50.5	270.0	1.399	.94	4	15	6.81
90	85.2	1.3	2.4	.3	57.0	324.0	1.488	1.00	4	16	7.26
95	90.2	1.4	2.4	.3	63.9	384.0	1.577	1.06	4	17	7.72
100	95.2	1.4	2.4	.3	71.18	452.0	1.667	1.12	4	18	8.17
110	104.8	1.8	2.6	.3	86.26	603.0	1.949	1.31	4	21	9.53
120	114.0	1.8	3.0	.3	102.1	776.0	2.470	1.66	4	27	12.26
125	119.0	1.8	3.0	.3	111.2	882.0	2.574	1.73	4	28	12.71
130	124.0	1.8	3.0	.3	120.7	998.0	2.678	1.80	4	29	13.17
140	133.0	2.0	3.5	.4	138.9	1232.0	3.363	2.26	2	18	8.17
150	143.0	2.0	3.5	.4	160.6	1531.0	3.690	2.48	2	20	9.08
178	171.0	2.2	4.1	.4	229.5	2618.0	4.30	2.89	2	26	11.80

Special Wall PYREX®

Size O.D. mm.	ID in	ID mm	Total Var. O.D. ± mm.	Wall mm.	Total Var. Wall ± mm.	Internal Volume cc/cm of length	Internal Volume Sphere cc	Approx. Kilos Per Meter	Approx. Lbs. Per Ft.	Approx. Pieces Per Case	Approx. Weight Per Case Lbs.	Approx. Weight Per Case Kilos
8	5.0		.3	1.5	.2	.20	--	.0684	.046	136	25	11.35
8	4.0		.3	2.0	.2	.126	--	.0848	.057	109	25	11.35
9	6.0		.3	1.5	.2	.28	--	.0789	.053	117	25	11.35
9	5.0		.3	2.0	.2	.20	--	.0982	.066	94	25	11.35
10	7.0		.3	1.5	.2	.38	--	.0893	.060	104	25	11.35
10	6.0		.3	2.0	.2	.28	--	.1131	.076	82	25	11.35
11	8.0		.3	1.5	.2	.50	--	.0997	.067	93	25	11.35
11	7.0		.3	2.0	.2	.38	--	.1265	.085	73	25	11.35
14	10.8		.3	1.6	.2	.92	.66	.1384	.093	67	25	11.35
19	14.5		.3	2.25	.2	1.65	1.60	.2649	.178	35	25	11.35
22	17.5		.4	2.25	.3	2.41	2.81	.3125	.210	30	25	11.35

Medium Wall PYREX®

Size O.D. mm.	ID in	ID mm	Total Var. O.D. ± mm.	Wall mm.	Total Var. Wall ± mm.	Internal Volume cc/cm of length	Internal Volume Sphere cc	Approx. Kilos Per Meter	Approx. Lbs. Per Ft.	Approx. Pieces Per Case	Approx. Weight Per Case Lbs.	Approx. Weight Per Case Kilos
6.3	3.9		.3	1.2	.1	.119	--	.0432	.029	276	32	14.53
12.7	9.5		.3	1.6	.2	.709	.45	.1235	.083	96	32	14.53
15.9	12.7		.3	1.6	.2	1.267	1.07	.1577	.106	75	32	14.53
19.0	15.8		.3	1.6	.2	1.96	2.06	.1949	.131	61	32	14.53
25.4	20.6		.4	2.4	.3	3.33	4.58	.3869	.26	31	32	14.53
31.7	26.9		.7	2.4	.3	5.68	10.2	.4910	.33	24	32	14.53
38.1	33.3		.7	2.4	.3	8.71	19.3	.6101	.41	18	30	13.62
44.4	39.6		1.0	2.4	.3	12.32	32.5	.7142	.48	15	29	13.17
50.8	44.4		1.0	3.2	.3	15.48	45.8	1.0714	.72	4	12	5.45
57.1	50.7		1.0	3.2	.4	20.19	68.2	1.2202	.82	4	13	5.90
63.5	57.1		1.4	3.2	.4	25.61	97.5	1.3541	.91	4	15	6.81
69.8	63.4		1.5	3.2	.4	31.57	133.4	1.5029	1.01	4	16	7.26
76.2	69.8		1.5	3.2	.4	38.27	178.0	1.6368	1.10	4	18	8.17
82.5	76.1		1.7	3.2	.4	45.48	231.0	1.7856	1.20	4	19	8.63
88.9	82.5		1.8	3.2	.4	53.46	294.0	1.9344	1.30	4	21	9.53
101.6	92.0		1.8	4.8	.5	66.48	408.0	3.2290	2.17	4	35	15.89
114.3	104.7		1.8	4.8	.5	86.10	601.0	3.6456	2.45	2	20	9.08

Heavy Wall PYREX®

Size O.D. mm.	ID in mm	Total Var. O.D. ± mm.	Wall mm.	Total Var. Wall ± mm.	Internal Volume cc/cm of length	Internal Volume Sphere cc	Approx. Kilos Per Meter	Approx. Lbs. Per Ft.	Approx. Pieces Per Case	Approx. Weight Per Case Lbs.	Kilos
9.5	5.5	.3	2.0	.2	.238	.09	.1042	.07	96	27*	12.23
12.7	7.9	.3	2.4	.2	.49	.26	.1637	.11	61	27*	12.23
15.9	11.1	.3	2.4	.2	.968	.72	.2232	.15	45	27*	12.23
19.0	12.6	.3	3.2	.4	1.25	1.05	.3571	.24	28	27*	12.23
22.2	15.8	.4	3.2	.4	1.96	2.06	.4166	.28	30	34*	15.44
25.4	17.4	.4	4.0	.4	2.38	2.76	.5952	.40	24	38	17.25
31.7	23.7	.7	4.0	.4	4.41	6.97	.7886	.53	18	38	17.25
38.1	30.1	.7	4.0	.4	7.12	14.28	.9523	.64	12	31	14.07
44.4	36.4	1.0	4.0	.4	10.41	25.3	1.1309	.76	10	30	13.62
50.8	41.2	1.0	4.8	.5	13.33	36.6	1.5624	1.05	4	17	7.72
57.1	47.5	1.0	4.8	.5	17.72	56.1	1.7707	1.19	4	19	8.63
63.5	53.9	1.5	4.8	.5	22.82	82.0	1.9790	1.33	4	21	9.53
69.8	60.2	1.5	4.8	.5	28.46	114.0	2.2022	1.48	4	24	10.90
76.2	66.6	1.5	4.8	.5	34.84	155.0	2.3808	1.60	4	26	11.80
82.5	72.9	1.8	4.8	.5	41.74	203.0	2.6338	1.77	4	29	13.17
88.9	79.3	1.8	4.8	.5	49.39	261.0	2.8421	1.91	4	31	14.07
101.6	88.9	1.8	6.4	.7	61.93	368.0	4.2408	2.85	2	23	10.44
114.3	101.6	1.8	6.4	.7	81.07	549.0	4.7616	3.20	2	26	11.80
127.0	114.3	1.8	6.4	.7	102.61	782.0	5.3568	3.60	2	29	13.17
139.7	127.0	2.0	6.4	.7	126.68	1073.0	5.9520	4.00	2	32	14.53
152.4	136.6	2.0	7.9	.9	146.55	1335.0	8.0352	5.40	2	43	19.52
165.1	149.3	2.0	7.9	.9	175.07	1743.0	8.7792	5.90	1	24	10.90
177.8	158.8	2.2	9.5	1.1	198.06	2097.0	11.2344	7.55	1	30	13.62

DURAN® TUBING

ID in mm	O.D.	WALL	Internal Volume cc/cm of length	Internal Volume Sphere cc	LENGTHS CASE	POUNDS CASE
1.6	3	.7	.020	--	941	35.3
2.4	4	.8	.045	--	555	33.1
3.4	5	.8	.091	--	343	26.5
4.0	6	1.0	.126	--	245	28.7
3.0	6	1.5	.071	--	211	33.1
5.0	7	1.0	.20	--	190	26.5
4.0	7	1.5	.126	--	172	33.1
6.0	8	1.0	.28	--	149	24.3
5.0	8	1.5	.20	--	147	33.1
7.0	9	1.0	.38	--	119	22.0
6.0	9	1.5	.28	--	119	30.9
8.0	10	1.0	.50	--	95	19.8
7.0	10	1.5	.38	--	90	26.5
5.6	10	2.2	.246	--	56	22.0
9.0	11	1.0	.64	--	86	19.8
8.0	11	1.5	.50	--	74	24.3
6.6	11	2.2	.342	--	42	18.7
10.0	12	1.0	.79	--	130	33.1
9.0	12	1.5	.64	--	67	24.3
7.6	12	2.2	.454	--	42	20.9
7.9	12.7	2.4	.49	.26	42	24.0
11.0	13	1.0	.95	.70	119	33.1
10.0	13	1.5	.79	.52	56	22.0
8.6	13	2.2	.58	.33	36	19.8
12.0	14	1.0	1.13	.90	110	33.1
11.0	14	1.5	.95	.70	46	19.8
9.6	14	2.2	.72	.46	30	18.1
12.6	15	1.2	1.25	1.05	86	33.1
11.4	15	1.8	1.02	.78	56	30.9
10.0	15	2.5	.79	.52	25	18.1
13.6	16	1.2	1.45	1.32	81	33.1
12.4	16	1.8	1.21	1.00	49	28.9
11.0	16	2.5	.95	.70	25	19.4
14.6	17	1.2	1.67	1.63	76	33.1

DURAN® TUBING

ID in mm	O.D.	WALL	Internal Volume cc/cm of length	Internal Volume Sphere cc	LENGTHS CASE	POUNDS CASE
13.4	17	1.8	1.41	1.26	49	30.9
12.0	17	2.5	1.13	.90	25	20.9
15.6	18	1.2	1.9	1.99	66	30.9
14.4	18	1.8	1.63	1.56	49	33.1
13.0	18	2.5	1.33	1.15	20	17.9
16.6	19	1.2	2.16	2.40	63	30.9
15.4	19	1.8	1.86	1.91	42	30.2
14.0	19	2.5	1.54	1.44	36	34.4
12.6	19	3.2	1.25	1.05	35	20.8
17.6	20	1.2	2.43	2.85	55	28.7
16.4	20	1.8	2.11	2.31	36	27.3
15.0	20	2.5	1.77	1.77	20	20.3
19.6	22	1.2	3.02	3.94	42	24.3
18.4	22	1.8	2.66	3.26	30	25.4
17.0	22	2.5	2.27	2.57	30	34.0
21.6	24	1.2	3.66	5.28	38	24.3
20.4	24	1.8	3.27	4.45	25	23.1
19.0	24	2.5	2.84	3.59	25	30.9
22.4	25.4	1.5	3.94	5.88	25	20.8
23.2	26	1.4	4.23	6.54	33	26.5
22.0	26	2.0	3.80	5.58	25	27.8
20.4	26	2.8	3.27	4.45	12	18.1
25.2	28	1.4	4.99	8.38	25	21.6
24.0	28	2.0	4.52	7.24	20	24.3
22.4	28	2.8	3.94	5.88	20	32.6
27.2	30	1.4	5.81	10.54	36	33.5
26.0	30	2.0	5.31	9.20	16	20.7
24.4	30	2.8	4.68	7.61	16	28.2
29.2	32	1.4	6.70	13.00	25	24.9
28.0	32	2.0	6.16	11.50	16	22.3
26.4	32	2.8	5.47	9.63	16	30.4

DURAN® TUBING

ID in mm	O.D.	WALL	Internal Volume cc/cm of length	Internal Volume Sphere cc	LENGTHS CASE	POUNDS CASE
29.0	33	2.0	6.60	12.77	25	35.7
31.2	34	1.4	7.65	15.90	25	26.7
30.0	34	2.0	7.07	14.14	16	23.8
28.4	34	2.8	6.33	12.00	16	32.6
33.2	36	1.4	8.66	19.16	25	27.8
32.0	36	2.0	8.04	17.16	25	39.7
30.4	36	2.8	7.26	14.71	12	25.8
35.2	38	1.4	9.73	22.84	20	23.8
34.0	38	2.0	9.08	20.58	20	33.1
32.4	38	2.8	8.24	17.81	9	20.7
36.8	40	1.6	10.64	25.53	16	22.5
37.4	40	2.3	10.99	27.39	16	32.2
33.6	40	3.2	8.87	19.86	9	24.7
30.0	40	5.0	7.07	14.13	9	36.4
38.8	42	1.6	11.82	30.58	16	24.0
37.4	42	2.3	10.99	27.39	16	33.7
35.6	42	3.2	9.95	23.62	9	25.8
40.8	44	1.6	13.07	35.56	16	25.1
39.4	44	2.3	12.19	32.02	16	35.3
37.6	44	3.2	11.10	27.83	9	27.3
35.0	45	5.0	9.62	22.44	9	41.7
42.8	46	1.6	14.39	41.05	16	26.2
41.4	46	2.3	13.46	37.15	9	20.9
39.6	46	3.2	12.32	32.51	9	28.7
44.8	48	1.6	15.76	47.07	16	27.3
43.4	48	2.3	14.79	42.80	16	38.8
41.6	48	3.2	13.59	37.69	6	19.8
46.4	50	1.8	16.91	52.31	9	18.1

DURAN® TUBING

ID in mm	O.D.	WALL	Internal Volume cc/cm of length	Internal Volume Sphere cc	LENGTHS CASE	POUNDS CASE
45.0	50	2.5	15.90	47.71	9	24.7
43.0	50	3.5	14.52	41.62	9	34.0
40.0	50	5.0	12.57	33.51	6	31.1
36.0	50	7.0	10.18	24.42	6	41.9
32.0	50	9.0	8.04	17.16	6	51.1
48.4	52	1.8	18.40	59.36	9	18.7
47.0	52	2.5	17.35	54.36	9	25.8
45.0	52	3.5	15.90	47.71	9	35.3
50.4	54	1.8	19.95	67.03	9	19.6
49.0	54	2.5	18.86	61.60	9	26.9
47.0	54	3.5	17.35	54.36	9	36.8
45.0	55	5.0	15.90	47.71	4	23.1
52.4	56	1.8	21.57	75.33	9	20.3
51.0	56	2.5	20.43	69.46	9	27.8
49.0	56	3.5	18.86	61.60	9	38.6
54.4	58	1.8	23.24	84.29	9	21.2
53.0	58	2.5	22.06	77.95	9	28.9
51.0	58	3.5	20.43	69.46	9	39.7
55.6	60	2.2	24.28	90.00	9	26.5
53.6	60	3.2	22.56	80.63	9	37.9
51.6	60	4.2	20.91	71.94	4	21.6
50.0	60	5.0	19.63	65.45	4	25.4
46.0	60	7.0	16.62	50.97	4	34.4
42.0	60	9.0	13.85	38.79	4	42.5
60.6	65	2.2	28.84	116.52	8	25.8
58.6	65	3.2	26.97	105.36	4	18.3
56.6	65	4.2	25.16	94.94	4	23.6
55.0	65	5.0	23.76	87.11	4	27.8
65.6	70	2.2	33.80	147.81	8	27.6
63.6	70	3.2	31.77	134.70	4	19.8
61.6	70	4.2	29.80	122.39	4	25.6
60.0	70	5.0	28.27	113.10	4	30.0
56.0	70	7.0	24.63	91.95	4	40.8
52.0	70	9.0	21.24	73.62	4	50.9
70.6	75	2.2	39.15	184.25	8	29.8
68.6	75	3.2	36.96	169.03	4	21.4
66.6	75	4.2	34.84	154.68	4	27.6

DURAN® TUBING

ID in mm	O.D.	WALL	Internal Volume cc/cm of length	Internal Volume Sphere cc	LENGTHS CASE	POUNDS CASE
65.0	75	5.0	33.18	143.80	4	32.4
75.0	80	2.5	44.18	220.90	4	18.1
73.0	80	3.5	41.85	203.70	4	24.9
70.0	80	5.0	38.48	179.60	4	34.8
62.0	80	9.0	30.19	124.80	4	59.1
80.0	85	2.5	50.27	268.10	4	19.2
78.0	85	3.5	47.78	248.50	4	26.5
75.0	85	5.0	44.18	220.90	4	37.0
85.0	90	2.5	56.75	321.60	4	20.3
83.0	90	3.5	54.11	299.40	4	28.0
80.0	90	5.0	50.27	268.10	4	39.5
76.0	90	7.0	45.36	229.80	3	40.3
72.0	90	9.0	40.72	195.40	3	50.7
90.0	95	2.5	63.62	382.00	4	21.4
88.0	95	3.5	60.82	357.00	4	29.5
85.0	95	5.0	56.75	372.00	4	41.7
95.0	100	2.5	70.88	449.00	4	22.7
94.0	100	3.0	69.40	435.00	4	26.7
93.0	100	3.5	67.93	421.00	3	23.6
90.0	100	5.0	63.62	382.00	3	33.3
86.0	100	7.0	58.09	333.00	3	45.2
82.0	100	9.0	52.81	289.00	3	56.9
99.0	105	3.0	76.98	508.00	3	21.2
95.0	105	5.0	70.88	449.00	3	34.8
104.8	110	2.6	86.26	603.00	4	25.8
104.0	110	3.0	84.95	589.00	3	22.3
100.0	110	5.0	78.54	524.00	3	36.4
96.0	110	7.0	72.38	463.00	3	50.0
109.0	115	3.0	93.31	678.00	4	31.1
105.0	115	5.0	86.59	606.00	2	25.6
101.0	115	7.0	80.12	539.00	2	35.1

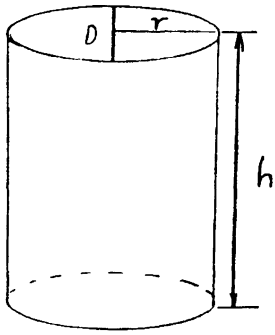
DURAN® TUBING

ID in mm	O.D.	WALL	Internal Volume cc/cm of length	Internal Volume Sphere cc	LENGTHS CASE	POUNDS CASE
114.0	120	3.0	102.07	776.00	4	32.4
110.0	120	5.0	95.03	697.00	2	26.7
106.0	120	7.0	88.25	624.00	2	36.6
102.0	120	9.0	81.71	556.00	2	46.3
115.0	125	5.0	103.87	796.00	2	27.8
107.0	125	9.0	89.92	641.00	2	48.3
124.0	130	3.0	120.76	998.00	4	35.3
120.0	130	5.0	113.10	905.00	2	28.9
116.0	130	7.0	105.68	817.00	2	39.9
112.0	130	9.0	98.52	736.00	2	50.5
125.0	135	5.0	122.72	1023.00	2	30.2
121.0	135	7.0	114.99	928.00	2	41.4
134.0	140	3.0	141.03	1260.00	4	38.1
130.0	140	5.0	132.73	1150.00	2	31.3
126.0	140	7.0	124.69	1047.00	2	43.2
135.0	145	5.0	143.14	1288.00	2	32.4
144.0	150	3.0	162.86	1563.00	2	20.6
140.0	150	5.0	153.94	1437.00	2	33.5
136.0	150	7.0	145.27	1317.00	2	46.3
132.0	150	9.0	136.85	1204.00	2	58.9
145.0	155	5.0	165.13	1596.00	2	34.8
150.0	160	5.0	176.71	1767.00	2	35.9
146.0	160	7.0	167.42	1630.00	2	49.6
155.0	165	5.0	188.69	1950.00	2	37.0
151.0	165	7.0	179.08	1803.00	2	51.1
160.0	170	5.0	201.06	2145.00	2	23.2
156.0	170	7.0	191.13	1988.00	2	52.9
152.0	170	9.0	181.46	1839.00	1	33.6
170.0	180	5.0	226.98	2572.00	1	20.3
166.0	180	7.0	216.42	2395.00	1	28.0
162.0	180	9.0	206.12	2226.00	1	35.6
180.0	190	5.0	245.47	3053.00	1	21.4
176.0	190	7.0	243.28	2855.00	1	29.7

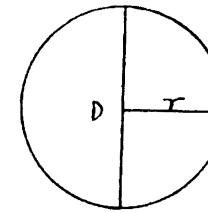
DURAN® TUBING

ID in mm	O.D.	WALL	Internal Volume cc/cm of length	Internal Volume Sphere cc	LENGTHS CASE	POUNDS CASE
190.0	200	5.0	283.50	3591.00	1	22.5
186.0	200	7.0	271.70	3369.00	1	31.3
182.0	200	9.0	260.20	3157.00	1	39.8
201.0	215	7.0	317.30	4252.00	1	33.7
197.0	215	9.0	304.80	4003.00	1	42.9
211.0	225	7.0	349.70	4919.00	1	35.3
207.0	225	9.0	336.50	4644.00	1	45.0
222.0	240	9.0	387.10	5729.00	1	48.1
240.0	250	5.0	452.00	7238.00	1	28.4
236.0	250	7.0	437.00	6882.00	1	39.4
232.0	250	9.0	423.00	6538.00	1	50.2
260.0	270	5.0	531.00	9203.00	1	30.7
256.0	270	7.0	515.00	8785.00	1	42.6
252.0	270	9.0	499.00	8379.00	1	54.5
290.0	300	5.0	661.00	12770.00	1	34.2
286.0	300	7.0	642.00	12249.00	1	47.5
282.0	300	9.0	625.00	11742.00	1	60.6
301.0	315	7.0	712.00	14279.00	1	49.9
297.0	315	9.0	693.00	13717.00	1	43.8
307.0	325	9.0	740.00	15150.00	1	65.9
305.0	325	10.0	731.00	14856.00	1	73.0

CYLINDER



CIRCLE



LATERAL AREA

$A = 2\pi r^2 h$

or

$A = \pi D h$

AREA

$A = \pi r^2$

CIRCUMFERENCE

$C = 2\pi D$

or

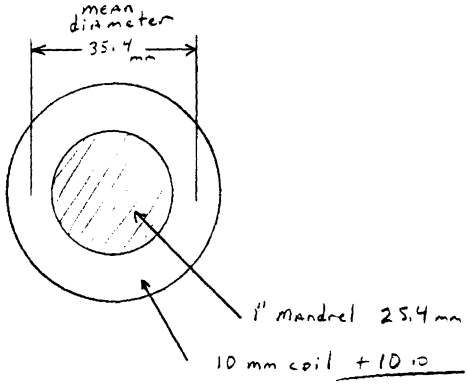
$C = \pi D$

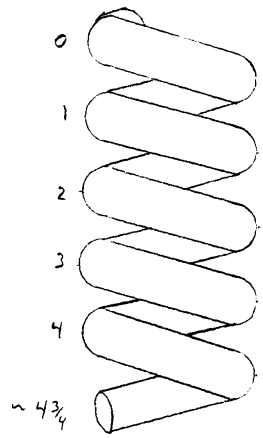
VOLUME

$V = \pi r^2 h$

Calculate Diameter when Length and Volume are given

$$D = \sqrt{\frac{4 \cdot V}{\pi \cdot h}}$$





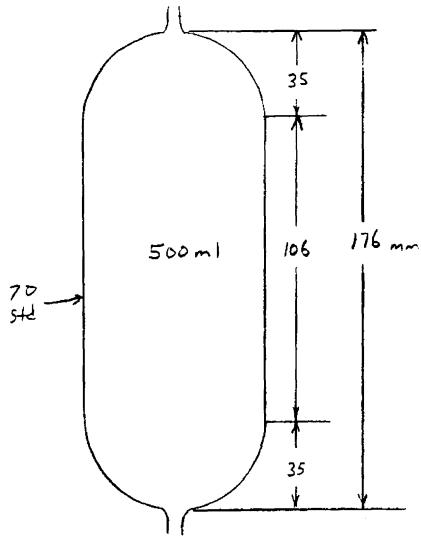
Length = π d cm

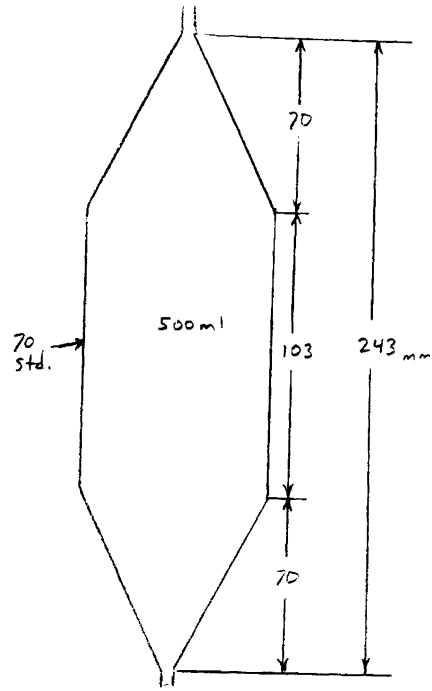
$3.1416 \cdot 3.54_{cm} = 11.12$ cm/coil

Surface area/coil = 1.0 cm · 3.1416 = 3.1416 sq cm/cm length

11.12 cm/coil · 3.1416 sq cm/cm length · 4.75 coil = ~166 sq. cm.

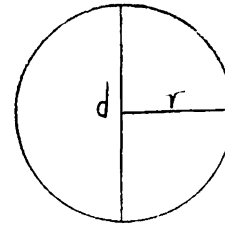
Divide by 6.4516 cm^2/in^2 to get sq. in. $\frac{166}{6.4516} = 25.73$ sq. in





scale: $\frac{1}{2} = 1$

SPHERE



SURFACE AREA

$$A = 4\pi r^2$$

VOLUME

$$V_{cc} = \frac{\pi d^3_{cm}}{6}$$

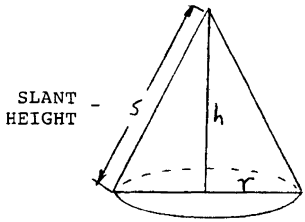
or

$$V_{cc} = (.5236)d^3_{cm}$$

Calculate DIAMETER for a given Volume

$$d_{cm} = \sqrt[3]{\frac{6 \cdot V}{\pi}}$$

RIGHT CIRCULAR CONE



VOLUME

$$V = \frac{\pi r^2 h}{3}$$

or

$$V = 1.047 r^2 h$$

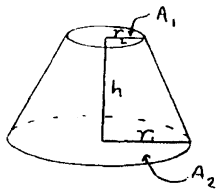
LATERAL SURFACE AREA

$$L = \pi r s$$

or

$$L = \pi r \sqrt{r^2 + h^2}$$

FRUSTRUM of a RIGHT CIRCULAR CONE



VOLUME

$$V = \frac{h}{3}\left(A_1 + A_2 + \sqrt{A_1 \cdot A_2}\right)$$

A_1 = Area of Large Base

A_2 = Area of Small Base

Rotating Spindle Viscometer

by
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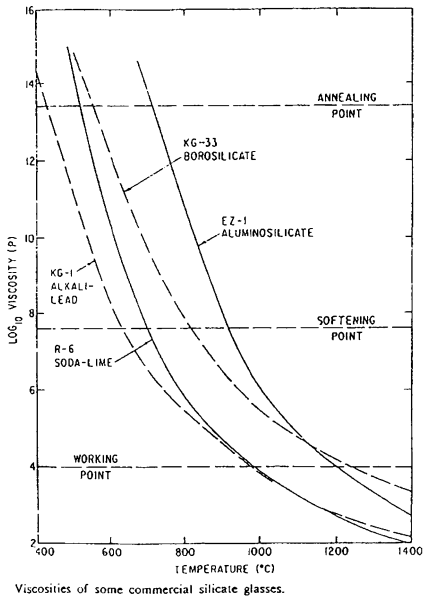
Westinghouse-Savannah River Company
Savannah River Technology Center
Aiken, South Carolina 29808

The viscosity of a glass is one of its most important properties. It determines the melting conditions, the temperature of working and annealing, fining behavior (removal of bubbles from the melt), upper temperature use and devitrification rate. The viscosities of different glasses vary enormously with composition and are strong functions of temperature.

When a shearing force is applied to a liquid, it flows, and the viscosity is a measure of the ratio between the force and the rate of flow. The unit of viscosity is the dyne second per square centimeter which is called the poise (P). Scientifically, this is being replaced by the Pascal Second (1 P = 0.1 Pa s). As a reference, the viscosity of most common fluids such as water and organic liquids is about one-hundredth of a poise at room temperature. Glycerin has a viscosity of about 10 P.

The working point of a glass is defined as the temperature at which it has a viscosity of 10^4P. At this temperature the glass can be readily formed or sealed. The softening point of a glass is the temperature at which it has a viscosity of about 10^8P. The annealing point is the temperature at which the viscosity is 10^{13}P and the strain point is a viscosity of $10^{14.5}$P. The viscosities of several silicate glasses are compared in Figure 1 as a function of temperature. The working, softening and annealing points are marked on the plot.

FIG. 1



Viscosities of some commercial silicate glasses.

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Figure 1.

There are two common methods to measure viscosity in glass. One method deals with low viscosity, from 10 to 10^8P. The other method deals with high viscosity, which is measured by the rate at which a glass rod elongates under a fixed force.[1]

This paper will discuss low viscosity glass above the softening point of $\leq 10^8$P. Two methods with comparable precision and accuracy are commonly used and differ only in the manner of developing spindle torque. One method employs a stationary crucible and a rotating spindle and the other employs a rotating crucible in combination with a fixed spindle.

FIG. 2

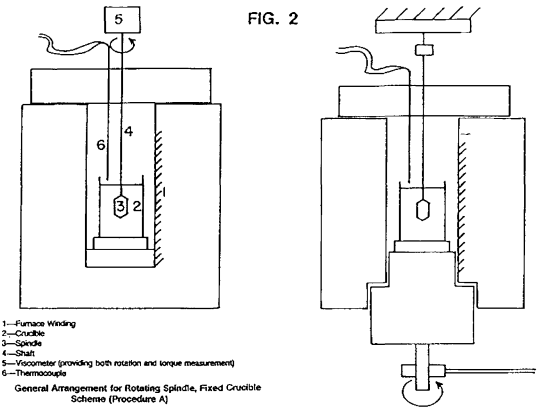

Figure 2.

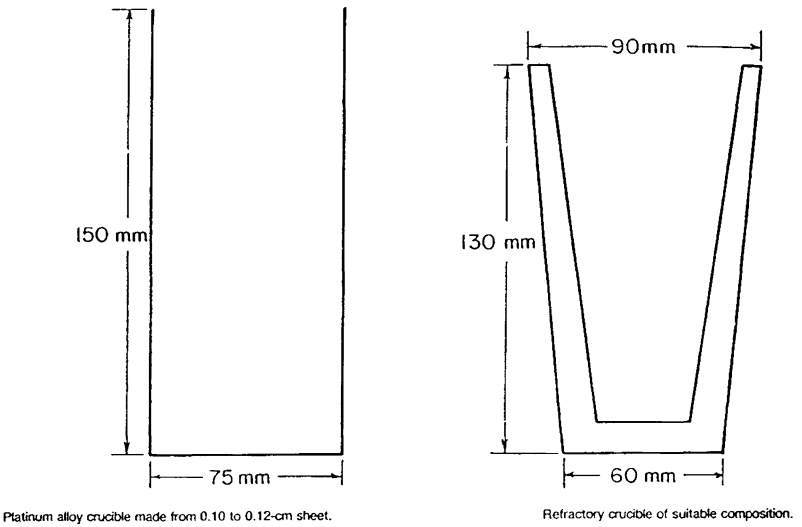

FIG. 3 **Two Types of Crucibles**

Figure 3.

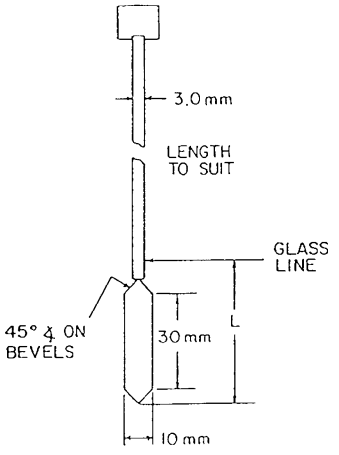

FIG. 4 Typical Platinum Alloy Spindle

Figure 4.

Figure 2 shows the basic design of these two viscosity apparatus. Figure 3 gives the shape and dimensions of the two types of crucibles and Figure 4 shows a typical platinum alloy spindle. Apparatus of these designs are used to determine the viscosity of glass above the softening point through the use of a platinum alloy spindle immersed in a crucible of molten glass. Spindle torque is measured and used to calculate viscosity. Generally, data are taken as a function of temperature to describe the viscosity curve for the glass, usually in the range of 10 P to 10^7P. A step-by-step procedure with the above figures is found in the 1994 ASTM Standards.[2]

The viscosity apparatus in use at the Savannah River Technology Center is a rotating spindle designed and built by GAFTECH of Nashville, Tennessee. It consists of an 80% platinum-20% rhodium alloy crucible 6.35 cm inside diameter by 11.2 cm in height with a 2.5 cm cone bottom. There is a drain hole in the bottom of the crucible that allows it to be drained after each run. The spindle is also a platinum-rhodium alloy 5 cm in length by 1.3 cm outside diameter. Both ends have a 0.5 cm taper with a 3 mm od platinum alloy rod attached to one end. Three sections of this rod are used to attach the spindle to a Brookfield RVDII viscometer. This instrument provides both rotation and torque measurements. The crucible has a pair of platinum alloy terminals welded to either side where copper leads are attached. Heating of the crucible is provided by passage of large electric current through the crucible. The copper leads are water-cooled to prevent melting. Two thermocouples are attached directly to the crucible. If any of the four leads separate from the crucible, the transformer will shut down. The entire crucible is embedded in a KAST-O-LITE 30 castable refractory. The power source is a 20 KVA 480 volt transformer (Figures 5-13).

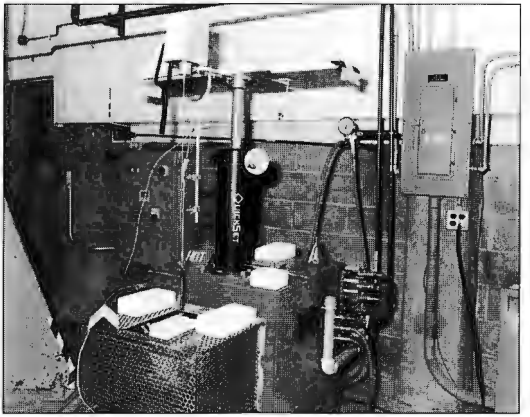

Figure 5

Figure 6

Figure 7

Figure 8

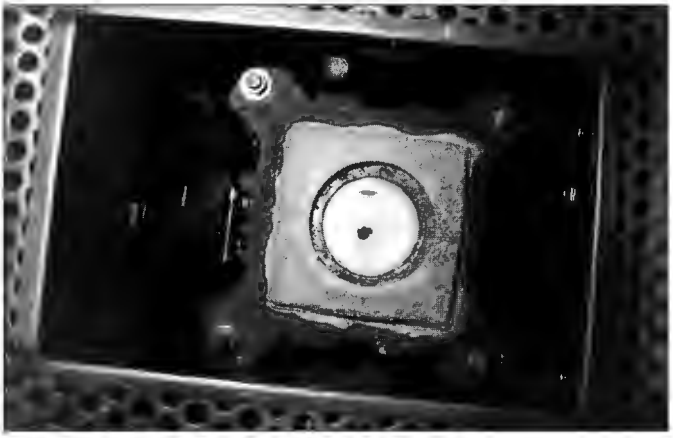

Figure 9

Figure 10

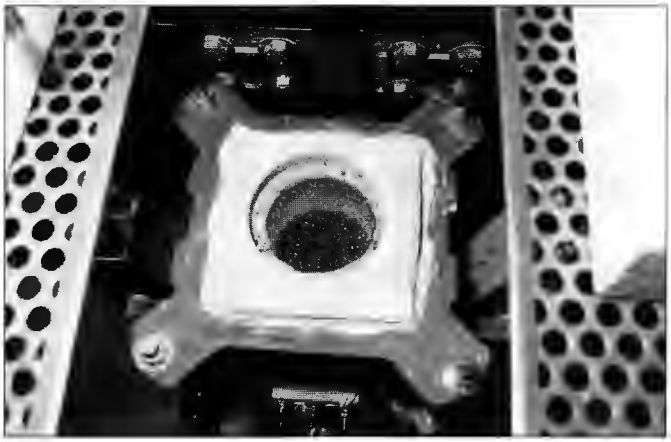

Figure 11

Figure 12

Figure 13

After our apparatus was set up and given a safety OK to operate, several cold runs were made. These were done with Brookfield viscosity standards; they are non-toxic silicone fluids accurate to ± 1% of the stated viscosity. These fluid standards are run to check calibration and mechanical operation of the apparatus. Once satisfied with the performance of the apparatus, a hot run could be made. We chose standard reference material (SRM) sample number 711 which is a lead silicate glass. This was our choice because of the three SRM glass viscosity standards, number 711 has the closest temperature-viscosity relationship to our glasses.

Our viscosity bushing is set with a maximum temperature of 1500°C and we heat at a rate of about 10 degrees per minute. The temperature parameters for each run are determined by the type of glass used. At present we are working with aluminoborosilicates and lanthanide borosilicates. However, several other glasses are also run. One of these is our DWPF (Defense Waste Processing Facility) glass. This is a borosilicate glass high in iron oxide with over 20 components. At DWPF, high-level radioactive waste from Cold War plutonium production is being mixed with a specially formulated borosilicate glass frit and poured into stainless steel canisters, 10-feet-tall, 2 feet in diameter, and sealed for long-term storage. I will discuss how our viscosity apparatus operates using a DWPF and a lanthanide borosilicate glass.

A measured amount of glass chunks or frit is placed in the crucible. We try to keep our melt volume at 250 cc. The unit power is turned on. For DWPF glass, the maximum temperature is set for 1200°C. It takes about two hours to reach maximum temperature. As soon as the glass is fluid, we lower our spindle and melt thermocouple into the glass. The spindle is allowed to rotate, aiding in a more uniform heating of the melt. At maximum temperature, the melt is allowed to equilibrate for about an hour. The melt thermocouple provides the actual melt temperature and in the DWPF glass may vary as much as 50°C from the crucible temperature. The lanthanide glasses typically vary between 20° and 25°C. This temperature variance is due in part to the thermal conductivity and infrared transmission of the different glasses.

To determine the viscosity, we need to measure the torque the glass applies to the turning spindle at given r.p.m.'s at various temperatures. We set our r.p.m.'s to keep the torque between 40-60%. Our first reading is taken at 1200°C for the DPWF glass. The r.p.m.'s at this temperature are about 20-30 and the torque is 50%. The temperature is decreased, the r.p.m.'s adjusted to stay within the 40-60% range, and readings are taken every 25-50°C. The temperature is held at the desired point and the batch allowed to reach a steady state. This take 15 to 20 minutes. This process is repeated until the spindle speed required to keep the 40-60% torque is about 1 r.p.m. This usually means six readings. Figure 14 shows some typical readings from the DPWF glass. After the last reading is taken, the melt is heated back to maximum temperature, the spindle and thermocouple are removed, and the crucible emptied by means of a drain in the bottom.

MELTER TEMP	MELT TEMP	RPM	TORQUE	TORQUE/RPM	VISCOSITY (p)
1200	1155	20	48.3	2.42	60.5
1150	1123	12	41.3	3.44	81.8
1100	1073	10	52.9	5.29	120.0
1050	1026	5	44.5	8.90	194.7
1000	978	4	61.3	15.33	327.7
950	933	2	59.5	29.75	626.3

TYPICAL DWPF GLASS

Figure 14.

The setup for a lanthanide borosilicate glass would be the same except the maximum temperature is 1475°C. The lanthanide series consists of 15 elements and any number of these has been used in different glass samples. A process is being developed to vitrify an actinide series solution using a mixture of eight lanthanide oxides. These are lanthanum, cerium, praseodymium, neodymium, samarium, europium, gadolinium, and erbium. The next figure shows the results of a lanthanide glass run.

MELTER TEMP	MELT TEMP	RPM	TORQUE	TORQUE/RPM	VISCOSITY (p)
1475	1459	60	40.8	0.68	24.6
1400	1386	30	44.2	1.47	41.0
1350	1334	20	56.3	2.82	68.8
1300	1282	10	60.4	6.04	135.5
1250	1231	3	47.4	15.80	337.6
1200	1179	1	46.0	46.0	962.7

TYPICAL LaBS GLASS

(Lanthanide Borosilicate)

Figure 15.

The numbers we are recording are the percent of torque per given r.p.m.'s at a given temperature. For most glasses, the data can be fitted to the following equation:

$$\log_{10} \eta = A + B / T - T_0$$

where T=temperature °C and A, B and T_0=adjustable constants. This formula, known as the "Fulcher Equation," is so widely accepted that Dr. Fulcher will soon disappear from the citation index because his equation has passed into public domain. Dr. Fulcher's original paper was published in the *Journal of the American Ceramic Society,* December 1925, and has proven to be the most accurate three-parameter equation developed to date.[3]

Our viscosity apparatus provides us with a safe, simple and reproducible method for measuring the viscosity of glass. The information obtained has been most helpful in developing new glasses for the vitrification of radioactive and hazardous materials.

I would like to thank Ray Schumacher and John Pareizs of the Vitrification Technology Section, and Amy McIntyre, a summer internist, University of Missouri, Rolla, for their help and assistance in preparing this paper.

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2. The 1994 ASTM (American Society for Testing and Materials) Standards, sect. 15: vol. 15.02 Glass; Ceramic Whiteware: 313-316.

3. *Journal of the American Ceramic Society*, 75.5 (May 1992).

Silvering

by
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Those of us involved in scientific glassblowing for any length of time have gone through several procedures for the silvering of glass vessels that are to be evacuated. When I started my career we used the Brashear process first published by Telescope Maker John Brashear in 1860. Those of you who have worked with this method know of the intricacies of working with it, the careful titration and then the cleaning of the vessels at the finish to remove all of the sludge. Some 25 years ago I finally became aware of the London Labs process and have proceeded happily with it until about 1-1/2 years ago. I had a batch with a remarkable shelf life of about 2-1/2 years. I was beginning to hear from other glassblowers in our Section about problems they were having with new batches. Rick Gerhart at Cal Tech finally called the Company and was told that they had made some subtle changes in the formula to meet the needs of some of their larger customers, mirror makers. Most of us in the Society are small users. At some point in the conversation, he was given the name of Peacock Labs and was told that they could make the old style solutions. It is the using of this solution HE-300 that I will be discussing today.

Cleaning and Preparation

As with any silvering, it is important that the surfaces are clean. Always be careful about selecting tubing or flasks to be used and make sure that they are free of airlines or bubbles. A good cleaning with a commercial glass cleaner before assembling the vessels will save a lot of time later. Also be careful about finger prints. In my Shop I have had good success using household window cleaner in a spray bottle. I feel I should say something about water here. All silvering solutions call for distilled or de-ionized water. In our building we have an in-house Di-water supply; this sometimes goes on the blink. Unless you are the person in charge of the supply, it would be wise to perform a simple test of your water before beginning. I usually put some of the water to be used in a beaker and add a couple of drops of the silver - if it becomes a cloudy white, you know the water is no good.

Silvering

The Peacock Labs formula contains three (3) solutions plus the sensitizer. These come in four (4) equal-size bottles and are mixed in equal amounts. This may be a little difficult at first if you have used the London Labs formula for any length of time. The solutions are labeled A, B, C and #93 (sensitizer). An example of how I mix the solutions follows:

20 mL A - 480 distilled H_2O

20 mL B - 480 distilled H_2O

20 mL C - 480 distilled H_2O

20 mL #93 - 480 distilled H_2O

One way I help to keep things clean in my Shop is I have four (4) 500 ml graduated cylinders marked A, B, C and 93; I keep these just for silvering and always use the same one for the same solution. When you are ready to begin, pour the sensitizer #93 into the vessel and leave it for 10-20 seconds; most of the things I do are shaken. After this is emptied out, rinse three to four times with distilled water. At this point you want to silver immediately. Pour together equal parts of solutions A, B and C into a beaker, stir, then decant into the vessel. You have approximately two minutes to do this and my experience is that you should leave it in for the whole time. After pouring out the spent silvering solution, rinse with distilled water, then dry. I usually dry them in an oven at 110°C. Through a conversation I had with Peacock, I was told that he could make changes to the solution to make it silver faster, if need be. The directions state that solutions A, B, C may be mixed and stored in the refrigerator for up to 30 days. The sensitizer #93 must be mixed up new each time. It is also not a good idea to mix up more than you can use in one hour. I do not have a shelf life for you but I have had my kit for slightly over a year.

Baking and Pumping

Since 1977, I have followed the procedure for baking and pumping outlined by Allan Brown in his paper presented at the 22nd Symposium. However, I have found that with the process described herein I have the most consistent results by only heating to between 450°-475°C. The vessels are sealed to the vacuum line in an oven. The oven is raised to the desired temperature while the vacuum line is open to the atmosphere. When the desired temperature is reached, the line is closed and the mechanical pump started. When the pressure is low enough, the diffusion pump is started and the traps filled (I have a metal oil diffusion pump). I pump at the maximum temperature for one hour while out gassing the system. Then the temperature is lowered to 400°C and pumped to a hard vacuum. With my system that is about 5×10^{-7}.

Re-Silvering

I have had to remove the silver from some things that were done by this process and have found it best to rinse several times with nitric acid as it may otherwise leave traces that cannot be seen by the naked eye. One dewar I thought to be clean and ran through the annealing oven came out a gold color.

Conclusion

In conclusion, I would recommend you try this. I am not trying to sell the product but present it as a satisfactory alternative to other procedures.

References

Peacock Laboratories Inc., Philadelphia, PA.

Brown, Allan B. "Fast Evacuation of Heated Dewars without Silver Loss." Proceedings of the 22nd Symposium of the Art of Scientific Glassblowing. Toledo, Ohio: ASGS, 1977. 85-90.

Manual of Scientific Glassblowing. British Society of Scientific Glassblowing: 1992. 194-195.

Posters

Converting a Milling Machine for Glass Cutting

by
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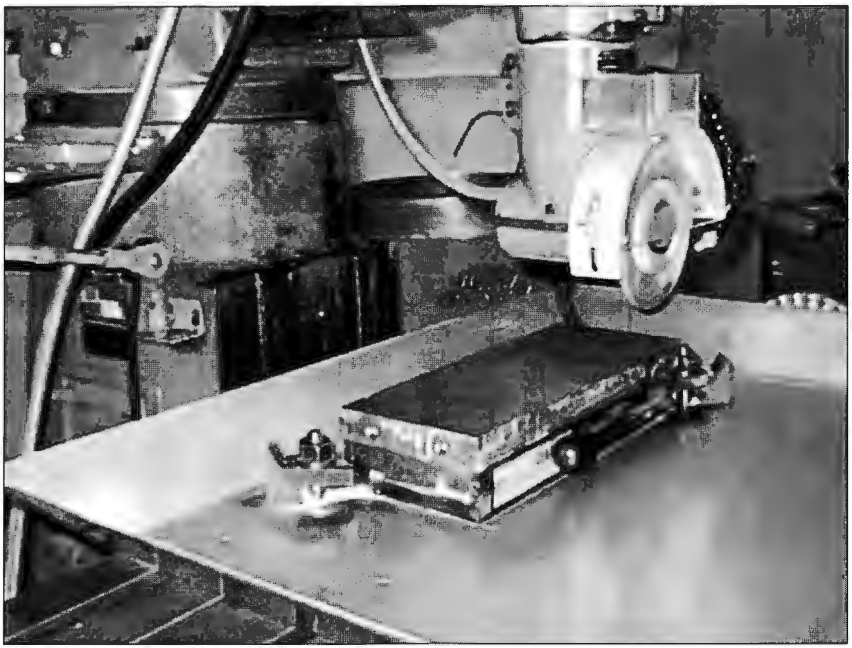
Materials Science and Technology Division
Los Alamos, New Mexico 87544

Front View of Mill

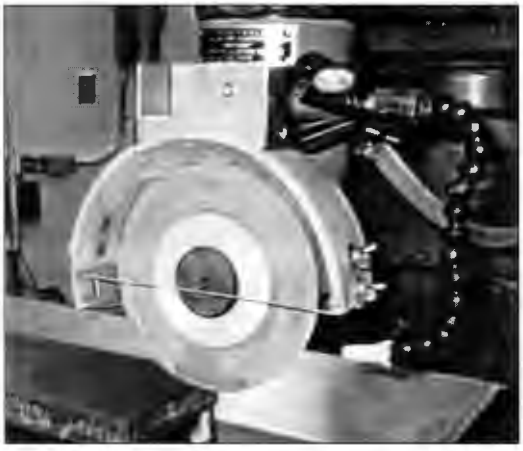
To accomplish high precision cutting not possible with a normal glass cut-off saw, we chose to adapt a common knee mill. Upon first placement of the mill in the glass shop, the machine was carefully leveled.

Right Angle Head Attachment With Diamond Blade



An R-8 Collet Right Angle Head Attachment, available from the manufacturer, was installed to achieve the proper relationship between the blade and the worktable. The head was fitted with a 1" cutter arbor to hold the diamond blade. We chose a 120 grit 6 inch diameter x .050" blade. The blade is run in a clockwise rotation (to avoid "climb grind") to clear the cut of excess material and wax and to pull the coolant into the cut.

Coolant Shielding



The shield for the diamond blade and coolant splash was manufactured by the Los Alamos machine shop. The shroud is made of aluminum with a plexiglass cover plate for visibility. Holes were drilled and tapped in the upper shroud for mounting to the mill head. Also pictured is the "snap-lock" coolant hose system. This attachment directs coolant from the pump to the diamond wheel and has a magnetic base for placement flexibility. Additional skirting made from heavy plastic sheet further contains the coolant "fantail" without interference to the work.

Reservoir

The reservoir is an off-the shelf item from the mill manufacturer. The large design helps catch unexpected drips during cutting. It came with a submersible pump and a pump area isolated from the main tank. Debris settles in the main tank away from the pump.

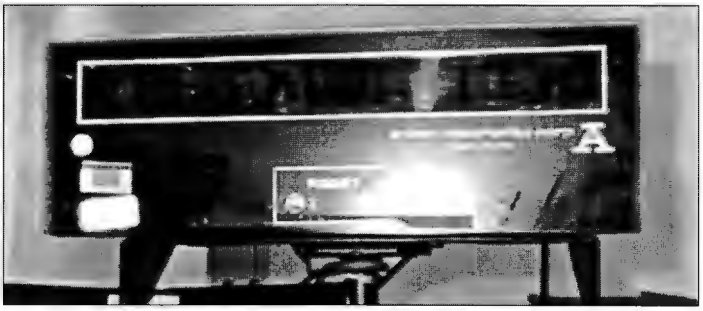
The Pump

The submersible pump is strong enough to deliver a liberal amount of coolant to the work area. The synthetic coolant should be water soluble, rust retardant and bacteria retardant. We placed the pump on an independent switch so the coolant could be turned off at will, thus making visual inspection easier.

Power Feed

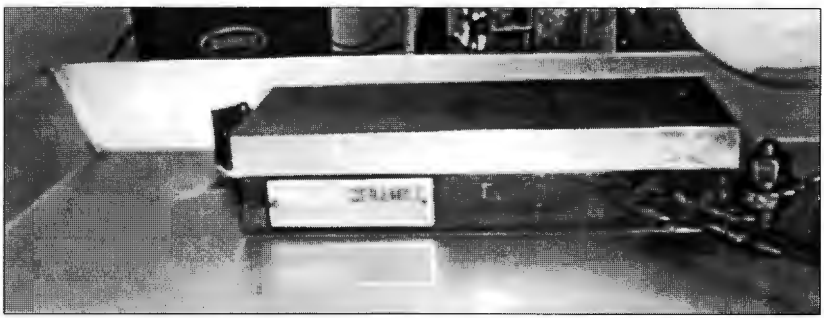
The head crank for the x-axis is fitted with a variable speed motor drive to speed and ease operation. We normally cut a rate of 1" to 2" per minute at a maximum depth of .100" per pass.

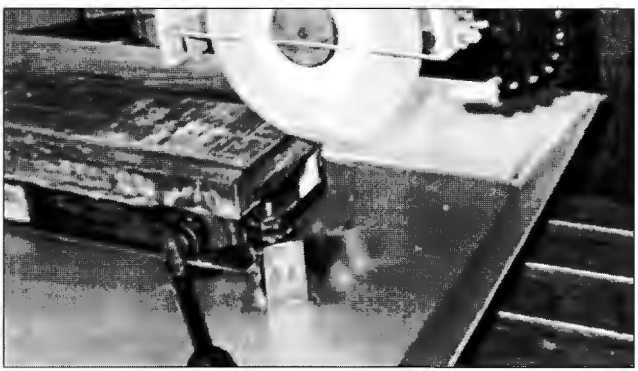
Digital Readout



A standard machine item, the digital readout gives an easily readable display of the X and Y axis to tolerances within .0001".

Magnetic Chuck





The magnetic chuck measures 6" x 12" and is manually operated. the lever in front engages and disengages the magnet. The chuck is aligned to be square with the diamond wheel at installation of the right angle head. The rectangular steel plate has been previously machined to right angles. Glass work is waxed to a 6" x 12" plate of cold rolled steel and aligned to the edge of the steel plate. The plate is aligned to the edge of the magnetic chuck and the magnet is engaged.

Coolant Worktable Tray

We made an over-sized stainless steel tray to catch the coolant spray. The tray is sandwiched between the magnetic chuck and the mill table. The tray is secured with the same bolt used to secure the chuck. Additional holes were drilled in the tray to provide adequate drainage. The worktable is fitted with drains and hose to return the coolant to the lower reservoir.

Since the addition of the precision cutting capability, we have accepted a large number of precision cutting jobs. The capability has also been combined with more complex jobs with beneficial effect to a wide variety of finished products.

Crack-Off Cutting from a Different Angle

by
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Introduction

Crack-off cutting joints on angle, that will be used as side necks on flasks or other apparatus, is a simple task. It will save time and mess over the traditional wet saw cutting and acid etching method.

First construct a fixture.

Cut an outer joint to the desired length and angle on the wet saw. Rinse and dry. This will be the model for making the fixture. In this example I am using a 24/40 joint.

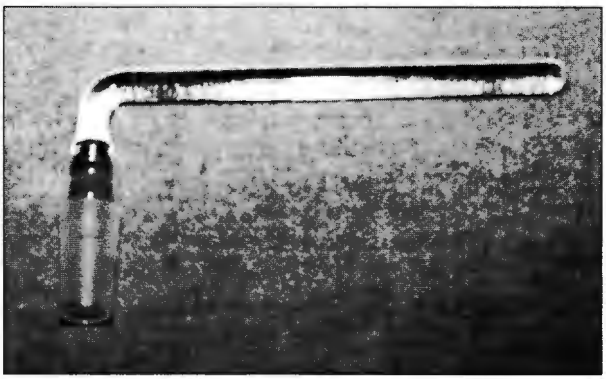

Photo 1.

Seal inner 24/40 joint to a 1/2" od rod 10 inches long. Bend the rod 90 degrees about 2 inches away from the 24/40 joint.

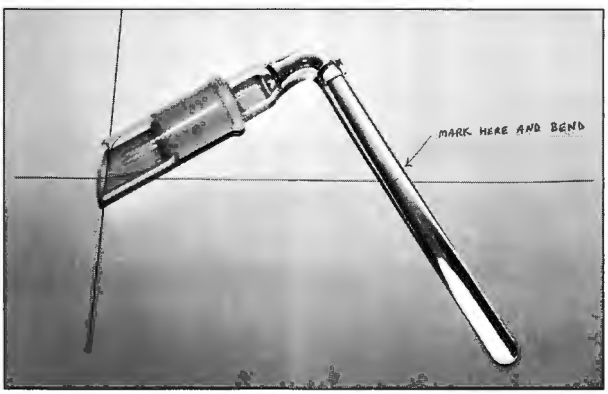

Photo 2.

Slip the angle-cut outer joint on to the 24/40 inner fixture that you have made. Set the center of the cut 24/40 outer on a straight edge and mark the 1/2" rod as shown.

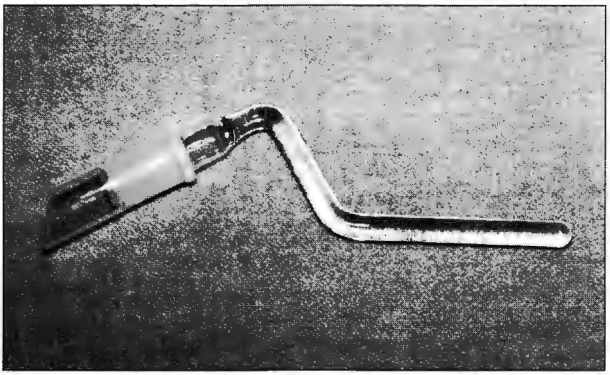

Photo 3.

Bend to make alignment.

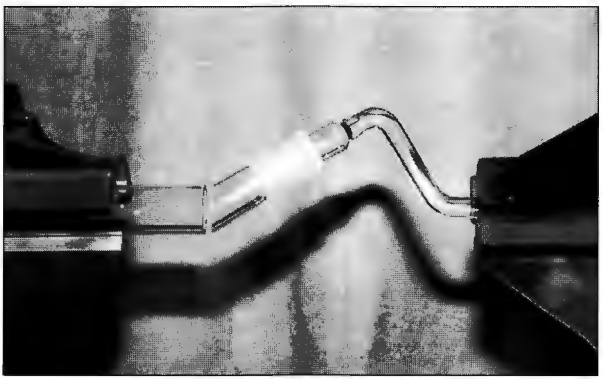

Photo 4.

To get precise alignment, use a lathe. Chuck the 1/2" rod in the tailstock. Chuck a piece of 32mm od tubing in the headstock. Move the tailstock until the cut joint and the 32mm tubing barely touch. Now heat the 1/2" rod on the fixture and make alignment adjustments to the fixture.

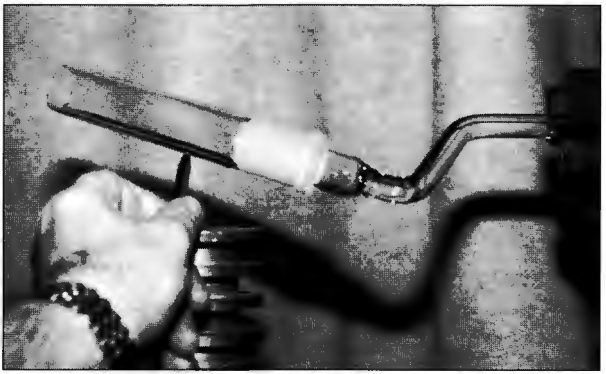

Photo 5.

Slip an outer joint on to the fixture. Use a hand rest under the center of the joint as it rotates in the lathe. I am using three large chuck doughnuts as a handrest. Turn the lathe on at a medium speed, not too slow. Use a sharp diamond pencil to lightly scratch a line while the lathe is turning. It will take a little practice to find the center of the turning glass. You will only be able to scratch the high spots, not all the way around. however, scratching the high spots is sufficient.

Photo 6.

While the lathe is turning, heat the scratch with a small sharp fire for 7 or 8 seconds. Squirt the scratch with water.

Photo 7.

Stop the lathe. Remove the joint from the fixture. Pull to separate the cracked pieces.

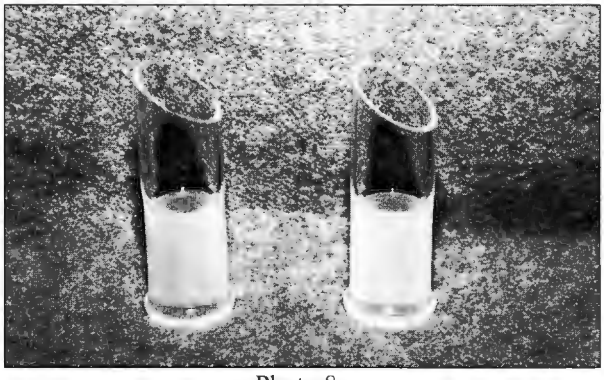

Photo 8.

The joint on the left is cracked off, and the joint on the right is a wet saw cut.

Conclusion

You now have an angle cut joint that you do not have to etch with Hydrofluoric Acid before it is sealed to apparatus. I suggest cracking a group of joints and storing them in plastic bags until needed.

How to Break Glass With Flame

by
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Two things simultaneously required for glass to break: a flaw and strain (in tension)

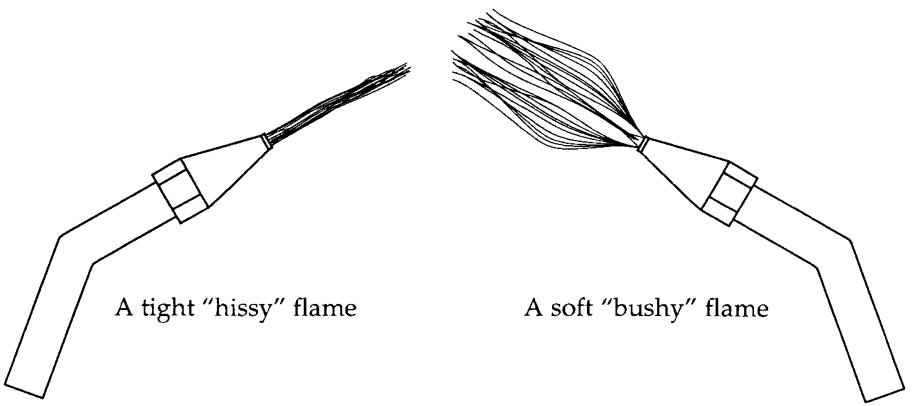
Glassblowers can create a flaw with a glass knife.

The tension (strain) may be created physically, by flexing the glass, or thermally, by applying heat. Typically, thermal strain is created by heating a glass rod in a torch and then touching the heated tip of the rod to the flaw on the glass tube.

This poster provides an alternative approach, applying the flame to the glass tube itself to create failure. This technique is successful on tubing from 4mm to 65mm in diameter. Although the cracks on larger size tubing may drift, this is still an excellent approach to obtain a more convenient shorter tube with which to work.

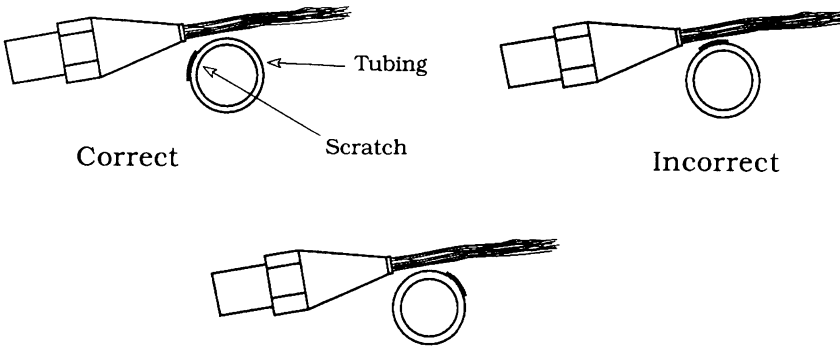
Successful glass cutting with a flame is largely dependent upon two criteria; the shape of the flame, and where the flame touches the glass.

A small tight "hissy" flame is easier to use than a soft "bushy" flame.



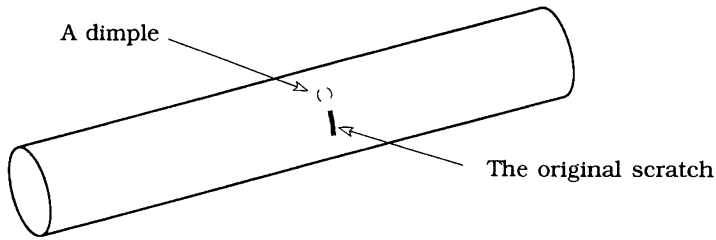
The other element for success is proper placement of the flame against the glass.

Place the flame on the far side of the scratch. Place it near, BUT DO NOT TOUCH, the flame onto the scratch.

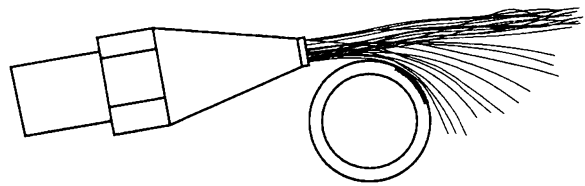


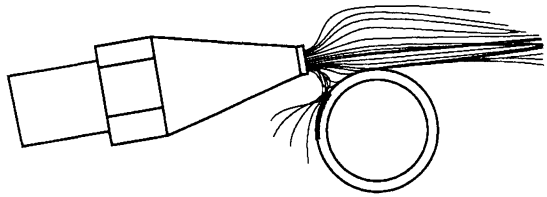
The glass should crack within a second. If you've been waiting longer than a second, or if you start to see a dimple where the flame touched the glass, it will not crack.

In this case, what probably occurred is the flame grazed across the scratch, firepolishing the scratch. Once the scratch is removed from the glass, there is no flaw, and the glass cannot break.



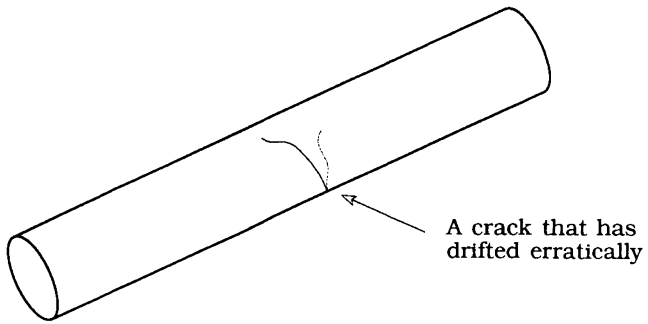
Probable causes for sealing (firepolishing) the flaw are flame/flaw orientation or flame shape.





If it doesn't crack, there are two choices:

1) Create a new scratch a reasonable distance (1-2cm) away from any latent strain and start again.
2a) If it's small tubing, flame anneal the entire area of the scratch, let cool, re-scratch, and re-attempt the crack.
2b) If it's medium or large size tubing, (over 15mm), oven anneal the tube before re-scratching, or the resultant crack will tend to drift erratically.



Tubing Sizes can affect performance:

Very thin wall tubing	• It is very difficult to flame cut on thin wall tubing because it is difficult to develop thermal strain in thin wall tubing.
Standard wall tubing	• Very easy.
Medium wall tubing	• Very easy.
Heavy wall tubing	• It is difficult (but not impossible) to flame cut heavy wall tubing because the time required to develop strain creates a chance for the glass to soften before a crack is initiated.

It is a good idea to....

Firepolish fire-cut tubing immediately.

Having introduced strain to the glass to cause it to break, fire cutting is likely to result in some remaining (thermal) strain in the glass. There is less of a chance for cracks in the tube to occur if firepolishing is done immediately after fire-craking than if done after the piece cools.

Sample Column

by
Gary Dobos
Westinghouse Savannah River Company
Aiken, SC 29808

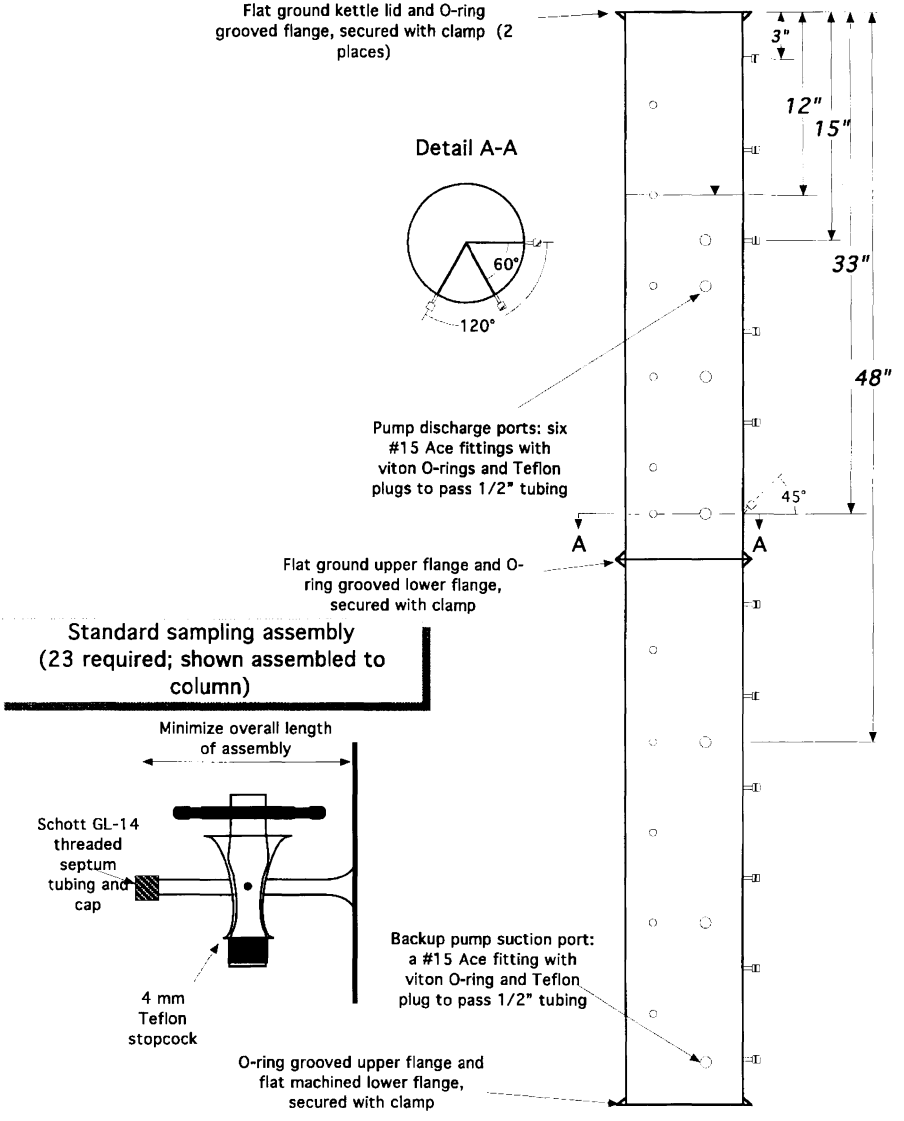
The sample column that is displayed is an apparatus used to study benzene levels suspended in solution. The column enable samples to be taken every three inches. The column also allows the researcher to watch for separations in the solutions. Pump ports were added to feed the column with solutions and to aid in mixing the column. The top and bottom caps were made of stainless steel. In addition to providing support, the caps were also used to connect many instruments, inert gases, thermocouples, heating, and cooling coils to the column.

The columns were made in an estimated time of thirty hours each. All the large seals were made by using a stainless steel six fire swivel Litton Burner. To achieve the working temperature required, we used a hydrogen and oxygen mix. The three-foot sections were sealed in four steps to prevent breakage. The torches used were a combination of national, and multi-mix hand torches. The hand torches were run on propane and an oxygen mix. The columns were filled and tested over a two month period.

The pump ports were #15 Ace-Threds. The Ace-Thred enabled easy connection of the ports with an Ace adapter. The adapter has an Ace-Thred on the outside and a Swagelok® NPT on the inside. This was a very efficient way of connecting all seven ports per column.

A reaction o-ring kettle flange was chosen to seal the three-foot section together. The 45 degree outer taper enabled our machinist to match the metal caps to the glass flanges and make a liquid tight seal. The Duran® quick release clamp was used to firmly hold flanges and lids together

The sample ports were made from a four millimeter Teflon stopcock and a GL #14 glass screwthread. To hold the viton septa in place, we used a GL #14 cap with aperture. The ports were sampled with a ten gauge needle. The Teflon stopcocks allowed the septa to be easily changed without disturbing the sample columns.


Flat ground kettle lid and O-ring grooved flange, secured with clamp (2 places)
Detail A-A
60°
120°
3"
12"
15"
33"
48"
Pump discharge ports: six #15 Ace fittings with viton O-rings and Teflon plugs to pass 1/2" tubing
45°
A
A
Flat ground upper flange and O-ring grooved lower flange, secured with clamp
Standard sampling assembly (23 required; shown assembled to column)
Minimize overall length of assembly
Schott GL-14 threaded septum tubing and cap
4 mm Teflon stopcock
Backup pump suction port: a #15 Ace fitting with viton O-ring and Teflon plug to pass 1/2" tubing
O-ring grooved upper flange and flat machined lower flange, secured with clamp

Vacuum Jacketed Distillation Receiver

by
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Introduction

A distillation receiver comes in many forms. Specifically, I will be referring to Barrett or Dean Stark, where the chemist has specific needs to azeotropically remove water from a reaction, containing a less dense solvent (e.g. toluene). In this demonstration, the side arm is jacketed, because the chemist needs to use a real high boiling solvent (diphenyl ether) for their experiment.

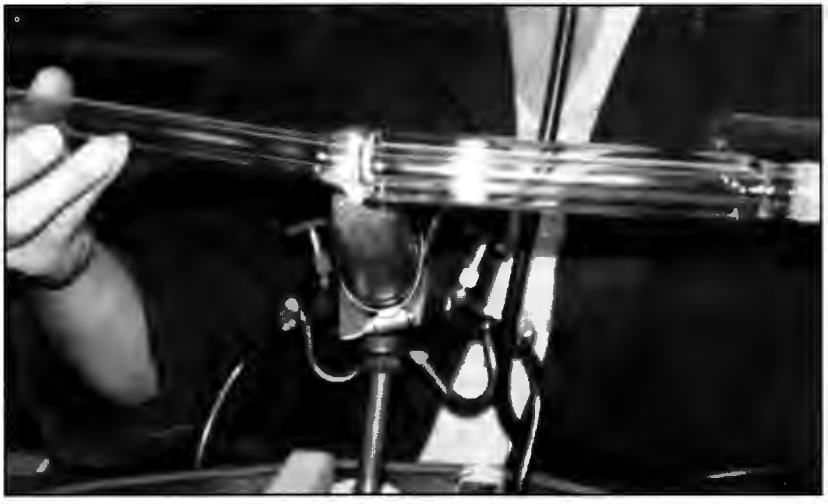
A calibrated reservoir is made from a 10ml calibrated centrifuge tube. To begin, seal on a 2mm bore glass stopcock to the tapered graduated end of the centrifuge tube; try to minimize a change in volume. A 24/40 outer ground glass joint is pulled off two inches below the joint; then seal on the centrifuge tube and blow out a hole on the side (to attach the side arm in later steps of fabrication). Flame anneal and set aside.

Begin the jacketed side arm like a condenser. Prepare a 16mm od tube for the inside by forming a bead at each end of the tube with a total length of six inches. The outside 28mm od tube is shaped into a round bottom, then the 16mm tube is inserted to make the ring seal (in this case the tube is floated into the ring seal).

Once the ring seal is fused, blow out the end and seal on a 24/40 inner drip joint. Try to minimize the distance between the ring seal and the joint to prevent any temperature loss during the distillation.

Cut an 8mm (medium wall) side arm tube long for the vacuum seal off; seal and bend the tube towards the joint. Bend the side arm to lay parallel with the 24/40 drip joint tube. Flame anneal.

Attach two blow hoses to the tubes with straight swivels: one to control the pressure on the inside tube and the other to control the pressure on the outside. Pull down the 28mm tube, finish the ring seal, and seal on a 5/8" medium wall tubing.



Set up a support or harness to easily reach with your mouth to blow into each blow hose separately. Begin heating the tubing with a large bushy fire, slightly puffing up the outside tube.

Once the inside tube begins to collapse, BEND! Control the air pressure by alternately blowing into the outside and inside tubes until the bend is complete.

Pull off the 5/8" medium wall tube about 10mm away from the ring seal. Begin heating the calibrated tube and stopcock (set aside earlier) and seal to double bend piece.

Oven anneal and vacuum test for leaks. The trap is ready to seal on to the vacuum line to evacuate. Evacuate the trap by standard procedure: seal on to vacuum line in oven and slowly bring up to temperature (500 degrees Celcius). Once the trap has soaked about an hour at temperature, check vacuum pressure to verify an excellent vacuum has been reached and seal off.

The completed vacuum jacketed distillation column.

Acknowledgements: Randy Matthews, Eileen Fletcher and Hal Langworthy.

What Do You Mean You Can't Read a Ruler?

by
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Knowing how to read a ruler and other precision measuring tools is one of the most important pieces of knowledge a glassblower can possess. It never ceases to surprise me how many people lack this basic skill. Although this was something that was taught to me twenty-five years ago in math and shop class, I question whether this is still being taught when I'm trying to train somebody today. There are a number of other precision tools that can be used. The tools in this poster are the most commonly used of all precision tools. Although they can be intimidating, these tools are easy to use, and as they are used in virtually every facet of glassblowing, it is important that we familiarize ourselves with their use. The ruler, the vernier caliper and the micrometer are tools that I use every day. I cannot imagine being able to efficiently produce precision glassware and not have a working knowledge of how to use them.

I would like to thank Nancy Hagmaier for helping me prepare this poster. I would also like to thank Technical Glass Products for providing me the resources to make this poster.

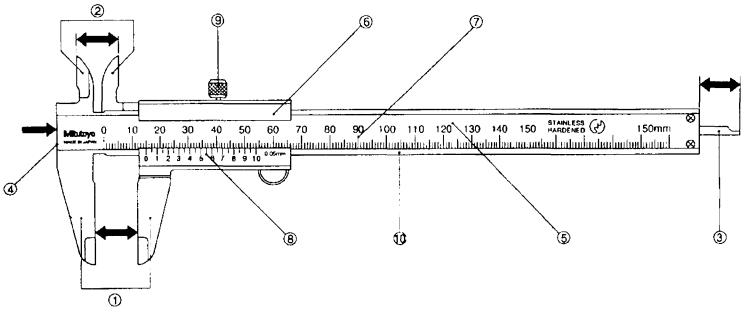
Rulers

Shown below are various types of rules and tapes available today. Of all basic measuring tools, the simplest and most common is the steel rule. This rule is usually 6 or 12 inches in length, although other lengths are available. Steel rules may be flexible, but the thinner the rule, the easier it is to measure accurately, because the division marks are closer to the work.

Generally a rule has four sets of graduations, one on each edge of each side. The longest lines represent the inch marks. On one edge, each inch is divided into 8 equal spaces; so each space represents 1/8 inch. The other edge of this side is divided into sixteenths. The 1/4th inch and 1/2 inch marks are commonly made longer than the small division marks to facilitate counting, but the graduations are not, as a rule, numbered individually, as they are sufficiently far apart to be counted without difficulty. The opposite side is similarly divided into 32 and 64 spaces per inch, and it is common practice to number every fourth division for easier reading.

Another variation is the metric rule. The graduations are broken down into millimeters, or tenths of millimeters. The large graduations are normally in powers of tens.

Vernier Calipers



1)Outside Jaws

2)Inside Jaws

3)Depth Bar

4)Step Surface

5)Main Beam

6)Slider

7)Main Scale

8)Verner Scale

9)Clamp Screw

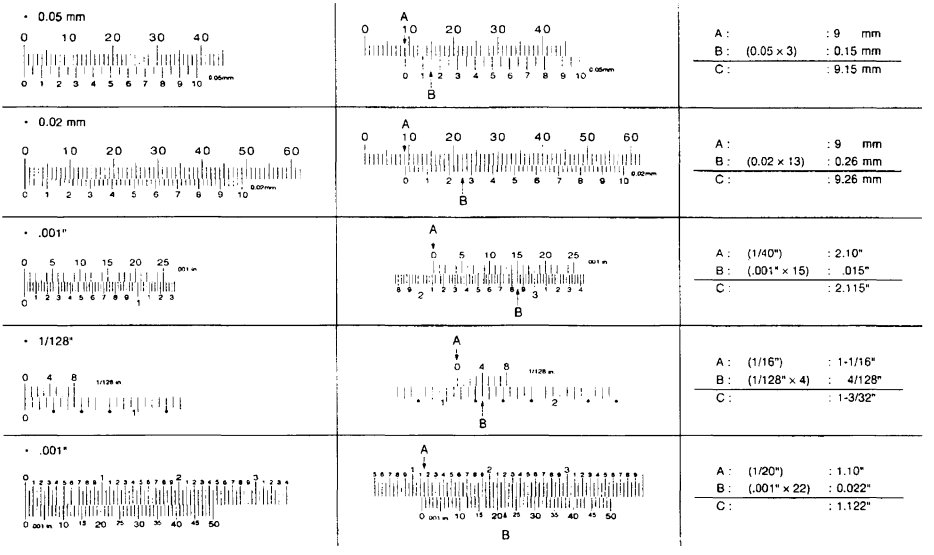
10)Refrence Surface

Metric Scale

A:Main Scale

B:Vernier Scale

C:Reading



Precautions for using vernier caliper

Wipe clean dust dirt from sliding surfaces, and graduated surfaces.

Make sure that zero lines coincide when the jaws are closed and that no slit is observed between the jaws when exposed to light.

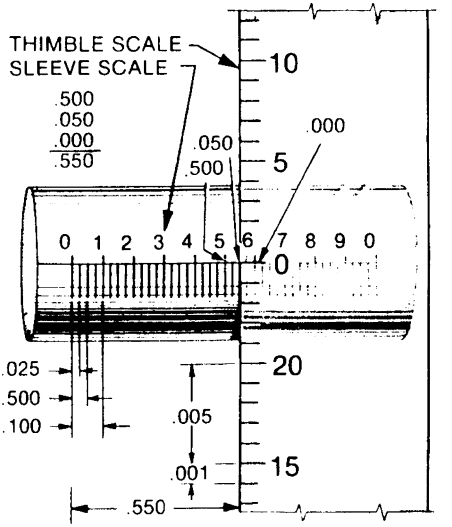
Apply clean oil to the sliding surfaces. Lack of oil may cause scratches on the sliding surfaces, resulting in poor sliding efficiency.

The Micrometer

One of the most important instruments for precision measuring is the micrometer (mike). It is an essential tool for anyone working with machinery or in a machine shop. Micrometers are used to measure distances to the nearest thousandth of an inch or tenths of a millimeter. The measurement is usually expressed or written as a decimal, so to use the micrometer you must understand the decimal system.

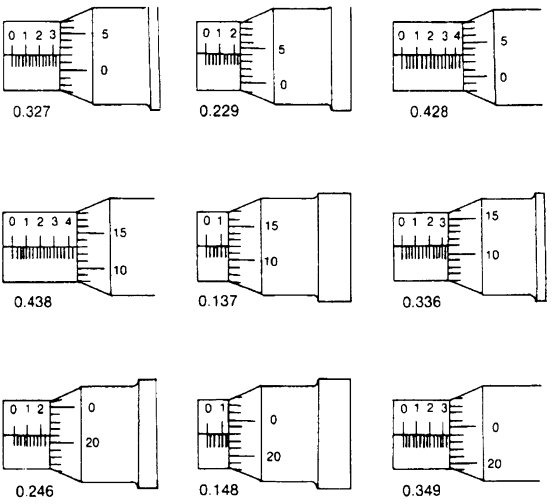
The sleeve and thimble scales of the micrometer caliper are shown enlarged in Figure 3. To understand these scales, you need to know that the threaded section on the spindle, which revolves, has 40 threads per inch. Therefore, every time the thimble completes a revolution, the spindle advances or recedes 1/40 inch (0.025 inch).

Notice that the horizontal line on the sleeve is divided into 40 equal parts per inch. Every fourth graduation is numbered 1,2,3,4, etc., representing 0.100 inch, 0.200 inch, etc.. When you turn the thimble so that its edge is over the first sleeve line past the 0 on the thimble scale, the spindle has opened 0.025 inch. If you turn the spindle to the second mark, it has moved 0.025 plus 0.025 inch, or 0.050 inch. You use the scale on the thimble



to complete your reading when the edge of the thimble stops between graduated lines. The scale is divided into 25 equal parts, each part representing a twenty-fifth of a turn. A twenty-fifth of 0.025 inch is 0.001 inch. As you can see, every fifth line on the thimble scale is marked 5,10, 15, etc. The thimble scale, therefore, permits you to take very accurate readings to the thousandth of an inch, and, since you can estimate between the divisions on the thimble scale, fairly accurate readings to the ten-thousandth of an inch are possible.

Read each of the micrometer settings in Figure 4-6 so that you can be sure of yourself when you begin to use this tool on the job. The correct readings are given following the figure so that you can check yourself.



Care and Maintenance for Precision Tools

From time to time you will find the need to maintain, clean or repair your tools. Here are some simple suggestions as to what you will need to do this and how to do it.

Tools you will need

1. A regular blade (small) & Phillips screwdriver
2. Soft lint-free rag or towel
3. #00 steel wool
4. W-D40 and light oil
5. A small fine file
6. A micrometer wrench
7. A precision gauge block to calibrate your tools
8. A small brass wire brush
9. A bottle of blue LOC TITE
10. A black Sharpie

Repair and Maintenance

1) You will find that with time and use the adjusting screws on your calipers will come loose. You can use your screwdriver to adjust them. Be careful not to over tighten them. When you have them where you want them, apply a small amount of blue LOC-TITE to the screw tops.

2) You can use the W-D40 to clean your tools. Apply liberally and then clean with the #00 steel wool. Wipe dry with the rag. After you have them clean and dry, apply a very small amount of oil to the moving parts or slide.

3) If the numbers become hard to read, you can cover over them with a black Sharpie and them wipe it right off. It will leave the numbers and lines easy to read.

4) After you are done, you can check your tools, especially, your micrometer with your gauge block.

5) No matter how careful you are, you may accidentally drop your calipers on the ground. You can very carefully use the small file to remove nicks or dents.

Conversion Tables

INCH	INCH	MM	INCH	INCH	MM	INCH	INCH	MM	INCH	INCH	MM
1/64	.015625	.3969	17/64	.265625	6.7469	33/64	.515625	13.0969	49/64	.765625	19.4470
1/32	.03125	.7938	9/32	.28125	7.1438	17/32	.53125	13.4938	25/32	.78125	19.8438
3/64	.046875	1.1906	19/64	.296875	7.5407	35/64	.546875	13.8907	51/64	.796875	20.2407
1/16	.0625	1.5875	5/16	.3125	7.9375	9/16	.5625	14.2876	13/16	.8125	20.6376
5/64	.078125	1.9844	21/64	.328125	8.3344	37/64	.578125	14.6844	53/64	.828125	21.0345
3/32	.09375	2.3813	11/32	.34375	8.7313	19/32	.59375	15.0813	27/32	.84375	20.4313
7/64	.109375	2.7781	23/64	.359375	9.1282	39/64	.609375	15.4782	55/64	.859375	21.8282
1/8	.125	3.1750	3/8	.375	9.5250	5/8	.625	15.8751	7/8	.875	22.2251
9/64	.140625	3.5719	25/64	.390625	9.9219	41/64	.640625	16.2719	57/64	.890625	22.6620
5/32	.15625	3.9688	13/32	.40625	10.3188	21/32	.65625	16.6688	29/32	.90625	23.0188
11/64	.171875	4.3656	27/64	.421875	10.7157	43/64	.671875	17.0657	59/64	.921875	23.4157
3/16	.1875	4.7625	7/16	.4375	11.1125	11/16	.6875	17.4626	15/16	.9375	23.8126
13/64	.203125	5.1594	29/64	.453125	11.5094	45/64	.703125	17.8594	61/64	.9 53125	24.2095
7/32	.21875	5.5563	15/32	.46875	11.9063	23/32	.71875	18.2563	31/32	.96875	24.6063
15/64	.234375	5.9531	31/64	.484375	12.3032	47/64	.734375	18.6532	63/64	.984375	25.0032
1/4	.25	6.3500	1/2	.5	12.7001	3/4	.75	19.0501	1	1.00	25.4001

MM = INCHES	MM = INCHES	MM = INCHES	MM = INCHES
1 = 0.0394	26 = 1.0236	51 = 2.0079	76 = 2.9921
2 = 0.0787	27 = 1.0630	52 = 2.0472	77 = 3.0315
3 = 0.1181	28 = 1.1024	53 = 2.0866	78 = 3.0709
4 = 0.1575	29 = 1.1417	54 = 2.1260	79 = 3.1102
5 = 0.1969	30 = 1.1811	55 = 2.1654	80 = 3.1496
6 = 0.2362	31 = 1.2205	56 = 2.2047	81 = 3.1890
7 = 0.2756	32 = 1.2598	57 = 2.2441	82 = 3.2283
8 = 0.3150	33 = 1.2992	58 = 2.2835	83 = 3.2677
9 = 0.3543	34 = 1.3386	59 = 2.3228	84 = 3.3071
10 = 0.3937	35 = 1.3780	60 = 2.3622	85 = 3.3465
11 = 0.4331	36 = 1.4173	61 = 2.4016	86 = 3.3858
12 = 0.4724	37 = 1.4567	62 = 2.4409	87 = 3.4252
13 = 0.5118	38 = 1.4961	63 = 2.4803	88 = 3.4646
14 = 0.5512	39 = 1.5354	64 = 2.5197	89 = 3.5039
15 = 0.5906	40 = 1.5748	65 = 2.5591	90 = 3.5433
16 = 0.6299	41 = 1.6142	66 = 2.5984	91 = 3.5827
17 = 0.6693	42 = 1.6535	67 = 2.6378	92 = 3.6220
18 = 0.7087	43 = 1.6929	68 = 2.6772	93 = 3.6614
19 = 0.7480	44 = 1.7323	69 = 2.7165	94 = 3.7008
20 = 0.7874	45 = 1.7717	70 = 2.7559	95 = 3.7402
21 = 0.8268	46 = 1.8110	71 = 2.7953	96 = 3.7795
22 = 0.8661	47 = 1.8504	72 = 2.8346	97 = 3.8189
23 = 0.9055	48 = 1.8898	73 = 2.8740	98 = 3.8583
24 = 0.9449	49 = 1.9291	74 = 2.9134	99 = 3.8976
25 = 0.9843	50 = 1.9685	75 = 2.9528	100 = 3.9370

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Mike Armijo - *Sonic Mill*
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Roland Trinquart
Dale Tupa
Herman E. Van Bragt
Sandra Van Bragt
Stephen J. Volpe
Emilie M. Waddington
Robert L. Waddington
Kristin M. Wahner
Karl H. Walther
Andrew J. Wargo
Pat Wargo
Bob Webster
Nancy Webster
David Wedsworth
Woody Weed
Joseph West
Mike Wheeler
Ed Whitehouse
Amy Wicker
Mark Wicker
Klaus Widmann
Sieglinde Wickmann
Randolph B. Wilkin
Amy Wilkinson
Jim Wilson
Christine S. Wilt
Daniel J. Wilt
William J. Wilt
Meghan Wingate
David Wise
Eva Wood
Keith Wood
Frank Wright
M.D. "Shorty" Yeaman
Heather Young
Jody Young
Lois Zavoda
Oliver T. Zavoda
Garson Zawadzki