

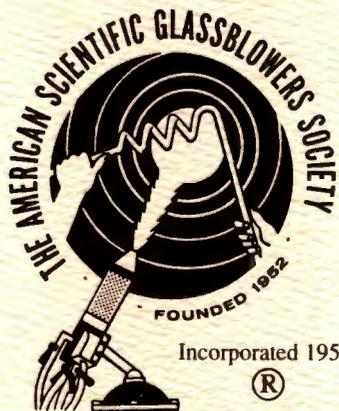
# *PROCEEDINGS*

THE FORTY-THIRD ANNUAL  
SYMPOSIUM

ON THE

**ART OF SCIENTIFIC  
GLASSBLOWING**

**1998**



Incorporated 1954



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# Papers

# **Altering Apparatus to Prevent Breakage and/or Insure Easier Repair or “No Strain, No Pain”**

by

**Gary S. Coyne**

**Chemistry Department Glass Shop**

**California State University, Los Angeles, CA 90032**

In a perfect world, everything we make for everyone would be treated with the same respect and dignity accorded the finest of treasures. The reality is items that we make get broken. It is unfortunate that this happens to the glassware, but fortunately we can usually repair the item. Although it is extra business, not all of us want to repair glassware contaminated with mercury, biological hazards, or even silicon grease.

It is to our advantage to make glassware as robust as possible. This is not only for our health, but the glass shop that sells apparatus that seems to survive the rigors of the laboratory better than another glass shop will likely do better business. There are essentially three ways to make glassware more robust:

1. Make the apparatus stronger. Anything that is less likely to break is stronger by definition.
2. Move vulnerable components of an apparatus to a safer and easier location to repair.
3. Change the design so strength is not required.

## **How Glass Breaks**

To fully appreciate how to make glassware stronger, let us first consider how glass breaks. Most of you are aware of the two requirements for glass failure: flaw and strain. A flaw is a scratch caused by any material that is as hard, or harder than glass. For example, the incidental dirt on a table can create flaws on glassware that is slid across the surface. Likewise, flaws can be caused by glass scratching against glass or by aggressive digging with a metal spatula. For all intents and purposes, it is essentially impossible to prevent glassware from being scratched.

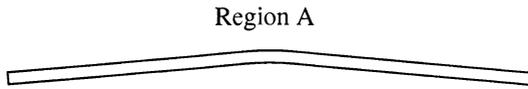
Strain, on the other hand, is something we can control. Glass is susceptible to two types of strain: physical and thermal. Because we have some ability to control physical and thermal strain, we can, if we know how the apparatus is to be used, build strength into the apparatus by design.

## **Physical Strain**

We can make glass physically strong by preventing it from flexing. If the glass cannot flex, there can be no physical strain. If there is no strain, any flaws are irrelevant, and the glass is not going to break.

There are two ways to prevent glass from flexing:

1. Make the glass thicker. Glass that is thicker is more difficult to bend. If it cannot bend then it cannot be flexed. If it is not being flexed, there is no strain. (See Fig. #1)



Thinner glass is easier to flex creating strain at "Region A."

Thicker glass has no strain because there is no flexing

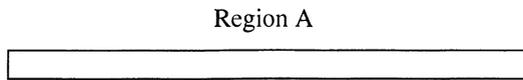


Figure 1

2. Reduce the lever arm. The longer the lever arm, the more strain created at the base of the lever arm. (See Fig. #2)

The longer the lever arm, the easier it is to create strain.

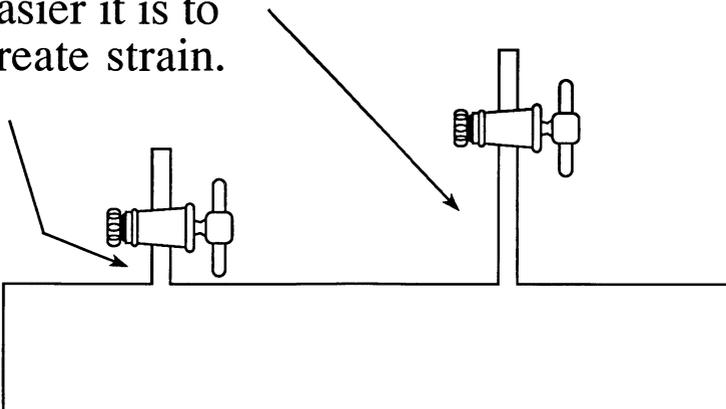


Figure 2

The most common device used to increase the strength of glassware is the support rod, as seen on the short path distillation apparatus in Fig. 3. Without the support rod, the lever

### A support rod for strength

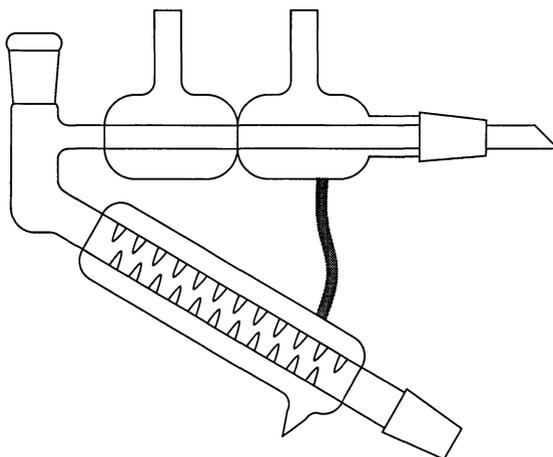


Figure 3

arm's length on the joining tube is sufficiently great so as to make the failure of this piece of glassware in the average lab almost guaranteed. By simply placing a support rod and reducing the length of the lever arm, if breakage does occur, it is caused more by incompetence than by a simple accident.

Another lever arm is the rubber tubing connected to a hose connection. It is all too common for hose connections to be broken off as the user attempts to remove the tubing by pulling or yanking. I have found no way to prevent hose connections from breaking, but I have been very successful in limiting the amount of breakage by supplying a razor blade with every item that has a hose connection. Breakage has not been reduced by

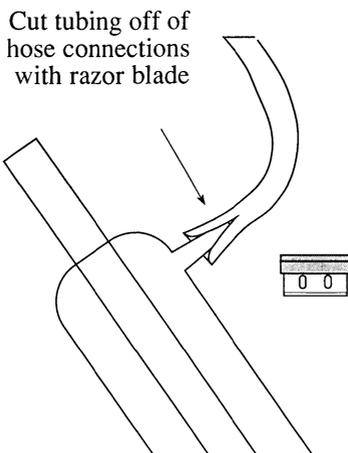


Figure 4

instilling fear into students to cut their wrists if breakage occurs, but rather by explaining that by cutting off the tubing, they will be saving many dollars and lost time (See Fig #4).

Occasionally there are apparatus with a known vulnerable area. One example is when a small stopcock is attached to a large apparatus—especially if it is attached near a ring seal (see Fig. #5). The very nature of a piece like this says that it is likely to be knocked

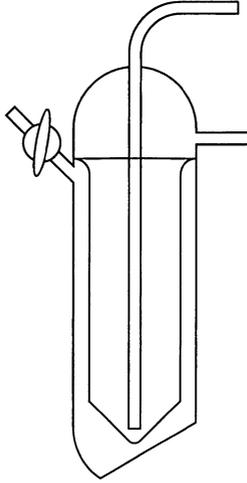


Figure 5

off. What's worse is that repair of an item like this can be tricky because one must carefully pre-heat the area to prevent the ring-seal from breaking. One way to circumvent this problem is to move the location of the weak spot to an area that is easier to repair. By sealing the narrow tubing of the stopcock to either 1/2 or 3/4 inch MW tubing and sealing that to the same area, there is a relocation of the weak spot to an area that is now easy to repair (see Fig. #6). If the customer even notices the adaptation, there is never

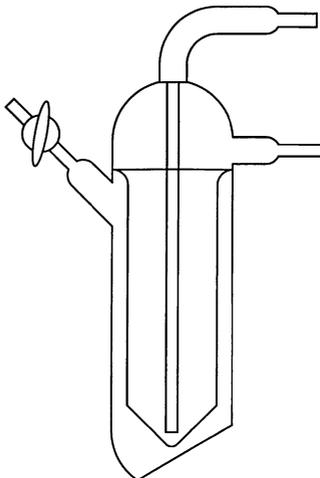


Figure 6

a complaint when the reason is provided. Depending on design, this approach can be extended to some hose connections, and is especially advantageous to cold fingers.

### Thermal Strain

Thermal strain is developed by the heating and/or cooling of glass. Like all materials (except water), glass expands with increasing heat. If an entire apparatus is heated uniformly, there is no problem. However, if glass is heated non-uniformly coupled with extremely radical changes in temperature, failure can occur. Even borosilicate glass, of a design not meant for heating, can break from the relatively low heat of a Bunsen burner. There are two ways to reduce or prevent strain by temperature:

1. Thin glass is thermally stronger than thick glass. Because glass is a poor conductor of heat, the heat can transfer across thin glass sufficiently fast so as to not develop strain.
2. Some glass shapes are more tolerant of heating than other shapes. A round bottom flask can disperse the expansion of the heated bottom. The beaker, on the other hand, can be vulnerable if the bottom is rapidly heated and expands faster than the sides (see the top of Fig #7). When a beaker is heated over a Bunsen burner without the aid of a wire screen to disperse the heat, it is very likely to break.

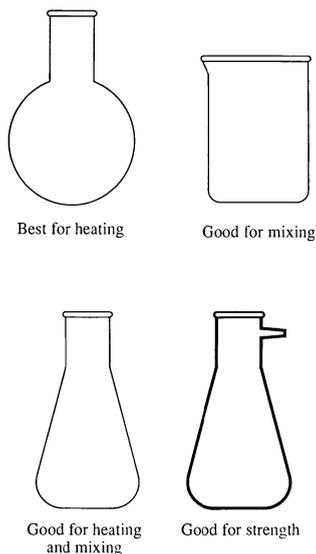


Figure 7

Manufacturers already utilize this knowledge in such apparatus as the Erlenmeyer flask and the filter flask (see the bottom of Fig. #7). The Erlenmeyer flask is thin-walled and the junction between the bottom and the sides is fairly rounded. Both of these features are conducive to use in heating. The filter flask, on the other hand, is thick-walled to withstand the forces of the atmosphere when used in a vacuum condition. The Erlenmeyer flask is very likely to implode if placed in a vacuum condition, and a filter flask is very likely to break if subjected to any heating. These two items are a classic example of “form follows function.”

I recently received a pear-shaped Schlenk flask to repair the lower third of which was severely crazed. It was obvious that the flask had been heated very hot and then placed in some very cold liquid. I talked to the professor to ask him if this was going to be a new commonly-executed procedure, and if so, I would make a special set of Schlenk flasks with a thinner wall on the base to prevent future surface crazing. Apparently a student had heated a solution, and, with the hoped result of fast precipitation, tried cooling it rapidly—in other words, poor chemistry. The professor requested that the flasks continue to be made with the normal heavy wall to protect against physical abuse as the thermal abuse was not likely to happen again.

To eliminate or reduce failure caused by thermal strain, the glassblower needs to discuss with the customer how the apparatus is going to be used. Questions to be asked can include:

1. If the apparatus includes medium or heavy walled tubing, will it be near a great heat source, or was the tubing thickness selected for its physical strength.
2. If the apparatus requires a flat bottom (like a beaker), ask if it will be heated, and if so, ask if it will be heated by a direct heat source. A flat bottom can be used if a heat diffuser (such as a wire screen) is used, and/or the walls are adapted to better deal with changes in temperature (such as an Erlenmeyer flask). Keep in mind that flat surfaces are not safe with any type of vacuum.

### Side-stepping Stressful Conditions

Finally, it is sometimes possible to prevent glass failure by side-stepping stressful conditions in the first place. It is all too common for glassware to be made a certain way because that is the way it has always been made. By thinking creatively, it is sometimes possible to make the same item, accomplish the same purpose, with essentially the same design, but with some minor alteration that eliminates an inherently bad feature.

Typically, all one needs to do is observe how the glassware is broken to get a reasonably good idea of what caused the damage. For example, chromatography columns often have either longitudinal cracks running the length of the column, or the tip is smashed off. When used, chromatography columns are supported by a two- or three-finger clamp squeezed against the column (see Fig. 8). It was obvious, in this case, that the columns had either been squeezed too forcefully, or held insufficiently. A true “damned if you do and damned if you don’t” problem for the user.

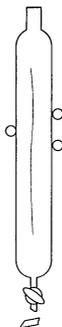


Figure 8

The resolution for this problem was a minor alteration of the design to add a small maria at the very top of the column (see Fig 9). One may need to point out to the user that there is no need to crank down on the clamp with excessive force; rather, simply place the two- or three- fingered clamp, sufficiently high so as to stop the column from slipping down at the maria. This approach eliminates the cause of the longitudinal cracks and prevents

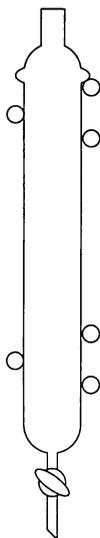


Figure 9

the column from slipping, which caused the tip to break. With this setup, the user may need to add a second clamp to the lower half of the column to prevent it from swinging.

The larger the size of a standard taper joint, the greater the surface of the ground area. While this does help insure a great vacuum seal, it also makes separation of large joints very difficult (see Fig. 10). While, in general, this is a nuisance, on a filled cold trap from a vacuum system in mid-experiment, it can be a full-fledged nightmare. It is not acceptable to allow a trap not to have a removable bottom by design or neglect. Likewise, it is unsafe risking a broken vacuum line due to a pair of hands that are too strong. An

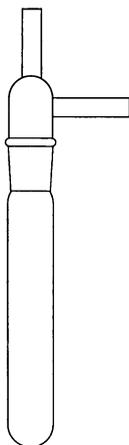


Figure 10

alternate solution is to side-step the problem by simply using O-ring joints in lieu of standard taper joints (see Fig. 11). With the O-ring joint, one needs only shut off the trap from the pump and the rest of the vacuum system, vent the trap, and after removing the O-ring clamp, the two members easily separate.

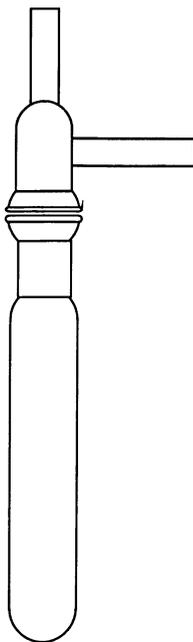


Figure 11

## Conclusion

By understanding how glass breaks and being willing to make changes on standard designs, it is possible to reduce the amount of glass breakage in labs. Techniques to increase the strength of glassware include

- increasing the thickness of the glass
- adding support rods
- decreasing the length of lever arms and/or moving the lever arm to a better location
- customizing the glassware for either thermal or physical strength
- or by-passing the problem by altering the original design so that the original problem is no longer an issue.

Potentially everything that is made can be enhanced by using these techniques, and very likely most glass shops have made many of these alterations without consciously analyzing the reasons for the changes made. Hopefully this paper provides some formal direction on future apparatus alterations. One caution: do not make any alterations without first discussing these changes (and their reasons) with the professor and/or researcher in charge. As in the Schlenk flask mentioned earlier, the chemistry being performed was wrong and this is what needed to be changed, not the glassware design. Alternatively, it's possible that proposed changes may deleteriously affect other operations.

If the proposed changes are accepted and successful, not only will the glassware be more robust, perform its intended operation, AND survive in the laboratory, but the glass shop will be appreciated as a vital operation that is an integral part of the design process.

# Considerations for the Construction and Assembly of an Auroral Simulator

by

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## Abstract

A device to simulate the Aurora or Northern Lights can be made by the placement of a glass sphere containing a magnet (a “Terrella” or “Little Earth”) inside a partially evacuated glass vessel. Electron guns are used to irradiate the Terrella and under specific conditions of magnetic excitation, localized discharges around the magnetic poles of the Terrella are produced. The paper outlines some considerations for the fabrication of such a system.

## Introduction

At the turn of the 20th century, a Norwegian scientist made a device that simulated the Northern Lights. It was made at a time when cathode rays were known to exist but they were only beginning to understand that these rays were made of tiny negatively-charged particles later to be known as “electrons”. It was also a time when it was generally accepted that “something” was bombarding the earth causing magnetic storms and polar aurora, but what that “something” was and where it came from was not exactly clear. In 1896, Kristian Birkeland proposed that a beam of cathode rays emitted by the sun caused auroras and that those rays reaching the vicinity of the earth would be profoundly affected by the earth’s magnetic field and guided to the high latitude regions to create the aurora.<sup>1</sup> By 1910, after three expeditions to the Arctic, Birkeland built a model of the Aurora. In a large continuously-pumping vacuum chamber made from thick glass plate, he placed a cathode ray source, representing the sun, and aimed it at a magnetized sphere, representing the earth. He ran his discharge at thousands of volts and high currents and at a pressure of about  $10^{-3}$  Torr. His results were remarkable and are consistent to a large extent with modern-day understanding of the Northern Lights.

Birkeland called his sphere, representing the earth, a “Terrella”. This was perhaps in tribute to William Gilbert, personal physician to Queen Elizabeth I and part-time physical scientist. In 1600, Gilbert introduced a magnetic model of the earth in a treatise called *De Magnete* explaining the magnetic nature of the earth. It was an important publication that launched the study of geomagnetism. In Latin, Gilbert described the interaction of a spherical magnet and iron spikes placed upon it. The spherical magnet was used as a model of the earth and so he called it a “Terrella” or “little earth”.<sup>2</sup>

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<sup>1</sup> Robert H. Eather, *Majestic Lights* (Washington, D.C.: American Geophysical Union, 1980) 132.

<sup>2</sup> Sydney Chapman and Julius Bartels, *Geomagnetism Volume II* (Oxford: The Clarendon Press, 1940) 915.

One day last year, an internationally-known multimedia artist paid a visit to my glassblowing facilities. She was the “artist-in-residence” at the National Gallery of Canada. Her recent works are said to have been inspired by what she calls “primitive” technologies from the early days of electricity, electromagnetism and optical components. She was now working on an exhibit called “Charged Hearts” and wanted to re-create Birkeland’s Auroral Simulator. To that end, she needed some glassblowing done. This is how I met artist Catherine Richards.

She began by explaining that the “Charged Hearts” exhibit she envisioned would require the viewer to pick up one of two bell jars containing an anatomically-correct glass heart filled with neon. As the heart was picked up, it would begin to “pulse” a neon discharge, which, in turn, would induce a centrepiece display of the Northern Lights. After the Northern Lights display completed its cycle, the viewer would take a ticket from a printer with a number that would let them interact further with the exhibit when they get home and onto the Internet.

The message Ms. Richards sought to deliver is complex. It is supposed to demonstrate how new technologies are changing the time and space dimensions of human interaction. With today’s wireless technologies, one can easily get the sense, Ms. Richards explained, that we are becoming more and more “unplugged”. But, as wireless technologies require us to set up electromagnetic environments all around ourselves, we are virtually becoming more and more “plugged in”. So “Charged Hearts” brings to the surface what it means to interface with technology. The heart is affected by electromagnetic radiation. The Northern Lights are an electromagnetic phenomenon. Using the analogy of a computer, when we interface with this exhibit, the heart is the mouse, the Northern Lights display is the monitor.<sup>3</sup>

There are many parts to the exhibit and teams of people who contributed to it. This paper focuses only on the display of the Northern Lights and the device that made the display possible, the Auroral Simulator.

Making a modern-day model using the same concepts as Birkeland did almost ninety years ago captured the interest of the artist. An exact duplication would not be safe for public viewing nor would it be portable. Furthermore, the high current and voltage requirements (30,000 Volts) and continuous chamber pumping of that model would not suit the “Charged Hearts” exhibit well. It needed to be as maintenance-free as possible, able to travel with the exhibit and operate at much lower voltages and currents than the original. With input from the artist, the glassblower (me) and a physicist (Dr. Peter Sewell), an Auroral Simulator with a Terrella inside was designed to meet these requirements.

---

<sup>3</sup> Conversations with the Artist, Catherine Richards.

Birkeland's model, like the real thing, works in the following way.<sup>4</sup> Electrons coming from an electron source such as the sun or a cathode ray gun hesitate to cross magnetic

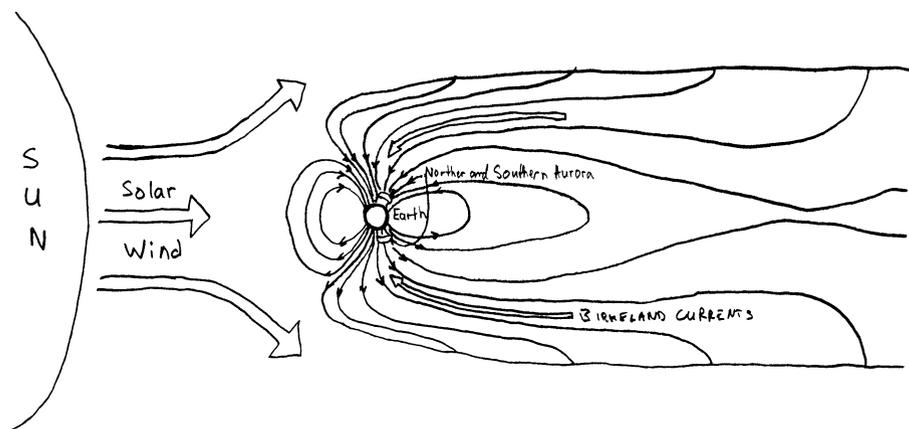


Figure 1: Simplified Representation of Sun-Earth Interaction Causing Aurora

field lines *perpendicularly*. Instead they are deflected away. As a result, a magnetic “bottle” forms around the earth known as the magnetosphere. The force of the Solar Wind sweeps the field lines so that the magnetosphere takes on the shape of a comet. Somehow, and there is not current agreement on how this happens, some electrons *do* enter the restricted area way out in the tail of the magnetosphere. Electrons travel well *along* the lines of a magnetic field and end up concentrating around the poles of the magnetic source such as the earth. These electrons then bombard the particles they come in contact with. Atoms and molecules in our ionosphere are bombarded by these electrons, thereby triggering the Northern Lights.

## Materials and Methods

We all agreed that the overall shape of the vacuum chamber should be a sphere, for display purposes and for safety. Within that spherical chamber, a smaller sphere representing the earth, our Terrella, was to be somehow suspended. At some location away from the inner sphere, the energy from the sun was to be represented in the form of two electron guns. Magnetic field lines around the earth were also needed as well as computer controls to cycle through the display.

After considering various sizes of round-bottomed flasks and the limitations of the glassblowing equipment available, we decided upon a 12-liter flask with a 70mm Terrella.

---

<sup>4</sup>See figure 1.

# 1.The Terrella

In representing the earth, the inner sphere not only had to be a sphere, it also had to simulate the magnetic field lines generated by the earth. Birkeland had used an electromagnet. We originally were going to use a metal sphere but had some difficulties with it. We then turned to a glass sphere with an alnico magnet embedded in it at about 20 degrees off axis, consistent with the geomagnetic axis of the earth. The problem we faced was that most of these bar magnets have a low Curie temperature, above which they lose their magnetism. Since we were planning on getting things hot in the fabrication process, we opted for a simple iron bar which generates no field lines by itself. The field lines would instead be generated by Helmholtz coils, which will be explained later.

Within the 70mm glass sphere, a 13mm tube, sealed in place with a ring seal at either end, was used to secure the iron core.<sup>5</sup> It had a tiny hole for pressure equalization and two

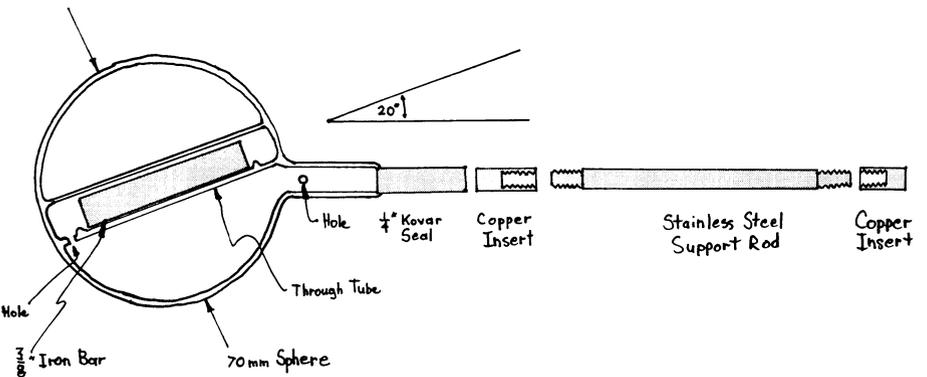


Figure 2: Terrella

dimples for holding the iron in place. At 20 degrees to the inner tube, a 1/4 inch Kovar seal was attached to the sphere. It also had a tiny hole for pressure equalization. A close-fitting copper plug, threaded on its inside, slipped into this Kovar seal and was silver soldered into it using a silver bearing solder type alloy. A supporting rod could then be screwed into this and its connection silver soldered as well. We started with an 1/8th-inch stainless steel rod but later replaced it with a 3/16th piece. Soldering of the parts was done with Eutectic Rod 157 and 157B, silver bearing solder type alloy for joining stainless steels and dissimilar metals. The active temperature range for this solder is 177C to 317C.

Once the inner sphere was made, it was coated with several coats of spray-on graphite, Aerodag from G. Acheson Colloids Canada Ltd., Brantford, Ontario. This gave the sphere a contrasting black matte finish, and, more importantly, made the surface of the sphere conductive with electric continuity between the glass sphere, the Kovar seal and

<sup>5</sup> See Figure 2.

the metal support rod. On the last coating of Aerodag, while it was still wet, the sphere was held in a two-liter beaker containing phosphors (Phosphor 1154). The powdery phosphors were blown around by spraying air from an air can into the bottom of the beaker. The phosphors adhered to the wet graphite to give a light coating of phosphors to the sphere. The phosphors glow when bombarded by electrons the same way as atoms and molecules in our ionosphere glow when bombarded by electrons (originally) from the sun.

## 2. The Electron Guns

Two low-energy electron guns,<sup>6</sup> used as a representation of the sun, were supplied and made by the physicist (Lab 6 Woodlawn, Ontario). They were made of platinum with a

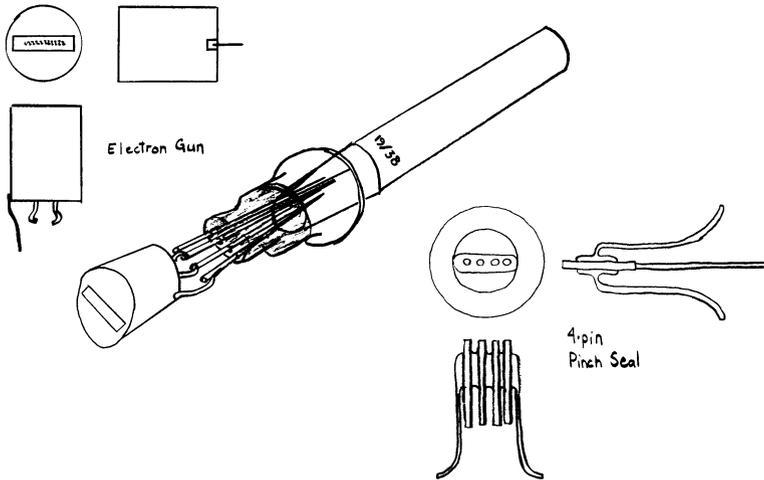


Figure 3: Electron Gun Assembly

special robust filament, made from platinum and barium strontium oxide, able to withstand higher discharge pressures than many other guns ( $10^{-2}$  compared to  $10^{-4}$  or  $10^{-5}$ ). There were leads going to each end of the filament as well as one to the main body of the gun. They measured 20mm in diameter and were about 35mm long.

These guns were mounted on 4-pin pinch seals by spot welding. The pinch seal was a canary glass (Corning 3320) to tungsten seal graded to borosilicate (Corning 7740) with four feed-throughs, although only three were used. They were available through NRC; their original source is unknown. The guns were mounted in such a way so as to have the slit in the same plane as the feed-throughs. The pinch seal/electron gun assembly was temporarily fitted with a holder made of a 19/38 male standard taper.

The electron guns had the potential to operate in two different modes: the Electron Beam Mode and the Gas Discharge Mode. In the original design, it was intended that the system operate with the Electron Beam Mode generating low energy electron beams from each of the electron guns. The electrons were to be accelerated by the gun structure held at a potential of some 100 to 500 volts negative with respect to the Terrella. A

<sup>6</sup> See Figure 3.

positive potential was then to be applied to the Terrella and the electron acceleration voltage increased from 500 to 1000 volts causing plasma to fill the region around the Terrella and ending up exciting the phosphors on the surface of the Terrella. The problem with this mode is the potential for run-away electron voltage. That is, some of the ions produced by the guns would end up hitting the guns themselves, affecting their performance and causing them to increase output to higher and higher voltages. While circuitry exists to stabilize this effect, it was not included in this simulator. Instead, the guns were operated in the Gas Discharge Mode.

In the Gas Discharge Mode, effects are produced similar to those of the Electron Beam Mode, at least as far as a representation of the aurora is concerned. In this mode, the filaments of the electron guns are not used. The entire gun assemblies are used as negative electrodes and the Terrella as a positive electrode. As the potential of the negative electrodes increases to 400 or 450 volts, a gas discharge initiates in the region of the guns by an intense electric field. As the potential difference between the guns and the Terrella is increased to 1000 volts, the plasma extends towards the region of the Terrella in a broader more diffuse and less intense effect than the Electron Beam Mode would have provided, but without the possibility of run-away.

### 3. The Outer sphere

A 12-litre flask was used as the chamber containing the display.<sup>7</sup> Two 100mm open-ended sidearms were added to the flask as well as a Kovar seal slightly recessed into the flask and a 10mm pumping port with a restriction close to the main body of the flask. The

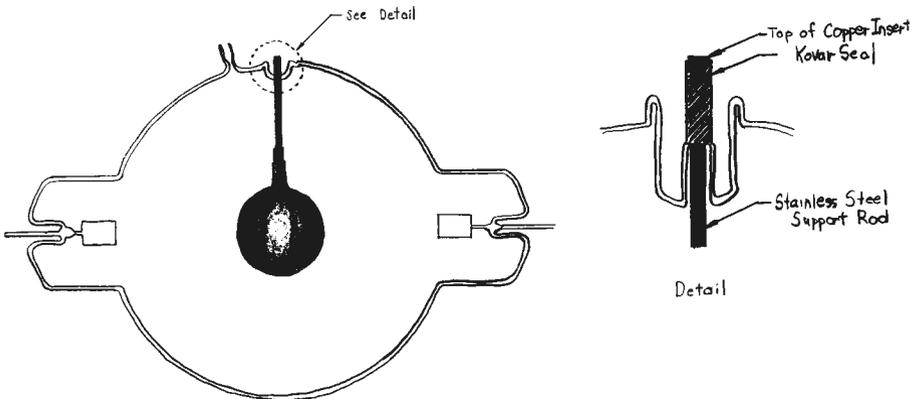


Figure 4: 12-Liter Outer Sphere with Terrella Suspended and Electron Guns in Place

Kovar seal was originally a 1/4" diameter but was later changed to a 3/8" as the size of the support rod and insert changed. At this stage of the assembly, the flask was oven annealed as it was the last opportunity in the process to do so. Once annealed, the 100mm

<sup>7</sup> See Figure 4.

sidearm on the side of the flask with the pumping port was domed off and an electron gun installed. Care was taken to ensure that the orientation of the gun was such that the slit in the gun was in a plane perpendicular to the eventual axis of the supporting rod. In other words, the gun was installed with the slit in the horizontal plane.

It was at this point that the suspension of the Terrella was done. This was a matter of carefully positioning the Terrella (without brushing off any phosphor powders) by placing it in through the open 100mm sidearm and up into the Kovar seal on the flask. The support rod was poked out of the top and the copper insert was threaded on. The Terrella was then lowered back down into the flask and soldered to the Kovar seal. The remaining 100mm sidearm was then domed off and an electron gun installed in the same orientation as the opposing gun. All annealing and preheating was done by flame. In order to eliminate the risk of sooty carbon deposits on the inside of the flask from a natural gas/oxygen flame, the gun insertions and flame annealing was done on this side with a hydrogen/oxygen flame.

#### **4. The Pump-Out**

The procedure of evacuating, backfilling, baking out and pressure adjustment was done many times with a variety of conditions, partly to get things just right and partly because of the frequent modifications to the simulator. The length of time for diffusion pump pump-out was anywhere from a few hours to a few days. The various backfilling and pressure adjustment gases were neon, neon/krypton mix, nitrogen and water vapour. Some heating was done on some trials for degassing. On other trials, after pump-out and backfill, the guns were turned on for further degassing.

Before seal off in each case, the Kovar seal attachment to the Terrella was connected to the positive end of a power supply. The electron guns were connected to each other and to one end of a 57-kilo ohm resistor. The other end of the resistor was connected to the negative terminal of the power supply. A combination of fine voltage adjustments and backfill / pump-out pressure adjustments balanced the striking voltage, the voltage at which a visual display of discharge was observed. Once this was achieved in cases where degassing was done by running the guns, the voltage was increased so that 1 or 2 milliamps of current were going to the guns. This was measured as a voltage drop across the resistor (57 Kilo ohms) of 60-120 Volts. After this, the guns were turned off and the pressure / striking voltage balance was again done. Seal off pressures were done at approximately  $10^{-2}$  Torr.

#### **5. Helmholtz Coils**

As mentioned earlier in this paper, the iron bar in the Terrella does not produce any magnetic field line itself. Instead, the placement of the two Helmholtz Coils provides the magnetic field necessary for the Simulator to function.<sup>8</sup> The coils are nonmagnetic rings wrapped with several turns of copper wire and then attached to a power supply. The

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<sup>8</sup> See Figure 5.

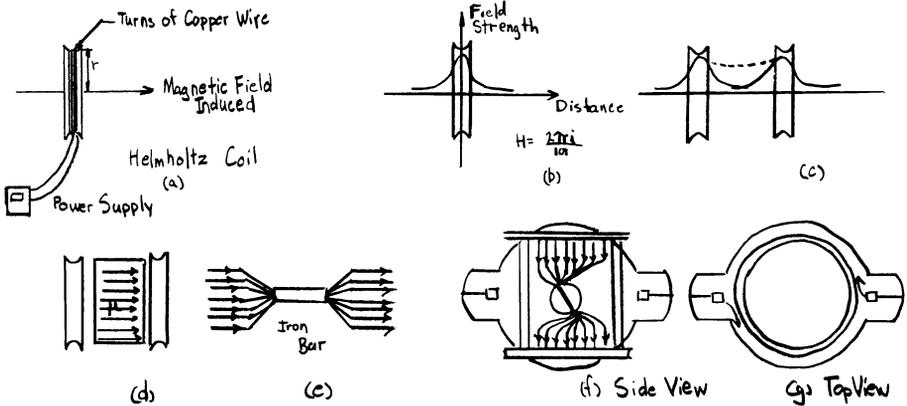


Figure 5: Field Generated by Helmholtz Coils

power supply pushes current through the copper, which induces a magnetic field according to the “Right Hand Rule”. The magnetic field along the axis of the current loop peaks in the vicinity of the centre of the loop and quickly drops off farther away from the centre. If two coils are aligned properly and in close proximity, their contributions add up to the overall field. Furthermore, the permeability of these fields in air, or in partial vacuum as the case may be, is less than the permeability of the flux in an iron bar. So the iron bar diverts field intensity from the Helmholtz coils, producing a field distribution similar to, but much different than that of the earth.

## Results

After the construction and assembly of the parts of the Auroral Simulator were completed, the piece was sent to the National Gallery of Canada where the Helmholtz coils were put on. Unfortunately, the installation team dropped the Simulator (Simulator #1) while trying to install it in its display tank. A second Simulator (along with some handling instructions) was quickly shipped out. On this simulator (Simulator #2), the Terrella seemed to have quite a bit of movement within the 12-litre flask, perhaps due to its unscrewing itself from the top insert. It was left in place for the opening of the exhibit, but another was made to replace it. A larger diameter support rod (3/16 inch), outer insert, and outer Kovar seal were used and the threaded mating parts were soldered together on the next replacement (Simulator #3). The exhibit then moved to “The Power Plant” in Toronto and the replacement simulator (Simulator #3) was sent and the other (Simulator #2) was sent back to me for rebuilding with a heavier support rod. This involved cracking off one sidearm, removing and reinstalling the Terrella, replacing the electron gun and repumping. The exhibit then travelled to Banff but not before both existing Simulators (Simulators #2 and #3) were returned to me. The computer control

team claimed that the pressure had gone up in both. Both were repumped, degassed by running the guns and gentle oven heating, sealed off and sent to Banff.

When a person interacted with the exhibit, she would pick up a bell jar containing a heart. This would trigger the computer controls to abruptly initiate a striking voltage for the Gas Discharge Mode and gradually increase this voltage to produce a bright plasma in the simulator. The computer would then slowly ramp up current through the Helmholtz coils to about 35 Oersted inducing the magnetic field and restricting electrons from entering the area of the field. The region of the field would then become dark due to absence of gas ionization. With adjustment of the voltage and the magnetic field, conditions would exist such that some electrons were able to make their way into the field along the direction of the magnetic field. These then would spiral down into regions of field concentration around the poles and result in the emissions of low energy electrons (50 eV) which, in turn, would be trapped in the field close to the Terrella and result in localized plasma on the surface of the Terrella around the regions of the magnetic poles. The voltage would then be dropped off and the magnetic field dropped off soon after that, completing the computer cycle.

## Discussion

Just as all the details of the formation of the atmospheric aurora are not fully understood, so too some of the details of how this simulator forms its model aurora are not yet clearly understood. When the Helmholtz field is applied to the simulator operating in the Gas Discharge Mode, electrons are sharply deflected from their trajectories towards the Terrella and execute tightly-curved paths limiting their passage to the centre of the structure. They are retained in orbits in regions close to the electron guns. As the electron accelerating voltage and the magnetic field are adjusted, electrons somehow reach the Terrella and excite a local plasma where the magnetic field is concentrated. This may be because some electrons loop along the walls of the glass vessel, generating, as they hit the glass, more secondary electrons that are also closely constrained in curved paths at the surface of the glass. The electric field gradient is towards the Terrella so these electrons finish up in regions opposite the poles of the Terrella. Secondary electrons emitted from the glass in these regions then travel along the direction of the field lines, rather than being deflected at right angles to them. They would then accelerate towards the Terrella. These electrons would typically have too high an energy level (50 eV) to excite the gas molecules around them but would instead bombard phosphors on the Terrella. The lower energy (10-15 eV) secondary emission of the phosphors would be more effective at causing the gas around them to emit light as they are trapped in the field at the surface of the Terrella. This may be the mechanism of surface plasma formation.<sup>9</sup>

The degassing of the simulator by the running of the electron guns required the monitoring of current or voltage drop across a resistor that was dependent on the pressure inside the flask. Operating this way, the simulator could be thought of as a huge, uncalibrated Ionization Gauge.

There were some details of the construction of the Simulator that caused some unwanted features. The silver solder used for suspending the Terrella was a low temperature type

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<sup>9</sup>From notes and conversations with Peter Sewell.

(active range 177°C to 317°C). While this is lower than conventional silver solder and was chosen to avoid any “hot spots” in the assembly procedure, it did have some drawbacks. The solder temperature limited the degassing temperature of the simulator on the line.

There were some problems with the computer cycle that were blamed on increasing pressure within the completed sphere. At first, this was thought to be a sign of leakage and the simulators were repumped. When the pressure increase happened again, it was thought to be degassing caused by electron bombardment of surfaces in the simulator. Repumping of the simulator was done with the guns running. This has seemed to solve the problem, although the simulators seem to require work every time the exhibit changes locations.

## **Conclusion**

The performance of any scientific apparatus can be rated by how well it meets the requirements of the person who requested it. In light of this, the Auroral Simulator performed well. This piece was intended to be an artistic representation of the Northern Lights for an audience of the general public. It was not a device for experiments, and as such, cannot be compared to the work of Birkeland nor the many other model experiments aimed at studying certain details of the aurora or the aurora as a whole.<sup>10</sup> The plasma did concentrate at the poles where it was supposed to concentrate, it was safe to view, portable, operated at low voltages (relative to Birkeland’s), and was somewhat maintenance-free. As part of the “Charged Hearts” exhibit, the Auroral Simulator started its service at The National Gallery of Canada. It has since travelled to equally-renowned centres in Toronto (The Power Plant), and in Banff. It may be going to Detroit next and perhaps Paris after that.

As a piece outside the exhibit, it may prove to become more interesting as we approach the year 2000. The next 11-year peak of solar activity is expected then. In fact, the first of what are expected to be many Magnetic Storm Warnings building up to the peak was issued May 1st 1998.<sup>11</sup> At the last peak in 1989, a super aurora knocked out Quebec’s entire power grid and the Northern Lights were seen as far away as Mexico. Up to one million amperes of current flowed along the aurora setting up fluctuations in the earth’s magnetic field, inducing electric currents in long conductors such as telephone wires, power lines and pipelines. The mechanism responsible for this phenomenon can be explained, at least in part, with the operation of the Auroral Simulator. Aside from the function of the piece, its construction provided some innovation in suspension of an inner sphere and use of electric and magnetic fields. These techniques may become useful in another applications.

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<sup>10</sup> Eather, 237.

<sup>11</sup> The Geophysics Division of the Geological Survey of Canada.

## **Acknowledgments**

I thank Catherine Richards for providing the challenge. I am deeply thankful for the teachings of Peter Sewell. Collaborating with him and developing our friendship have been the most satisfying parts of this adventure. I find it fitting to also mention here two photographers at the National Research Council of Canada, Dan Gamache and Tom Devecseri. Their help with this paper and the presentation of it is greatly appreciated.

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# **Fabrication of the Gregar Extractor**

by

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Extraction of solids by organic solvents is a fundamental chemical process. A number of procedures and devices are available for accomplishing this goal. The most common form of extractor presently used for extraction of solids, the Soxhlet extractor, works on the principle of cyclic replacement of solvent by fresh solvent. This design has been a mainstay in laboratory procedures for about a century. The Gregar extractor, unlike the Soxhlet design or any other previous design, works on the principle of continuous displacement of solvent. This allows the solid being extracted to be continuously immersed in solvent, and maintains a constant level of solvent within the extractor. Therefore, this new extractor avoids many of the problems typically encountered using Soxhlet extraction.

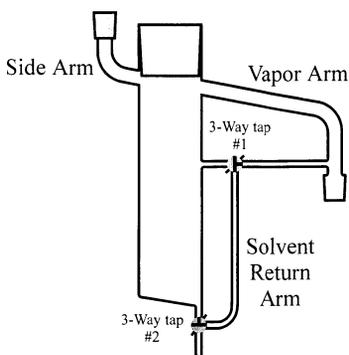
Solids such as natural and synthetic polymers and other materials which tend to either swell and/or become too sticky when exposed to organic solvents can be difficult to extract using conventional extraction procedures. This is due to particles binding together as solvent is cyclically replaced, leading to plugging of the extractor or swelling of the particles. Swelling makes extractor plugging even worse and leads to rupturing of extraction thimbles. The Gregar extractor alleviates these difficulties and provides a practical technique for extraction of many materials not well-suited for extraction by existing procedures.

Two alternative designs, differing primarily in the length and configuration of the vapor arm, have been fabricated and tested (see schematic illustrating extractor components and design). Both are equally effective in extraction of soluble products. Each has advantages for specific circumstances (Figure 1):

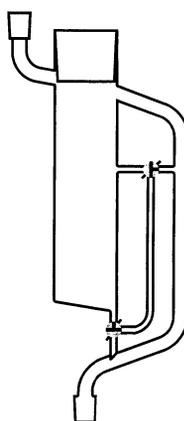
\* Author to whom all correspondence should be addressed.  
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# Extractor Body

## "Horizontal" Configuration



## "Vertical" Configuration



# Ancillary Equipment

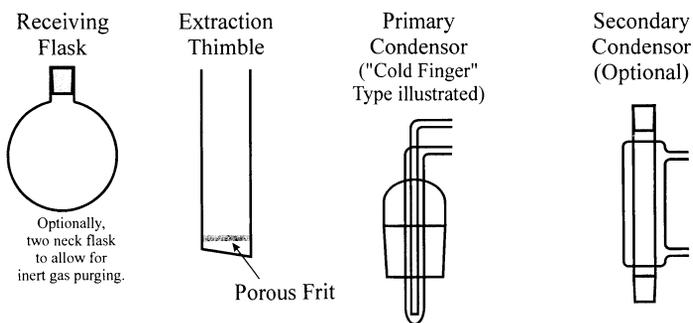


Figure 1

### **1. Horizontal Configuration.**

In this configuration, the length of the vapor arm is minimized. This lessens the vapor path, which is advantageous when extracting with high boiling solvents or when operating on a large scale. However, this configuration requires more space than the alternative vertical configuration. In this configuration, the drain for the extractor body (controlled by the 3-way stopcock (#2)) is not coupled to the vapor arm.

### **2. Vertical Configuration.**

This is the preferred configuration for use with low boiling solvents and for small scale operations. It occupies less space than the alternative horizontal configuration; in addition, the drain for the extractor body is directly coupled with the vapor arm. This allows the extractor body to be drained into the receiving flask without first requiring that

the receiving flask be separated from the extractor body. As noted above, however, the additional length required in the vapor arm in this configuration is a disadvantage when extracting with high boiling solvents. (Note: this can be overcome by adding additional heating and/or insulation to the vapor arm to increase the efficiency of vapor transport.)

Both designs are uncomplicated and fabrication is relatively inexpensive. Operation of the Gregar extractor is simple, convenient, and suitable for unattended operation.

**Example of Operation.** A fully assembled extractor in the vertical configuration set-up for normal operation is illustrated in Figure 2. The solid material to be extracted is

### Vapor and Liquid Flows During Normal Extractor Operation

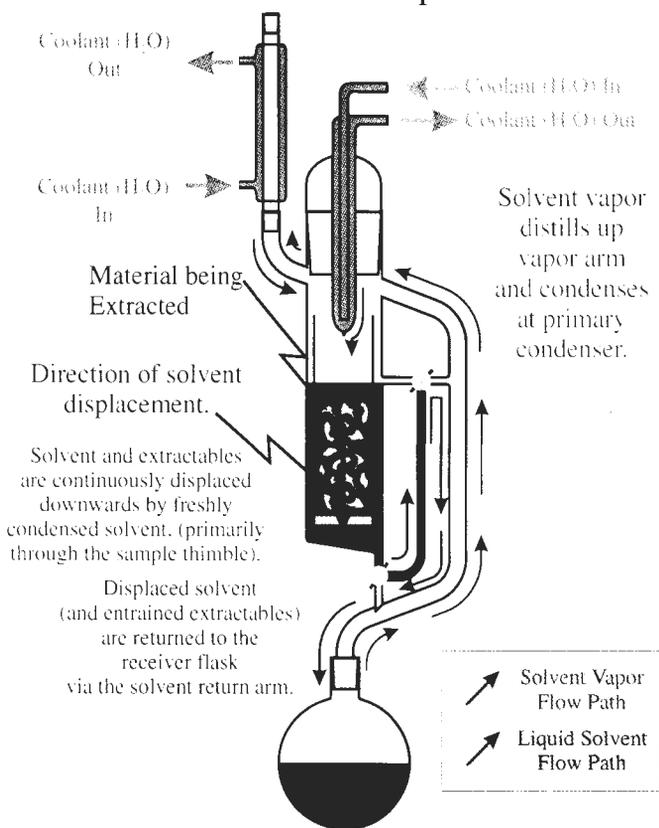


Figure 2

placed in the extraction thimble, and this is placed in the extractor. Ancillary equipment (condensers etc) are connected to complete extractor assembly. If desired, inert gas purging is established. Solvent can either be added to the appropriate level via the side arm, or may be distilled into the extractor body from the (pre-loaded) receiver flask.

Solvent is distilled from the receiver flask, allowed to pass through or over the solids and allowed to recycle to the receiver flask (via the solvent return arm) for as long as needed for adequate extraction of the sample. When extraction is complete, reflux is discontinued, and the extractor assembly allowed to cool to ambient temperature. Solvent remaining in the extractor body is then drained into the receiver flask by rotating the 3-way stopcock (#2) such that both the main extractor body and the solvent return arm are drained. The extraction thimble, now containing residual insoluble material, and the receiving flask are then removed and the desired products are recovered.

Some of the advantages of the Gregar Extractor (patent pending) are: (1) up to 50% faster extraction depending on the type of compounds (2) ability to perform normal solid to liquid extraction, liquid to liquid extraction and liquid to reagent extractions (depending on the compounds) and (3) several different designs and sizes are available.

Once we had finished with 3 or 4 prototypes and worked out the problems, I set forth to simplify the extractor for ease of operation and fabrication. The construction of the final version is fairly uncomplicated except for the last assembly, which includes the addition of the side vapor arm. This is more involved because the vapor arm is made from one piece of glass tubing and bent to fit the body's configuration. This is my personal preference, however, because I do not like to piece the vapor arm together and show several splices along its path. When using one piece, the finished extractor has a more professional appearance.

The first step is to build the body of the extractor (see Figure 3). In preparation for the first splice, I prepare a 14/20 outer joint that has been tooled on reduced diameter tubing.



Figure 3

We used to call this an old style outer joint. A 90 degree bend is required and the bent tube is cut at 1-1/2". This is measured from the outer edge of the vertical section of tubing to the mark. The end is picked clean to remove the knife mark with a hot glass rod and a slight flare is given to the end. As shown in Figure 4, I then splice a 14" long piece of matching diameter tubing onto an outer joint. In this case I used a 40/35 medium length



Figure 4

outer joint and 45mm standard wall tubing. Both ends of these parts are fire cut to eliminate knife or saw marks that may produce blemishes in the splice. Ideally, you should order a sufficiently long outer joint tooled special and eliminate this splice completely. By doing this, its appearance will be much nicer. After this splice is completed, I blow a hole in the tubing and add the prepared 14/20 joint (Figure 5). I then



Figure 5

blow a bubble directly across (180 degrees) from the joint. This top section is then flame annealed (Figure 6). I do not blow the bubble open until later. It will accommodate the side vapor tube.



Figure 6

With the body still positioned in the lathe, I round off the end at 11.5". This bottom is pulled slightly thinner than normal in preparation for creating the angle (Figure 7). I



Figure 7

reheat the end concentrating the flame more on the side with the joint. This will end up being the shorter side of the angled bottom. I use a graphite paddle to produce the angle I'm looking for (Figure 8). This area is then flame annealed. I then switch the body from

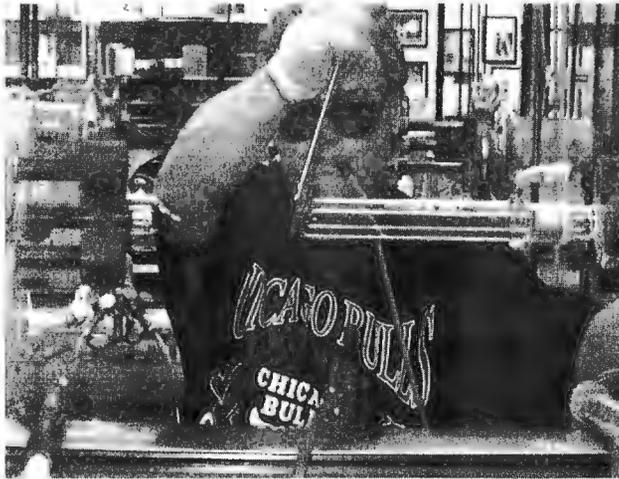


Figure 8

the lathe to my ring stand and splice on a 2mm bore, 3-way, right-angled Teflon stopcock barrel. Orientation is important, so be sure to have the 90 degree stopcock arm lined up with the bubble (Figure 9). I then flame anneal this section of the extractor to remove



Figure 9

the strain which allows me to work on the next portion of the fabrication (Figure 10). A



Figure 10

second 2mm bore, 3-way, right-angled Teflon stopcock is added and aligned with the same orientation as the first stopcock, 100mm up from the bottom and again flame annealed (Figures 11 & 12). This part is set aside to cool while I fabricate the arm to attach



Figure 11



Figure 12

the two 90 degree, right-angled arms coming off the stopcocks. I make a 90 degree bend with tubing that matches the stopcock arms. I hold it in place over the two stopcock arms and mark it and the two stopcock arms with a Sharpie. These tubes are cut with a glass knife, and also cleaned by wiping off the marks with a rod in the fire and then splicing the bent tube in place (Figure 13). You must attach the tube to one end and then work the second end while keeping the first attachment hot so it will not cool and crack (Figure 14). After you have the tube in place with the proper alignment, work the splices in quickly while keeping both ends hot (Figure 15). This area is then flame annealed and set aside for preparation of the final piece.



Figure 13



Figure 14

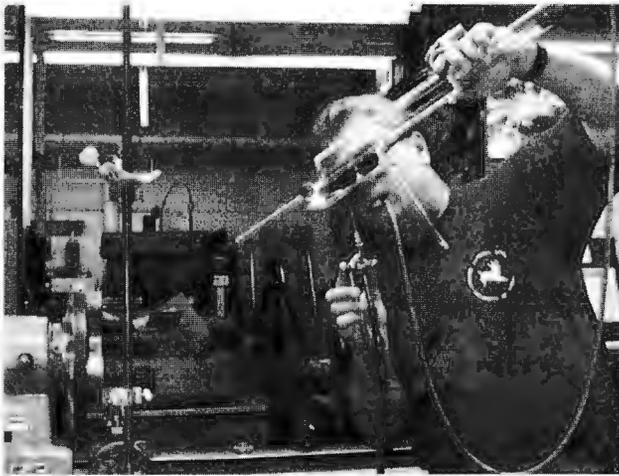


Figure 15

I splice a 14/20 inner joint onto a 30" long piece of 1/2" medium wall tubing. Figure 16 shows a prepared vapor tube whose fabrication is now described. As mentioned in the



Figure 16

beginning of the paper, this is my personal preference because I do not like to see splices along the vapor tube. It is more difficult, but the finished apparatus looks more professional when it is fabricated this way. I make the first bend one inch up from the top of the joint at a 110 degree angle. Secondly, I lay the tube alongside the body of the extractor and make a mark approximately 100mm up from the first bend. After this bend, the vertical leg of the vapor tube will be the proper distance out from the body so that it is aligned with the second stopcock. Again, I lay the vapor tube next to the body and

place a mark at the height necessary for the final bend at the top of the vapor tube. Once bent, this tube should line up with the bubble we made earlier. Laying the vapor tube alongside the body of the extractor again, I now place marks where the top bent tube will get cut off and the two holes will be blown open to align exactly with the stopcocks (Figure 17). I then produce a slight flare to the top end of the vapor tube that will attach to the body and then blow open the holes for the stopcock attachments (Figure 18).



Figure 17



Figure 18

Now this is when the glassblowing gets fun and exciting. You must attach the vapor tube to the extractor body. This means working three attachment points at the same time. The most important thing to remember is to achieve the proper alignment and keep all three areas being worked hot at all times (Figures 19-21). After all three connections are



Figure 19

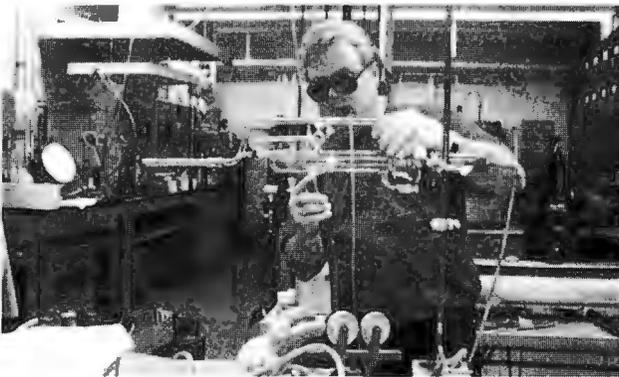


Figure 20

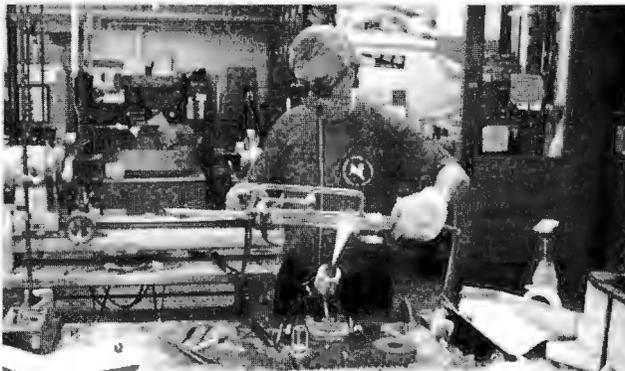


Figure 21

complete, it is important to flame anneal thoroughly so it does not crack before oven annealing. If you did not make perfect bends while constructing the vapor tube, now is the time to clean up these areas. While everything is hot and supported in so many places, you can re-work your bends without worry of sagging or breaking.

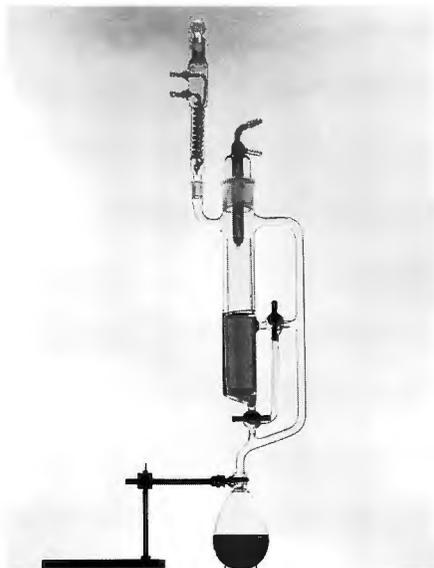


Figure 22

Figure 22 is a photo of the completed extractor with its ancillary equipment in place. This has been a very rewarding project for me. Working directly with the chemist and co-inventor, Dr. Ken Anderson, has created many opportunities for me. The design from this collaboration has produced the registration for a patent and, the ultimate honor, having it named the “Gregar Extractor”. I have also had the opportunity to meet and be recognized by our patent attorneys and Department of Energy representatives.

We made some improvements to enhance the extractor. An extra coarse fritted disc was added to the bottom of the sample thimble, and an equalizing slot was added to prevent the accumulation of gas or air bubbles. In addition, an angled bottom configuration on the thimble was made which reduces dead space and provides a low point drainage tap. Another improvement was to have a special cold finger type condenser in the upper body which adds compactness and increases the overall efficiency. Minimizing the annular gap between the body and the thimble reduces solvent volume and also increases efficiency. One final word: when a customer visits your glass shop and suggests an idea for an apparatus that seems to be somewhat vague, be interested and responsive. Don't hesitate to build prototypes because as professional scientific glassblowers we have a lot of knowledge and practical design ability to offer, and the rewards can be bountiful.

#### Acknowledgment

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# Heavy Duty NMR Tube Washer

by

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The NMR tube washer is just that: a device to clean the inside of NMR tubes, in this case 5mm. This is accomplished by inserting the washing device into a collection vessel, filling the reservoir with cleaning solution, then drawing a vacuum to pull the solution through the inner tube inside the NMR tube with the excess drained into the collection vessel. The 3mm inner tube is the most common repair that I make on NMR tube washers. The idea to lower the frequency of breakage is helped through the following. Diagram #1 shows the 3mm inner tube with a piece of 1/4" tubing on the end then bent upward to attach to the inside of the 24/40 inner joint. This, coupled with a smaller inside diameter of the shaft, does not allow enough movement to break the 3mm inner tube.

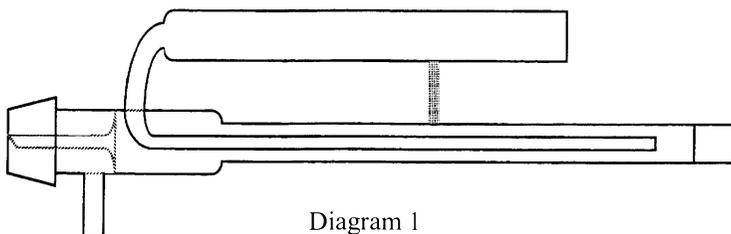


Diagram 1  
Heavy Duty NMR Tube Washer

Diagram #2 shows a holder for installing the 3mm inner tube. There is a small hole toward the front to assist in assembly. Masking tape is wrapped around the 5mm od tubing at two different diameters to hold the 3mm inner tube steady within the shaft.

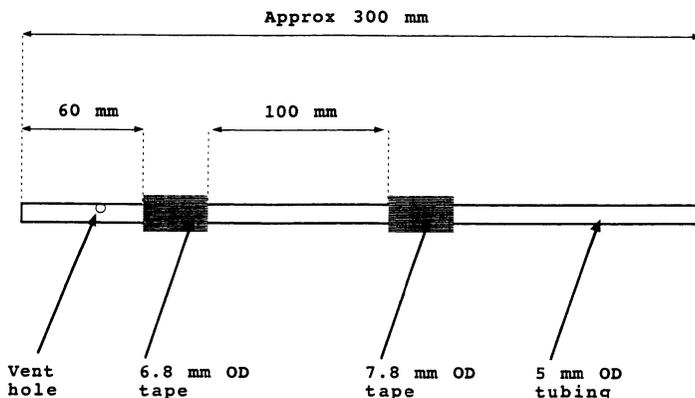


Diagram 2  
Inner Tube Holding Tool

Diagram #3 shows the inner tube. The length of the inner tube combined with the length of the shaft keeps the opening of the NMR tube away from the base of the inner tube where breakage could occur. Diagram #4 illustrates the shaft itself. Notice that the last

10mm of length is 10mm standard on the shaft of 10mm x 6mm tubing. The purpose is to plug the end with a septa (Aldrich p/n 10,070-6) which will seal the end and also hold the NMR tube. I have been able to flare the end of the 10mm X 6mm eliminating the need for two splices on the shaft.

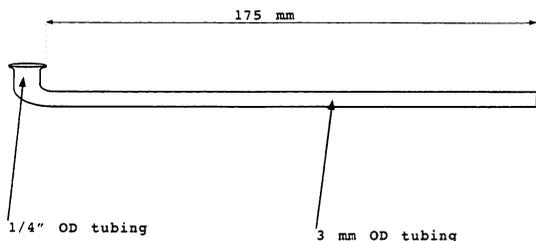


Diagram 3  
Inner Tube

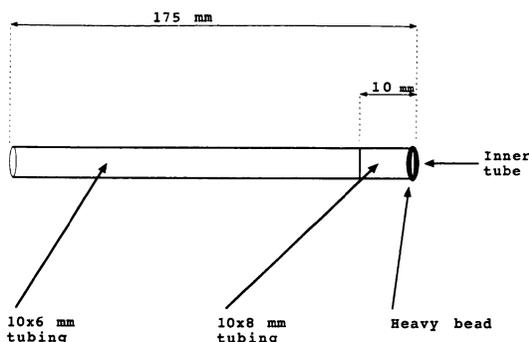


Diagram 4  
Top Shaft

Diagram #5 is the reservoir. This attaches from the outside to connect with the inner tube.

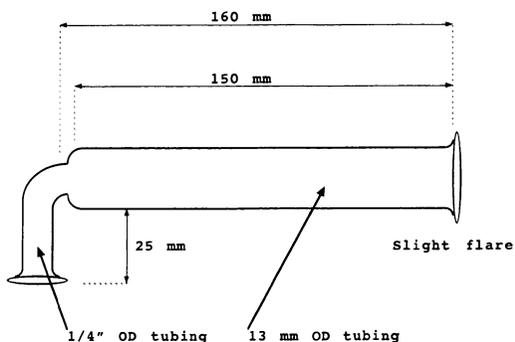


Diagram 5  
Reservoir

After the splice, install a 6mm rod brace between the reservoir and the shaft. Diagram #6 shows the bottom of the washer. A bubble is blown on one side to allow placement of the inner tube. At this point, parts can be assembled. Attach the shaft (#4) to the 24/40 (#6). The holder (#2) is inserted from the rear and the inner tube (#3) through the front into the holder and rested in the bubble. I have made adjustments just prior to this on the

inner tube (#3) to assure the fit in the bubble while in the holder. The holder will protrude past the back of the shaft just enough to grab and remove after sealing in the inner tube.

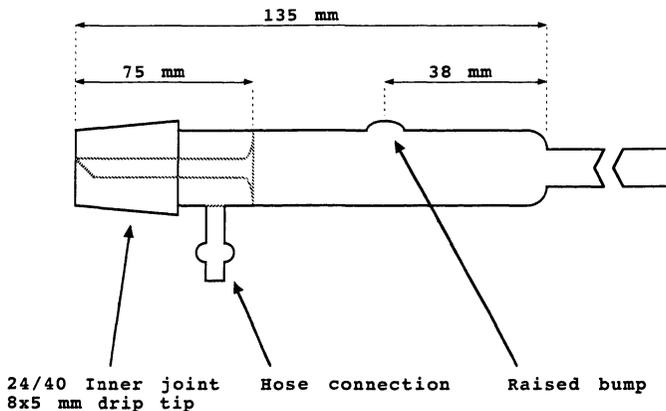


Diagram 6  
Bottom Assembly

A large piece of tubing is placed over the end for blowing which also clears the holder.

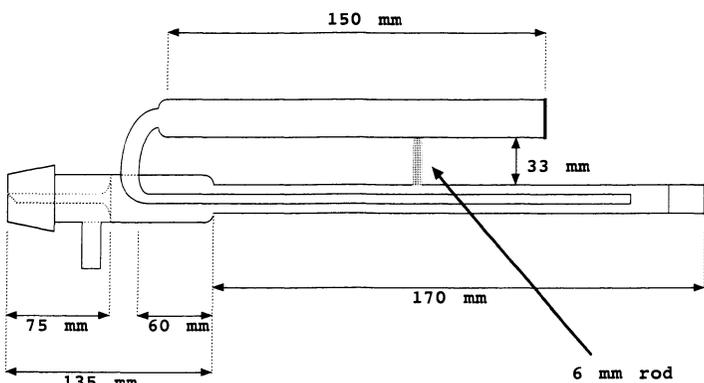


Diagram 7  
Heavy Duty NMR Tube Washer

At the same time of sealing in the 3mm inner tube, I seal on the reservoir (#5). I have done both, either seal the 6mm rod brace at this time or after installing the inner drip tip and hose connection which is assembled next. If you attempt to make this device and need any help or advice, please feel free to contact me at any time.

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# Lindbergh Perfusion Apparatus

by

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Most people recognize Charles Lindbergh, the aviator, but, first and foremost, he was an engineer with a wide variety of interests. In the early 1930's, he met a French scientist by the name of Dr. Alexes Carrel and became interested and excited about perfusion. Carrel had won a Nobel Prize for his technique in the suturing of arteries and, of course, Lindbergh was still fresh from his flight across the Atlantic. Lindbergh set to work with building a sterile environment in which to do the research. This was before any peristaltic type of tubing pumps, and they had a lot of grand ideas about what could be done with perfusion. Some of these included removing a diseased organ or body part and treating it in a vigorous manner without worrying about complications from the rest of the body, and then, when the organ or limb was healed, re-attaching or re-installing it in the body. Carrel also felt that this could be done using body parts from someone recently deceased. Another hope that they had was that with perfusion they could actually watch a disease develop in an organ.

One of the bigger hopes was to actually take glands such as thyroid, adrenals and other endocrine function to the point that they would yield an abundance of pure hormones, thus eliminating the need for using parts from animals. This would also have created the need to build much larger apparatus to accommodate human organs and, in their words, "transform medicine from a fine art to an exact science." However, most of the experiments were carried out using various organs from cats and chickens. Carrel and Lindbergh published several papers together and, because of their notoriety, every time they published, they had a large following in the media.

Now on to the apparatus. Lindbergh states that his first pump was built by the glassblower at Princeton but, in his 1935 paper from which these drawings were taken, he gives credit for the glassblowing to an O. Hopf who was the glassblower at the Rockefeller Institute. Since this was over 60 years ago, there is probably no one here who worked with him, but possibly someone who remembers the name. The perfusion pump is the most sterile part of the entire apparatus and was always handled with aseptic technique. The organ to be perfused is attached to the cannula (#3) and the apparatus sealed off. During operation neither the organ nor the perfusion liquid came into contact with any corks or the exterior. Following the arrows, we can see the flow of the liquid through the organ and back into the pump. The pump itself (Figure A) was a series of three float valves in chambers that opened and closed with the rotation of the pulsation valve. Pressure is put on the pump through the filter bulb (#12) at the arm (#11) and returns through the system at (#22) allowing the perfusion fluid to be moved through. The fluid is also filtered through the 150 mesh platinum screens (#21), then through the organ, after which it flows through silica sand held in place by two 52 mesh (#5) platinum screens. During the actual operation, #1 and #2 were sealed off. The center part of the

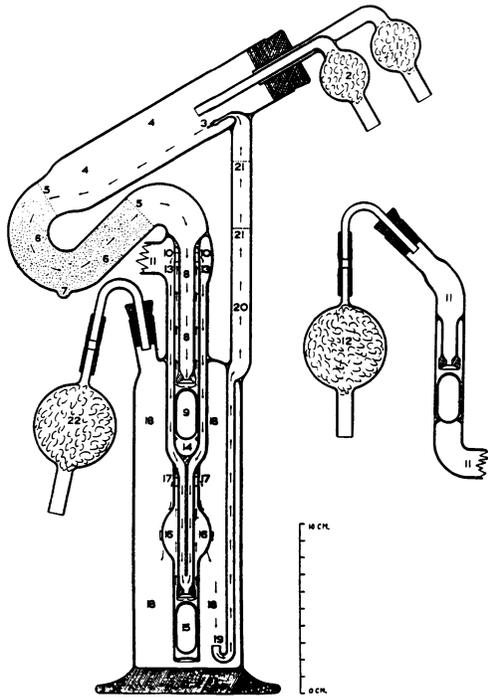


Figure A

Cross-section of pulsating perfusion pump, (1-22).

chamber (#8) with holes (#10 to #13) exists for pressure equalization. As stated before, this was before tubing pumps so Lindbergh came up with a pulsation valve (Figure B).

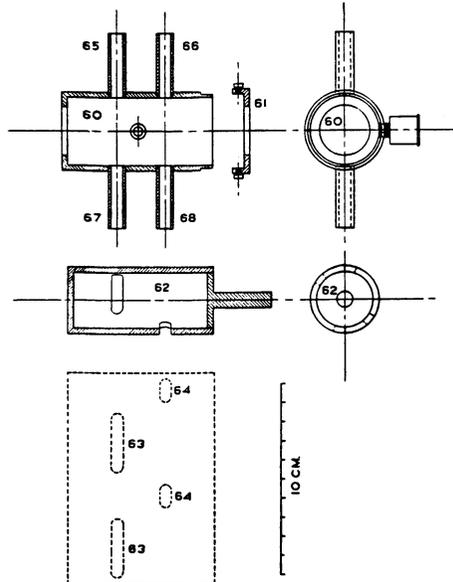


Figure B

Pulsation valve, (60-68).

The outer shell and cap (#60 and #61) were made of steel and the rotating center shaft (#62) was made of brass with two large slots and two small slots so that each rotation caused two pulsations. This was controlled by a small electric motor and a rheostat. This was used in conjunction with the oil flask (Figure C) so it never came into contact with

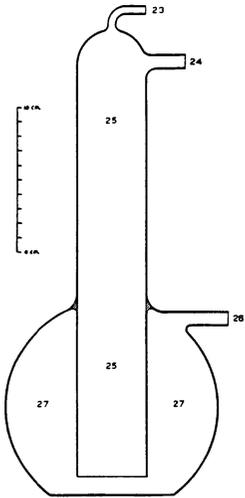


Figure C  
Cross-section of oil flask, (23-27).

the gas, in this case oxygen, or the perfusion fluid. The final piece is the combination oil check valve, oil trap, one-way gas valve and manifolds (Figure D) that controlled the

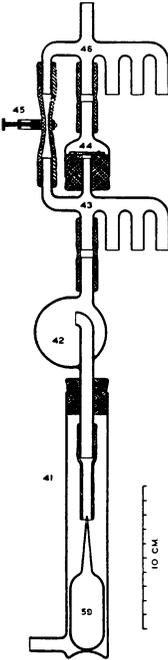


Figure D

directional flow through the organ. All of the glass parts (Figure E) are mounted in an incubator along with a copper coil which is used as a ballast and to maintain constant

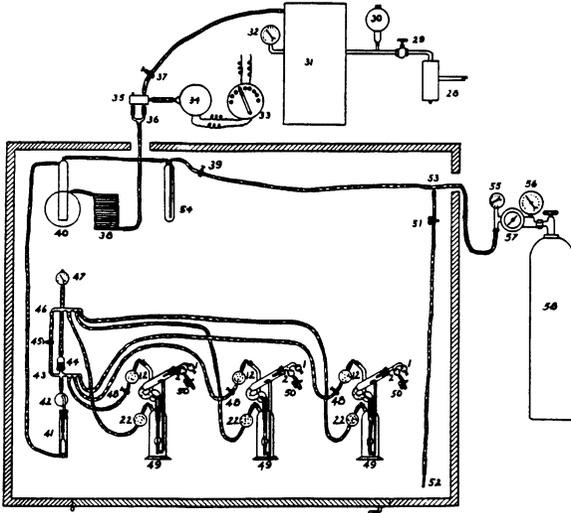


Figure E

Cross-section of oil check valve, oil trap and one-way gas valve assembly (41-46, 59).

temperature. The volume of the coil is greater than the volume moved by one pulsation so it minimizes the amount of new cold air introduced. By adding your fluids and organ to each pump, you are ready to do your perfusion. The actual pressure was fine tuned with several pinch clamps #37, 39, 45, 48. Lindbergh and Carrel worked on this until the late 1930's without achieving the desired results. The next view (Figure F), after the

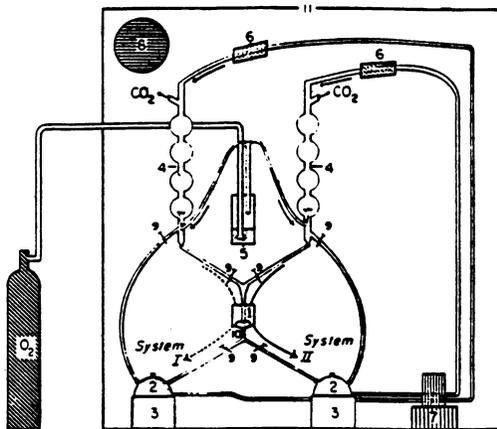


Figure F

Double perfusion apparatus for the isolated liver. (1) Liver in plastic chamber. (2) Reservoirs for perfusate. (3) "Magnestir" providing homogeneous mixing of the perfusate. (4) Glass "lung" in which perfusate is oxygenated. (5) Humidificator for oxygen. (6) Filter. (7) Pump. (8) Heater with thermostat maintaining temperature at 96°F. (9) Stopcocks. (10) Flowmeter. (11) Cabinet-containing the system.

invention of tubing pumps, is from some work in the lab of Dr. Robert Good when he was at the University of Minnesota.

During my years at the U of Minnesota, I had the opportunity to visit his labs and observe liver perfusion firsthand. At the onset, this seemed like a good idea. What I did not realize was that not only would I be there for the opening and removal of the liver but the initiation of the demise of the rat. I have to admit that in the hands of a skilled researcher, the organ can be removed and connected to the apparatus almost without missing a beat. In the set-up, we are still in the incubator box (11). We have two glass pieces to simulate the lungs (4), a bubbler for the oxygen (5), the pump (7), the glass containers (2) with magnetic stirrers (3). But, we are still fine tuning the glass container containing, in this case, the rat liver (1) with clamps (9). Things have gotten a lot simpler. Also, they were not as worried about outside contamination.

Over the years, I have built many different styles of perfusion apparatus but I became interested in Lindbergh's after seeing one of his pumps on display at his boyhood home in Little Falls, Minnesota. It is always interesting to follow a trail of development of a type of apparatus but also gratifying to see that, when Lindbergh set out to create a sterile environment, the first place he went was to a scientific glassblower.

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# Neon Vacuum Systems

by

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Since the first commercial neon signs were introduced in France in 1921, vacuum systems for evacuating and filling neon tubes have changed very little. Early vacuum systems used mechanical vacuum pumps, mercury diffusion pumps, and mercury manometers to process neon tubes. Glassblowers and manufacturers of scientific equipment have developed materials that are capable of producing excellent vacuum systems for the chemical laboratory and the technology transfer to the neon industry should be easy. Development of efficient mechanical vacuum pumps, high speed secondary pumps, electronic vacuum gauges, and improved glassware components have made processing neon signage more efficient and reliable. Most equipment suppliers to the neon industry make use of the latest technology.

It is generally accepted in the neon industry that the vacuum system be capable of evacuating the neon tube to pressures below  $10^{-3}$  Torr before the tube is filled with gas and sealed. Vacuum pressures at this level require equipment in good working order and the use of a secondary vacuum pump. The following is a guideline for equipment selection:

## MECHANICAL VACUUM PUMPS

Mechanical vacuum pumps are the primary vacuum source in any vacuum system. Generally, a pumping speed of at least 5.5 cubic feet per minute (160 liters per minute) is desired for fast evacuation of air and water vapor from the neon tube during and after bombardment. The pump should be a two-stage pump capable of ultimate vacuum pressures of  $5 \times 10^{-3}$  Torr. Direct drive or belt drive pumps with comparable pumping speeds work equally well.

## SECONDARY VACUUM PUMPS.

It is desired to reduce vacuum levels in the neon tube to levels below  $10^{-3}$  Torr. Several types of secondary vacuum pumps are available for this purpose.

### Oil Diffusion Pumps

Single-stage glass oil diffusion pumps are commonly used in the vacuum processing of neon tubes. With an average pumping speed of 8 liters per second and an ultimate vacuum pressure of  $5 \times 10^{-6}$  Torr, these pumps work quite well for processing one to two neon tubes at a time. Their simple design and low cost make them a valuable addition to the neon vacuum system.

Multi-stage glass oil diffusion pumps are available with pumping speeds of 375 liters per second and have operating vacuum pressures of  $5 \times 10^{-8}$  Torr. These pumps are capable of processing up to eight neon tubes per cycle (with additional bombarder

capabilities) and operate very well in high production situations. Up to 400 neon tubes per day can be processed with this high-speed manifold and diffusion pump.

Multi-stage metal oil diffusion pumps are adaptable to neon vacuum systems. These pumps have varying pumping speeds (50 - 1000+ liters per second) and ultimate pressures of  $5 \times 10^{-8}$  Torr. Although adaptable to neon vacuum systems, special adapters are required to connect the pump to the glass manifold.

Tests of various diffusion pumps were conducted in April 1997 at the Glass Art Society meeting in Tucson, Arizona. The tests were designed to compare different diffusion pump designs for speed and overall performance in pumping neon tubes. A glass single-stage updraft pump, a single-stage glass side draft pump, a glass three-stage down draft pump, and a metal three-stage down draft pump were tested. Diffusion pumps were operated on a special high vacuum manifold that allowed side by side testing to evacuate and fill an eight-foot long, 15mm neon tube. This test allowed identical conditions for each pump. (Figure 1 shows the test manifold for side by side testing of diffusion pumps.)



Figure 1

### Turbomolecular Pumps

Turbomolecular pumps are occasionally used as secondary vacuum pumps on neon vacuum systems. Turbomolecular pumps provide reliable hydrocarbon-free, high vacuum pumping. Several disadvantages include, high initial cost and susceptibility to failure due to in-rush of air.

### VACUUM AND GAS FILLING MANIFOLD

Due to the potential shock hazard during bombardment and mercury corrosion with metal vacuum manifolds, glass has become the material of choice for neon vacuum systems. (Figure 2 show a typical metal neon filling manifold.) Development of glass greaseless high vacuum valves and O-ring connectors has led to very reliable and durable vacuum systems. The outgassing properties of Teflon and Viton materials used in these components allow vacuum pressures into the  $10^{-7}$  Torr range. Glass O-ring compression fittings allow for easy disassembly when cleaning is necessary.

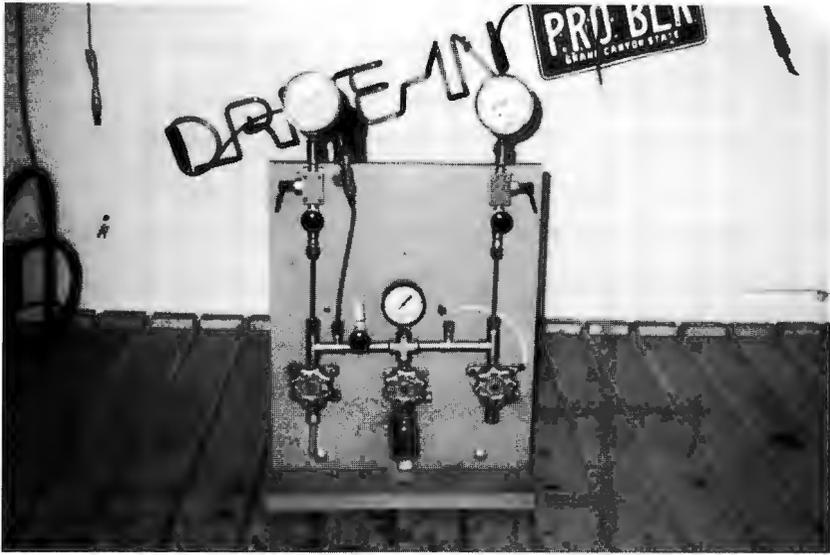


Figure 2

The manifold components should be of sufficient size to reduce conductance losses in the manifold and connecting tubes. The manifold is constructed to accommodate vacuum pump connections, vacuum gauges, neon and argon inlet valves, air inlet, and a solids particle trap.

Figures 3 and 4 are schematics for constructing neon vacuum and filling manifolds using glass O-ring compression fittings and Teflon high vacuum valves.

### STANDARD NEON MANIFOLD

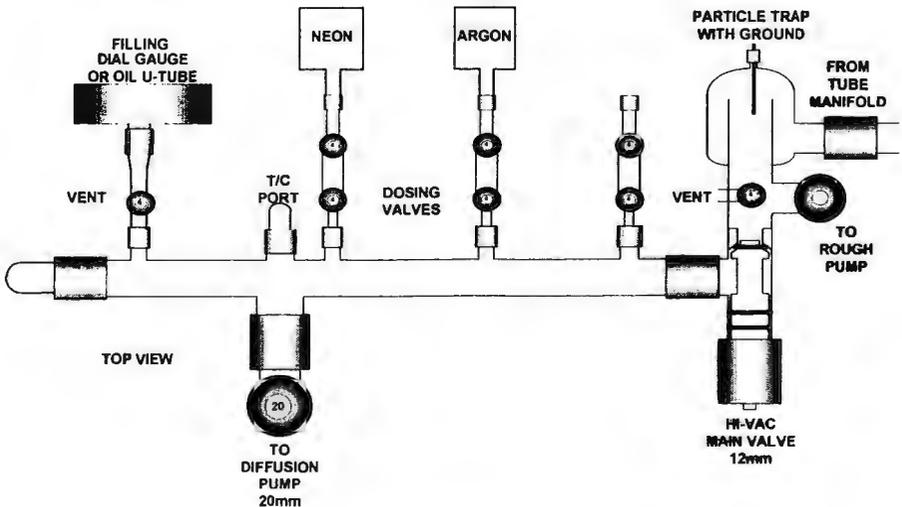


Figure 3

## HIGH PRODUCTION NEON MANIFOLD

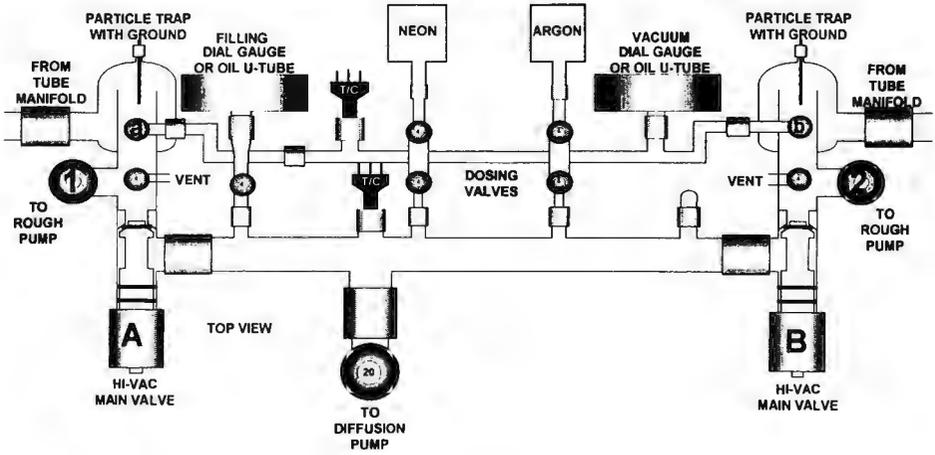


Figure 4

Figure 5 is a photograph of the glass high production neon manifold with a high conductance manifold as drawn in Figure 4.



Figure 5

## VACUUM MEASUREMENT

The all-glass vacuum system with a secondary pump will generally reduce the manifold pressures to levels in the  $10^{-4}$  to  $10^{-7}$  Torr range. Since thermocouple vacuum gauges indicate vacuum pressures from atmosphere to  $10^{-3}$  Torr, additional vacuum gauges capable of reading into the  $10^{-7}$  Torr range are necessary. The following gauge types are useful for measurement below  $10^{-3}$  Torr:

Cold Cathode Gauges indicate vacuum levels between  $10^{-3}$  to  $10^{-9}$  Torr and are very durable. The cold cathode gauge is not affected by sudden exposure to atmosphere. This is a good gauge type to use in a rapid cycle system such as the neon pumping system. The gauge should be placed in the manifold where it is isolated from possible high voltage flashback during the bombarding process.

Hot Cathode Ion Gauges indicate vacuum levels between  $10^{-3}$  to  $10^{-11}$  Torr and are more susceptible to sudden exposure to atmosphere. Positioning the gauge between the secondary pump and the manifold will reduce premature filament burn out. Additionally, the use of iridium coated filaments further reduces failure. This gauge should also be protected from flashback during the bombarding process.

## COLD TRAPS

Although addition of a liquid nitrogen trap would improve the ultimate vacuum of the system and eliminate back streaming of the diffusion pump oil, it is generally agreed in the neon industry that cold traps are not necessary. Several knowledgeable experts in the neon community have conducted tests to determine the effects of diffusion oil on the neon tube. Their findings indicate that there is no adverse effect on the finished product.

## CONCLUSION

Advances in vacuum equipment technology have allowed the construction of all-glass vacuum systems that are capable of producing vacuum levels in the  $10^{-6}$  to  $10^{-7}$  Torr range. The use of this type of vacuum system assures high quality processing of neon tubes.

# Overcoming Lack of Communication When Designing Equipment

by  
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## INTRODUCTION

There are different forms of communication, including the common use of signs, written and speech. Through communication we can share our knowledge with others and by doing this we can all grow in understanding. Understanding can build our confidence and lead to the respect of our peers.

## BACKGROUND

I have worked for the Miami University Instrumentation Laboratory for the past fifteen years. The Instrumentation Lab is unique in that there is no charge for our services. People wanting custom glassware supply the materials and drawings and I fabricate it. Now I have to be careful not to let people take advantage of my service.

## TYPICAL SITUATION WITH GRADUATE STUDENTS

A graduate student came into the glass shop and wanted me to make a jacketed apparatus for him (figure 1). I asked why he designed it that way. He said he needed to cool the inner

Original Drawing

Thermospray  
Liquid/Liquid  
Extractor

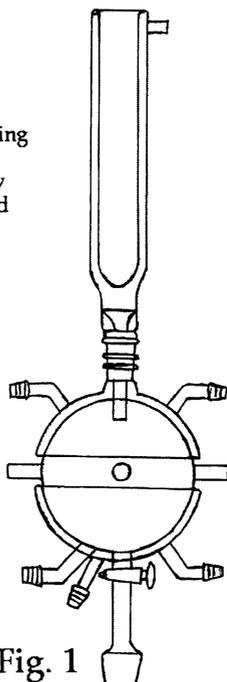


Fig. 1

flask and, at the same time, heat the probes as close to the inside flask as possible. I asked, "how big is the flask?" The student answered, "I don't know, about this big," as he held his hands out to approximate the size. At this point I asked what the student was trying to accomplish. "I can't tell you," he said, "because I'm trying to get a patent on the process." How can a client expect me to make something that will do what they want if we cannot discuss it? He said, "If this doesn't work, we'll make another one." I told him I did not want to fabricate this more than once, and instructed him to bring me the parts. I would then have some time to think about it.

## HUMOR

For some reason during our conversation, an amusing thought crossed my mind. I remembered Bill Cosby's record where he portrayed Noah. And God said to Noah to build an ark. Noah asked, "What's an ark?" And God said build it so many cubits long and so many cubits wide. Noah said, "Right. What's a cubit?" "What if," God said, "I can't tell you." Either Noah would be building his thousandth ark or he would have drowned in the flood.

## SOLUTION

During the next week while the grad student gathered up all his parts, I took the time to confer with a post doc working in the same research group. Using the information I obtained from the post doc, I was able to suggest changes to the original design to improve the effectiveness of the apparatus (figure 2). I cut tubing to use as spacers

### Final Drawing

### Thermospray Liquid/Liquid Extractor

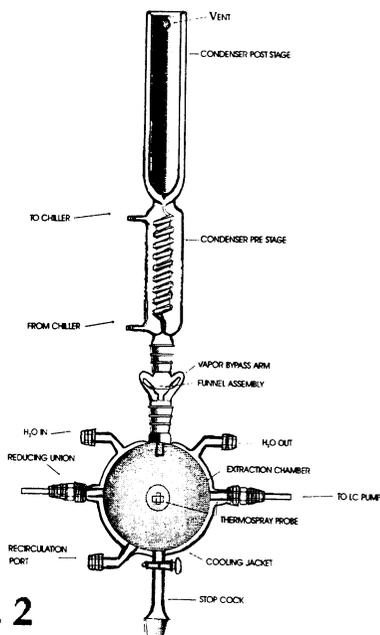


Fig. 2

between the inner and outer flask which reduced the exposed surface area. I also suggested using an adapter with a bypass, to go between the jacketed flask and the dewar condenser to make modifications easier. A spiral condenser had to be added to the dewar condenser later to prevent loss of product.

Many of the researchers I deal with in Botany, Zoology, Geology and Chemistry are having to use apparatus they are not familiar with. It is important for me to understand what they are trying to accomplish so I can better assist them in designing the equipment.

I knew from the first day I took the glassblower's position at Miami University that I would have to assist in the designing of equipment. Not being the mental genius some think I am, I knew it was going to take the help of others to do the designing. So, from the first day, I have shown an interest in what researchers were doing, and I am genuinely interested. Many of the professors and students now show me the equipment in operation and explain the process they are working with.

## CONCLUSION

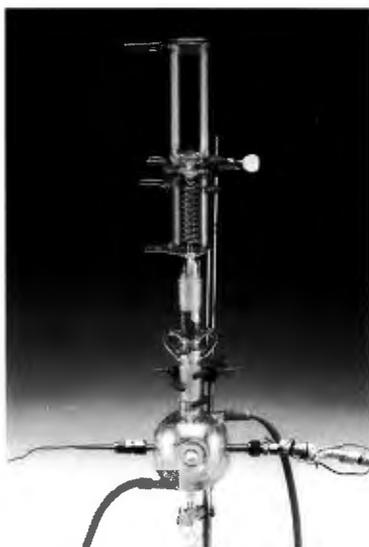
In most cases, it may be my lack of understanding rather than a lack of communication that gives me trouble when designing equipment. Having developed a good working relationship with the researchers, I am able to question their designs I do not understand with confidence. Some glassblowers do not care if it's going to work out or not, but I do, and the researchers learn this about me. At times, there are researchers who are not sure of what they need. Having developed a good working relationship with them, I feel comfortable discussing their designs with third parties.

## Acknowledgements

I am very grateful to the people of the Environmental Health and Safety Office at Miami University. Mr. David Michael Coons, Director, for editing this paper. Charles (Mike) Woodward for assisting with the drawing and Ms. Lisa Duckett for doing the typing. Thanks also to Virgil Sweeden, Photographer/Consultant, Audio Visual Service, Miami University.

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# Producing Side Seals at Various Angles Using Marias and Olives

by  
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Insertion Seal, Internal Seal, Ring Seal, Maria Seal, Olive Seal, Inner Seal, Triple Seal,<sup>1</sup> all refer to a type of seal where one piece of glass is sealed through the wall of another, most commonly at an end. The intricacy of this particular seal and the technique used to make it evolved over the years as a direct correlation of the development of research in the sciences and the need for elaborate experimental glassware. This paper will first review the development of the insertion seal and then provide details on a variation of it that I use and have found to work quite well.

Insertion seals have been described in many different ways. In 1898, in Glass Blowing and Working for Amateurs, Experimentalists & Technicians, Thomas Bolas affirms that the most common kind of internal seal was primarily used to make an air trap. Bolas prefers to free float his internal pieces, seal them into a round bottom, and then blow out the center; he uses the same method to make a condenser.<sup>2</sup> Bolas apparently had mastered his hand rotation skills as he makes no mention of any internal holding devices. Another early reference to this type of seal is by W. A. Shenstone in 1901. In his book The Methods of Glass Blowing, he describes the procedure as “To Seal a Tube inside a Larger Tube or Bulb”. To produce the seal, he uses a strip of writing paper, one inch in breadth, which he wraps around the insertion tube to support the loose end. He then heats the joint one spot at a time to achieve the finished seal.<sup>3</sup> In 1914, Francis C. Frary, Cyril S. Taylor and Junius David Edwards refer to insertion seals in their book Laboratory Glass Blowing as “Sealing a Tube through Another Tube”. Their method differs slightly and appears to be an attempt to simplify this advanced procedure for the less skilled glassblower. They recommend using a cork which they fit snugly in the end of the larger tube; they then bore a hole the same diameter as the inner tube through the center of the

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<sup>1</sup> The term most commonly used in early scientific glassblowing texts is Internal Seal (see Thomas Bolas, Glass Blowing and Working for Amateurs, Experimentalists & Technicians, London: Dawbarn & Ward, Limited, 1898, Bernard D. Bolas, Glassblowing Applied to Laboratory Work, London: George Toutledge & Sons, Ltd, 1910, H. P. Waran, Elements of Glass-Blowing, London: G. Bell & Sons Ltd, 1923, and M. C. Nokes, Modern Glass Working and Laboratory Technique, London: William Heinemann Ltd, 1937). Henri Vigreux refers to “une olive” [an Olive Seal] or “une soudure interne” [an Inner Seal] (Le Soufflage du verre dans les laboratoires scientifiques et industriels, Paris: Dunod, 1920). Wright uses the term Inner Seal as well as Triple Seal (R. H. Wright, Manual of Laboratory Glass-Blowing, New York: Chemical Publishing Co., Inc., 1943) and Julius D. Heldman prefers Maria Seal (Techniques of Glass Manipulation in Scientific Research, New York: Prentice-Hall, Inc., 1946). W. E. Barr and Victor J. Anhorn refer to a Ring Seal (Scientific and Industrial Glass Blowing and Laboratory Techniques, Pittsburgh: Instruments Publishing Co., 1949), and Edgar L. Wheeler uses Insertion Seal (Scientific Glassblowing, New York: Interscience Publishers, 1963). For the sake of simplicity, I will use the term insertion seal.

<sup>2</sup> Thomas Bolas 128.

<sup>3</sup> W. A. Shenstone, The Methods of Glass Blowing (London: Longmans, Green & Co., 1916) 43-46.

cork. Next they notch the full length of the outside of the cork in at least two places to allow hot air to pass out of the closed tube during the sealing process. A short piece of tubing, the same diameter as the inner tube, is passed through the cork which holds it snugly in place. Next they slip a piece of gum rubber tubing over the end of the stoppered glass tube; this becomes the holder. When the larger tube has been rounded at one end and the inner tube has been cut to the proper length, the inner tube is then slipped into the gum rubber tubing and the entire piece is inserted until the cork fits tightly. Once the seal has been made, the cork with the gum rubber tubing is pulled off, leaving the inner tube sealed to the rounded end.<sup>4</sup> The drawback of this method is that you blow only through the inner tube during the sealing process and not through the outer tube; as a result, the outer tube would collapse and could only be blown back into shape by putting the entire cork and tube in your mouth at once (this could be impractical if the tubing were larger than your mouth!). Recognizing the limitations of this method if the tube is large, Nokes suggests inserting a second tube through the cork which can be used to blow into the outside jacket.<sup>5</sup>

In all the previously mentioned techniques, the authors are in agreement on one thing: the inner tube needs a bulge of a slightly larger diameter for the seal to be completed (Photo 1). These bulges, which we call olives, are always hollow, and, in most cases,

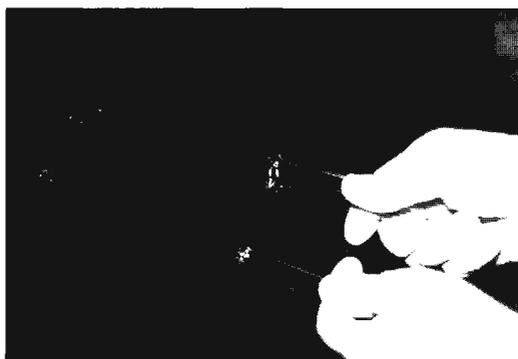


Photo 1. 8mm tube & 22mm tube with olive

blown to acquire their shape. Over the years, various mediums for supporting the inner tube have ranged from graphite rods to corks, corrugated cardboard, asbestos tape, aluminum foil, copper screen, and even writing paper. As glassblowers master hand control, they begin to free float the seals without any support at all and center the piece by eye as it cools while rotating or spinning it.

As chemical research grew, the demand for more intricate glassware expanded. The desire to have insertion seals on the side walls of flasks and tubes which are used as thermometer wells or feed throughs became the norm. These seals can be a glassblower's

<sup>4</sup> Francis C. Frary, Cyril S. Taylor & Junius David Edwards, Laboratory Glassblowing, (New York: McGraw-Hill Book Co. Inc, 1928) 52-55.

<sup>5</sup> Nokes 51.

nemesis if not made properly: a good insertion seal requires uniform wall thickness as well as uniform heating and cooling to give it strength and durability. If a portion of the bulge or olive is collapsed during the sealing, a stress point will be created at the seal, especially on a side wall. Moreover, this type of seal needs to be worked well and kept warm during the sealing-in process.

The next evolution came when someone decided they needed a side insertion seal with a slight angle so a liquid could be easily added. To produce this angle, one generally starts with the finished side insertion seal. The side wall glass is then gently heated in a wide area around the seal and the inner tube is then bent to the desired angle.

Our profession requires us to depend upon a certain number of very rudimentary seals or procedures. The basic seals that are now obvious to us originally were not. During the early learning stages in our profession, we are taught the basic seals to be used as building blocks. We need to recognize that there is a possibility that some of these seals could be made differently. The insertion seal is, in my opinion, a classic example of one such possibility. Through experimentation with 7740 and lower-melting glasses, I have discovered what I feel is a very simple method to achieve insertion seals. If you examine one of these seals that has been made by utilizing a blown bulge, you will notice that if the seal has been worked properly, that bulge collapses into a solid ring. If the desired end result is a solid ring as a part of the finished seal, then why not start with a solid bulge in the insertion tube. With this in mind, I decided one day that, instead of blowing a bulge, i.e. an olive, in the insertion tube, I would use a small, sharper flame and, while rotating the tube, slowly push the tube together firmly. I thus created a solid ring, i.e. a maria (Photo 2). I found it was extremely important that, when the glass starts to bulge, you

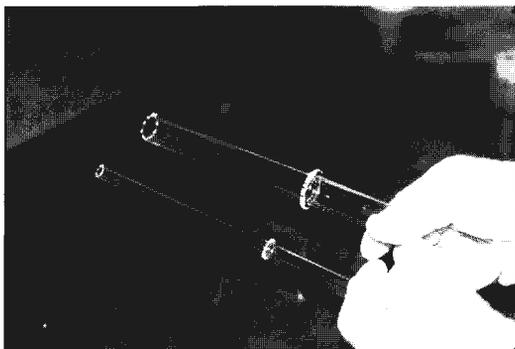


Photo 2. 8mm tube & 22mm tube with solid ring maria

must be sure that the glass is hot enough for it to seal together and be a solid ring. (If you are having a problem with this seal cracking, it could be caused by the ring not being fused together well enough.) Care should be taken to ensure that the bulge is sealed into a solid ring well. I have used this method for the last forty years with great success.

When making insertion seals in harder glasses such as Vycor® and Quartz, you should only use olive-type seals. The reason for this is quite simple. While working Quartz, you will notice that the heat is carried away very quickly and, in a lot of cases, will require

using heat on both the inner surface and outer surface at the same time. When you try to create a maria-type seal in these hard glasses, the Quartz or Vycor<sup>®</sup> does not get hot enough on the inside; a cold seal with a cleavage point is the result. When, on the other hand, you use an olive seal, the Quartz does not get hot enough to collapse and create the cold seal and cleavage point. Thus this technique allows you to seal small diameter Quartz tubes through side walls without collapsing them. The wonderful olive!

Once the concept of this technique is understood, it is possible to make minor changes in order to adapt it to different types of requirements. A side insertion seal at 45 degrees both inside and out is one such typical case. The way I was originally taught was to make a side insertion seal at 90 degrees; while it was still warm, a graphite rod was inserted in the tube through the seal. Then a wide area around the insertion seal on the larger tube was heated until pliable. At this time, the insertion seal with the graphite rod through it for support was bent until the desired angle was obtained. This procedure works well in this particular application (Photos 3, 4, 5 & 6). If this same type of seal were to be



Photo 3. Scribing one-liter flask



Photo 4. Round hole in one-liter flask

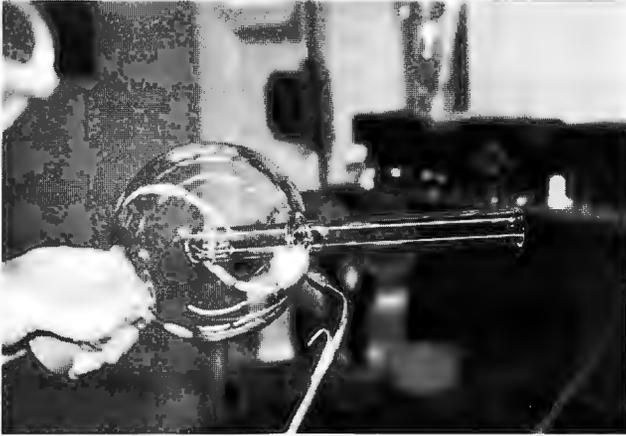


Photo 5. Spot heating insertion seal



Photo 6. Completed insertion seal

produced in the side of a one-liter flask, it would also work well. However, it would also form a platform around the seal, a platform on which any type of residue can collect and change the wall thickness on the side where it is stretched (Photos 7 & 8). In certain applications, this platform is not acceptable; consequently, this angle insertion seal needs to be made differently.



Photo 7. Insertion seal bent into place



Photo 8. Completed insertion seal

The technique that I use to make the insertion seal has a wide-ranging versatility because it eliminates the use of the supporting graphite rod and the development of the platform and thinner glass wall around the seal. The example chosen as a demonstration of this seal is a one-liter distillation flask with a 22mm tube inserted through the flask wall on an angle while still horizontal to the neck. The first step is to ascertain the angle required, in this case, 45 degrees. With a diamond stylus or a piece of glass cane, scribe a line at 45 degrees on the 22mm tube that will become the insertion seal (Photo 9). It is important



Photo 9. Marking 22mm tube at 45° angle

that the 45 degree line be scribed all the way around the tube so that when finished it appears elliptical. Start heating the 22mm tube on the scribed line with a small sharp flame and, following the line very carefully as you rotate the tube, apply a small amount of pressure end to end so that you produce a well-sealed solid bulge on the outside of the tube (Photo 10). You now have a maria at a 45 degree angle that is ready to be sealed into



Photo 10. 8mm tube & 22mm tube with 45° maria

your flask. To prepare the flask for the insertion seal, locate the spot where the seal is to be made, and mark it (Photo 11). With the new 45 degree angle insertion seal, an oblong hole is needed. This hole should be made by heating the flask and using a piece of cane to extract the excess hot glass, thus making the surface thin. An oblong hole can be made



Photo 11. Marking one-liter flask for 45° maria

by heating an oblong area or by pulling out the center and then reheating at one end of the hole and pulling out the excess. My personal preference is to heat an oblong hole. With a pair of calipers, measure the maria and then check to make sure that the hole is just large enough for the maria to fit into the hole. It is important that the glass around the edge of the hole be heated so that it is smooth, but also that it does not contain any excess weight (Photo 12). When the hole is ready for the seal, it should have the



Photo 12. Elliptical hole ready for sealing

appearance of a smooth slightly upraised oblong lip. Gently warm the hole and the maria (Photo 13). Insert the maria inside the hole, and with a sharp flame, heat one spot and



Photo 13. Warming 22mm insertion tube

touch the two together. Next, heat the opposite end of the seal, and with either a graphite rod or a tungsten pick, press the hot glass lip against the maria (Photo 14). Using a



Photo 14. Pressing hot glass lip on insertion seal

standard spot heating technique, work your way around the entire seal (Photo 15). I personally prefer to tack it all the way around and then, heating on the flask away from the maria, I work it until the glass flows and produces a beautiful smooth insertion seal at 45 degrees (Photo 16). This type of seal will have a large amount of stress; thus care should be taken to keep it warm enough to relieve some of the stress prior to the final annealing (Photo 17).



Photo 15. Spot heating technique



Photo 16. Completed 45° insertion seal



Photo 17. 45° Insertion Seals

The procedure of making the insertion seal has undergone several modifications over the years. It is interesting to note that in 1898 Bolas described a procedure for making a narrow ring by heating a tube with a small flame and gently compressing it until the ring was formed. He would make several of these rings very close together, then, with a larger flame, heat all of the rings and thus form a heavy-walled tube.<sup>6</sup> At the end of the nineteenth century, therefore, Bolas had pushed up the ring, but neither he nor others recognized that the ring could be used for an insertion seal instead of the blown bulge. The advantages of pushing up a solid ring for an insertion seal are numerous. It is expedient because none of the parts need to be blown or bent during the sealing process. It is stronger because it is solid and uniform in weight. And, most importantly, it provides the opportunity to create an angle of your choice. The basic techniques used by glassblowers have changed little in the past century, but over time, we can discover new and different uses for the techniques. The method of producing insertion seals described above has many applications and its versatility is only limited by you, the glassblower, and your imagination.

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<sup>6</sup>Thomas Bolas 98.

# A Short History of the Various Glass Apparatus Built for the Bose-Einstein Condensate Experiments by JILA

by

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**Abstract:** This paper outlines the evolution of the glass cells used in the first Bose-Einstein experiments conducted at JILA. Physicists Eric Cornell and Carl Wieman pioneered the technique of using small glass cells to create the first BEC. The approach has the distinct advantage of minimizing the number of devices housed in the vacuum chamber. Putting the various components comprising the optical and magnetic traps on the optical table and around a small cell not only makes achieving the necessary vacuum easier, but also allows for more rapid modification of the traps.

**Introduction:** In 1924, Indian physicist Satyendra Nath Bose enlisted the support of Albert Einstein for an idea he had concerning the nature of light. Einstein was taken by the idea, and expanded it to include atoms. The result, very simply expressed here, was the prediction of a new state of matter, which was to become known as a Bose-Einstein condensate (BEC). Sixty years later, experimentalists around the world began the first attempts to create what was later termed the “Holy Grail” of atomic physics. On June 5, 1995 at 10:54 in the morning MDT, in a dilute atomic vapor of rubidium cooled to 170 billionths of a degree above absolute zero, the first BEC appeared.<sup>1</sup> The team made images of the resulting “superatom “ after having cooled it further to 20 billionths of a degree above absolute zero, the lowest temperature ever achieved in a laboratory.

Judging from the spate of new papers, and they number in the hundreds, scientists involved in this new field of physics are growing in numbers and support. Appropriately, the small glass cell and its accompanying vacuum chamber are now housed among other national treasures in the Smithsonian’s collection of historic apparatus.

**Definition:** Bose-Einstein Condensate (BEC) is a form of matter as separate as liquid, solid and gas. Like other forms of matter, it forms when conditions are right. Simply put, BEC will form in a cloud of trapped atoms when they are cooled to the lowest quantum energy level. This puts the atoms in identical quantum states, thus rendering them indistinguishable from one another. In this way, many of them occupy the same space.

**Methods:** The JILA team formed the first BEC using two trapping methods. The first involves an array of six laser beams bombarding the rubidium atoms from all directions, driving them to the center of the cell. By carefully tuning the wavelength of the lasers, the photons only interact with the atoms moving toward them. This is due to the familiar Doppler shift. This resulting cloud of  $10^8$  atoms is 10 millionths of a degree above

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<sup>1</sup> M.H. Anderson, J.R. Ensher, M.R. Mathews, C.E. Wieman, and E.A. Cornell, "Observation of Bose-Einstein Condensation in a Dilute Atomic Vapor" *Science* 198 (1995): 296.

absolute zero, still too hot to form BEC. The lasers are turned off and the atoms are transferred into a purely magnetic trap. The atoms roll around in the trap much like marbles in a bowl. By creating a small hole in the bowl near the top, the atoms with the highest energies are allowed to escape. This is what is known as evaporative cooling; it is much the same phenomenon observed in steam leaving a hot cup of tea, taking some of the heat with it. In the simplest kind of magnetic trap there is a hole in the bottom as well. But with nothing to prevent the coldest atoms from falling out, there were few atoms left to form a condensate. The last breakthrough came with Eric Cornell's development of the time-averaged orbiting potential trap, a modification enabling the hole in the trap to move around faster than the atoms could respond.<sup>2</sup>

**Glass Cells:** The first glass cell made for the original apparatus was a 28mm square cell described in the 1994 ASGS Symposium *Proceedings*.<sup>3</sup> The trick was to fabricate a square cell with minimal optical distortion. Figure 1 shows the first BEC cell.

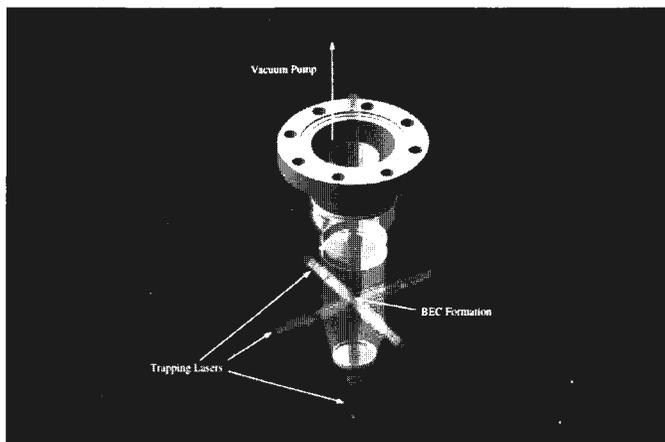


Figure 1

After the first success and some subsequent experiments, new magnetic coil designs necessitated different glass cell configurations. These gave rise to the next generation of BEC cells based on a six-way cross. The scheme changed, as well, employing a two MOT (Magnetic-Optical Trap) system. The first MOT is used as a place to introduce and trap large numbers of atoms. From this trap, another laser, carefully tuned to atomic resonance, pushes a cloud of atoms down a delivery tube and into the second MOT, hereafter referred to as the "science cell" (fig. 2). This process is repeated until the

<sup>2</sup> Joint Release by The National Institute Of Standards And Technology and the University of Colorado on 7/13/95.

<sup>3</sup> Hans Rohner, "A Borosilicate Square Cell With Optical Quality Surfaces for an Ultra-High Vacuum Rubidium Atom Trap" *Proceedings of the Thirty-Ninth Symposium on the Art of Scientific Glassblowing* (Pittsburgh, PA, 1994): 57-66.

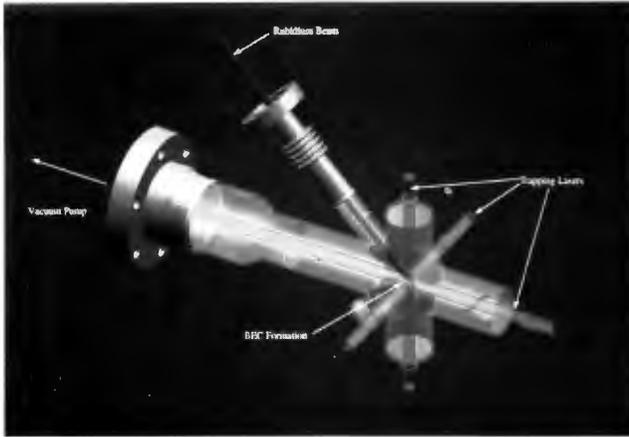


Figure 2

science cell is loaded with enough atoms to begin the evaporative cooling. This second-generation scheme used a first MOT made of stainless steel with glass window flanges. With this apparatus, researchers were able to create BEC with two million atoms, quite an improvement over the two thousand atoms left after the evaporative cooling in the original experiment.

This second phase scheme still used as its alkali metal source an ampoule crushed *in vacuo*, as in the first experiment. Heating the area containing the crushed ampoule provided the necessary background pressure of rubidium.

The third generation apparatus includes a first MOT made entirely of glass (fig 3). This development further reduces the need for more expensive and cumbersome stainless

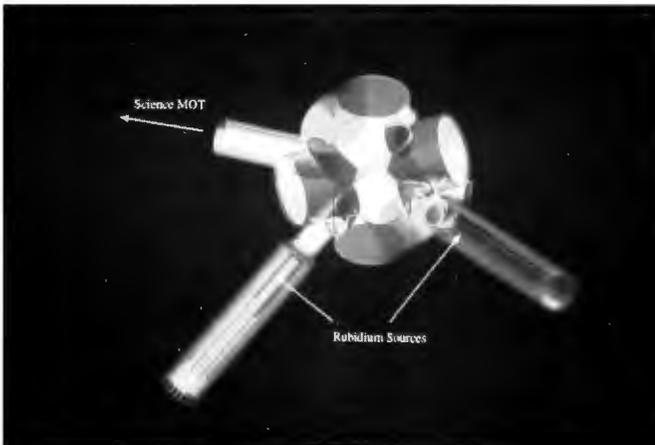


Figure 3

steel construction, while allowing for commercially available getter type metal sources. These are spot welded to leads from pin presses attached to additional side arms. These particular pin presses are not commercially available, and glassblowers should be

prepared to make them themselves. With the growing confidence in glass and its properties conducive to their goals, scientists were loath to sacrifice the elegance and cleanliness of glass and the flexibility of design it afforded them. This design helped further the automation of BEC in the lab, where researchers are able to form condensates at the rate of about three per minute. The entire apparatus can be vacuum baked at 350°C.

The third generation science cells differ in that they have more small side arm ports, used for imaging and alignment (fig. 4). The glassblower can modify the configuration, depending upon the required coil design.

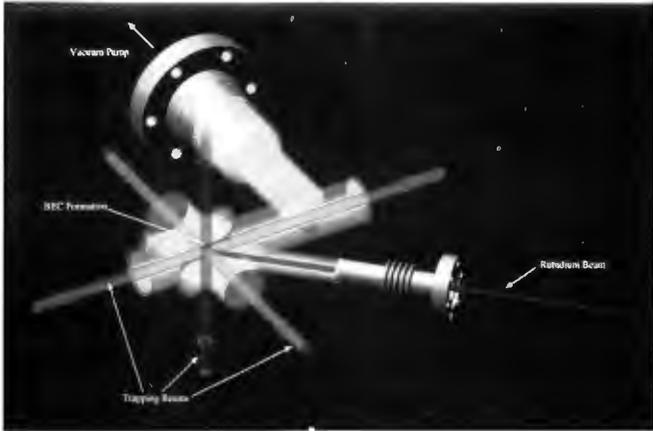


Figure 4

Another variation of the third generation apparatus is the one designed for the first attempt to create a quantum degenerate gas of the half-spin counterpart of the boson, the fermion. The first MOT (fig.5) shows the scheme. This trap has three different species

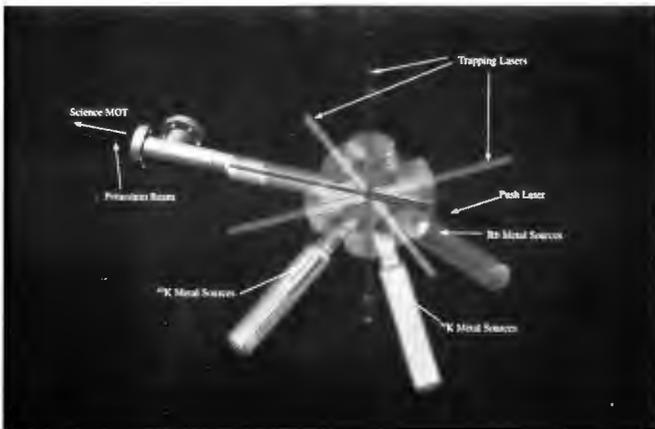


Figure 5

of getter type metal sources. Two of them are commercially available rubidium and potassium sources. The third is an in-house version of an enriched  $^{40}\text{K}$  source.  $^{40}\text{K}$ , a fermion, is a naturally occurring isotope, however rare. Its normal abundance in a typical sample is 0.01%. Obviously, any increase in the number of  $^{40}\text{K}$  atoms would be a welcome advantage. An enriched sample having a  $^{40}\text{K}$  abundance of 4.5% is available in the form of potassium chloride ( $\text{KCl}$ ). The source consists of calcium filings mixed with the enriched potassium chloride sample loaded into a nichrome boat. Calcium gives the chlorine atom something to bond with (due to calcium's greater affinity for chlorine) when the mixture is heated. This leaves the enriched potassium liberated to the confines of the trap.

Photo #1 shows the actual first MOT before it had its pin presses and source assemblies in place. For scale, the large windows in this MOT are typically 38mm in diameter.



Photo 1

The configuration of the science cell for this apparatus is also different, although it is based on the same six-way cross (fig. 6). The actual cells themselves (photo #2), show

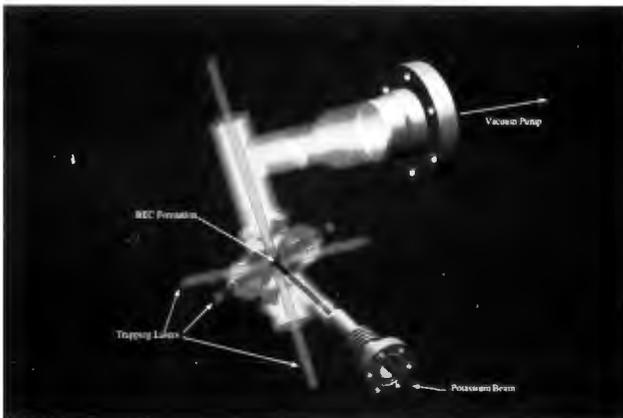


Figure 6

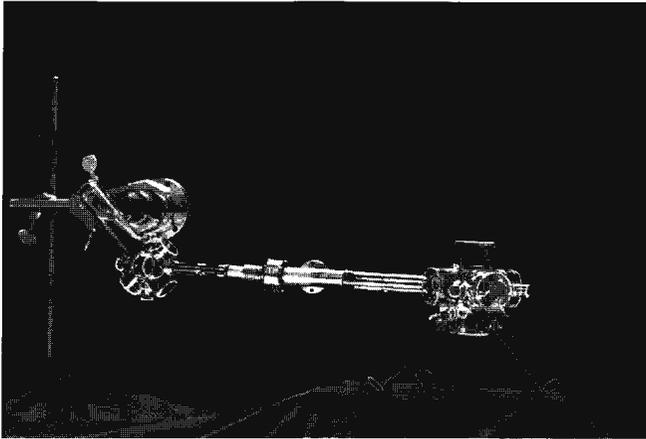


Photo 2

the basic configuration without the stainless steel delivery tube, which goes in between the two MOTs. The science cell typically uses 25mm diameter windows. The small windows on both cells are 13mm diameter; all windows are 1.5mm thick. The glassblower must take care to maintain the optical surfaces of the windows throughout fabrication. This is not a trivial undertaking, and planning the steps of the process greatly increases the chances for success.

The glass used for all of the apparatus thus far is Corning's 7740 (Pyrex). All the glass/metal seals are of the Housekeeper variety. The pin presses are five .040" diameter tungsten rods sealed directly to Pyrex. These have proven themselves quite robust, having survived repeated vacuum bake-outs to 350°C.

**Summary:** Scientific glassblowing has its roots in many traditions, far and away the largest being the field of chemistry. Glass was the material of choice for a large number of scientists in the 19th century. The sorts of apparatus described above arise from another tradition: that of the vacuum tube. The Special Techniques Lab at JILA, formerly called the Tube Lab, is a part of this tradition. Many of the problems facing experimentalists today can be reduced to the ancient question of materials. Leonardo designed flying machines in the early 16th century that look not too different from modern day hang-gliders, but failed because he lacked the appropriate materials. Glass may have been the material of choice back in the 19th century when metals, ceramics and plastics had yet to come into their own. The 20th century has seen the rise of materials science, bringing with it new materials of every sort. While this has led to the dilution of glass as material of choice, it has by no means diminished its importance, prompting Eric Cornell, in whose lab the first BEC was ever created, to predict that "as glass was the material of choice in the 19th century, it will surely reclaim that status in the 21st century."

The BEC apparatus at JILA come out of recycled techniques and technology of the vacuum tube tradition. The unique qualities of glass made possible the ease and flexibility with which the scientists could try new configurations to match new paths of inquiry. Dr. Paul Forman, Curator for modern physics at the Smithsonian, surely

recognized the unique importance of the roll of glass in the first apparatus. In a letter to Carl Wieman dated August 1, 1995 requesting whatever components could be set aside for the permanent collection, Forman wrote, "...I would be grateful if you'd consider now whether, for instance, the glass cell in which you first obtained BEC can now be identified and set aside, along with any ancillary items associated with this success, and whether you could consider tagging other components of the apparatus as intended for the Smithsonian...." This is an acknowledgment of which we can all be proud.

As we stand at the threshold of a new millennium peering through the portal to a new age, we enjoy a view made sharper and brighter through glass. Glass, one of the first viable materials of scientific endeavor, at once occupies a unique position in the historic annals of discovery, while at the same time augmenting the spectrum of modern research materials.

**Acknowledgments:** I would like to thank Brian Demarco and Deborah Jin for their helpful discussions, Eric Cornell and Carl Wieman for the original reason to embark on such endeavors, and Seth Wieman for his help with the graphics.

### Notes

<sup>1</sup>M.H. Anderson, J.R. Ensher, M.R. Mathews, C.E. Wieman, and E.A. Cornell, "Observation of Bose-Einstein Condensation in a Dilute Atomic Vapor," *Science* 198 (1995): 296.

<sup>2</sup>Joint Release By The National Institute Of Standards And Technology And The University Of Colorado on 7/13/95

<sup>3</sup>Hans Rohner, "A Borosilicate Square Cell with Optical Quality Surfaces for an Ultra-High Vacuum Rubidium Atom Trap," *Proceedings of the Thirty-Ninth Symposium on the Art of Glassblowing*, (Pittsburgh, PA, 1994): 57-66.

# The Way I Build a Jacketed 3-Liter Reactor

by  
**Timothy A. Drier**  
**The Dow Chemical Company**  
**Midland, Michigan 48667**

My name is Timothy A. Drier, and I work for the Dow Chemical Company in Midland, Michigan. First a little background on the 3-Liter Reactor that I am going to be building: I will be using 150mm od tubing for the inside pot, 180mm od tubing for the jacket, and 150mm Schott flanges for the top of the reactor and the lid. I will be using 1/2" beaded pressure pipe for the outlets of the jacket. The bottom drain valve is a 1" flush bottom that opens fully with a 1/2 turn, giving the ability to drain the reactor very quickly if needed. Since it is unique to Dow Chemical Company, I am not at liberty to go into depth on the construction of this valve.

I am working on a Litton KTB-197 model lathe with 10" bore and planetary chuck. My burners are Carlisle "CC" burners and a 6-tipped uni-torch on the large ring seal. Our natural gas is at 10 lbs. psi and our oxygen is at 15-18 lbs. psi.



Valve seat prepared for sealing to bottom of reactor.



Sealing valve seat to bottom of reactor.



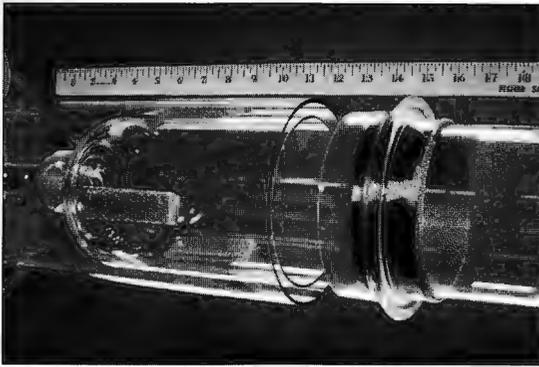
Pulling open valve seat body to desired inside jacket length.



Valve seat completed - ready to fit inside reactor dimension.



With fire proof tape pads, positioning reactor body inside jacket.



150mm O-ring flange positioned for top half of reactor assembly.



Bringing outer jacket to inside reactor lip.



Proceeding to seal 150mm O-ring flange to top of reactor and jacket.



Upper ring seal completed.



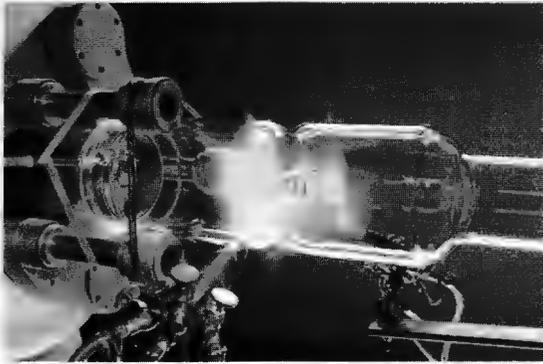
Sealing first jacket outlet: 1/2" beaded pressure pipe connection.



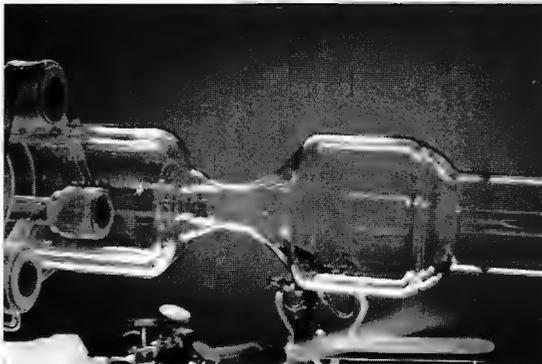
Pulling fireproof tape centering pads from between reactor and jacket.



After annealing, proceeding with setup for bottom-half completion of reactor.



Shaping outer jacket to valve seat lip.



Proceeding to pull off excess jacket body from reactor.



Preparing bottom valve opening for rest of valve crank assembly.



Attaching valve crank assembly to reactor.



Attaching second outlet: 1/2" beaded pressure pipe connection.



Preparing lid of reactor for main center joint.



Attaching 45/50 joint to lid of reactor.



Completed lid for reactor



Completed and Assembled Reactor.

In conclusion, we probably build 20-30 of these reactors a year in various sizes from 500ml up to 5 liter. As the title says, this is the way I build this reactor. I'm not saying it is the best way or the only way, it is one approach. In our shop alone, we have four different glassblowers and four different styles. Hopefully you picked up something from this presentation that you can take back and use.

I would also like to say I would not be as comfortable building glassware of this magnitude without a great deal of coaching and influences from the wide range of glassblowers I have had the privilege of working with and meeting through the American Scientific Glassblowers Society.

# Posters

# Atomic Force Microscopy Scans of Glass Surfaces Used in Polarization Cells

by  
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Princeton, NJ 08544-1009

## Introduction

Polarization cells are glass envelopes that contain highly magnetic gases. The procedures for making this gas have been well documented.<sup>1,2</sup> However, these cells have two criteria in determining their quality. First, one must measure the **percentage of the gas that is polarized**, and secondly, **the amount of time the cell can sustain polarization of the gas or T1**.

Experience in this field has shown that there is almost always a variance in the quality of cells produced. Since the fabrication of these cells are highly sophisticated, there are ample opportunities for these variances to occur.

One of the more obvious places to investigate as to how these anomalies occur would be the inner surface of the cell where gas interacts with the glass.

Atomic Force Microscopy (AFM) scans present an ideal image of the glass surface. Through the use of ARM, one can see exactly what a spinning nucleus sees.

## Polarization Cell



<sup>1</sup> W. Happer, F. Miron, et al, "Polarization of the Nuclear Spins of Noble Gas Atoms by Spin-Exchange with Optically Pumped Alkali Metal Atoms," *Phys. Rev.* 29, Suppl A (1984): 3092-3110.

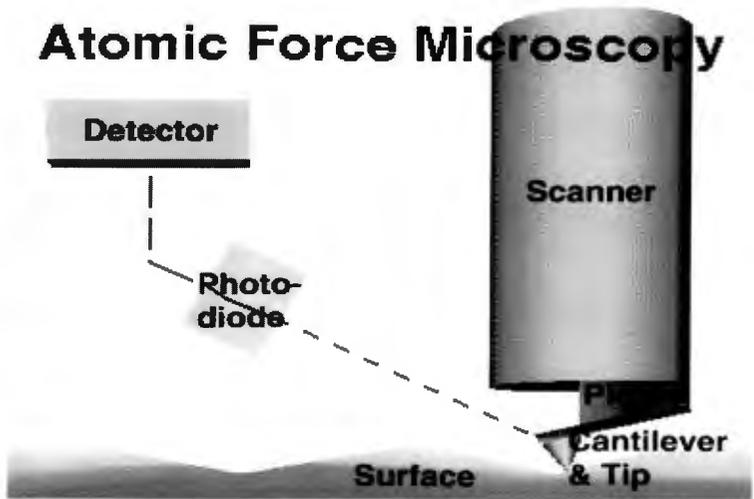
<sup>2</sup> H. Middleton, R.D. Black, B. Saam, et al, "MR Imaging with Hyperpolarized <sup>3</sup>He Gas," *Magn. Reson. Med.* 33 (1995): 271-275.

The Polarization cell as depicted contains approximately 10 atms. pressure (150 psi) of  $^3\text{He}$ .

The windows are extremely thin (2 mils or 0.05mm) and inverted to withstand high pressure. The cell is entirely made of "reblown" Corning 1720 aluminosilicate glass.

The silver appearing tip-off at the top of the cell denotes the presence of Rubidium.

Under this view, a Polariscope gives an indication as to the amount of stress the glass cell is under and also registers where the extreme strain points are. This cell is further pushed to higher pressure as it is heated to nearly 230c and the Rubidium transforms into a corrosive alkali vapor.



### Atomic Force Microscopy

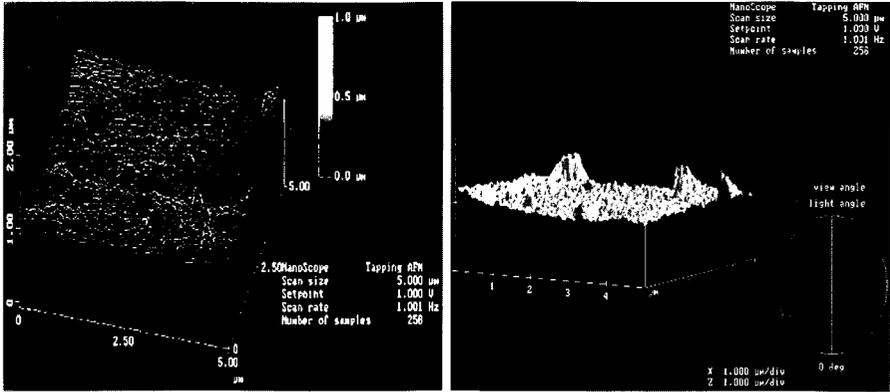
Atomic force microscopy (AFM) is an important new analytical technique which is utilized to study the surface features of material with a resolution down to the atomic level. **In comparison to traditional microscopes which use electron or photon beams to create images, an AFM uses a mechanical probe** which uses various surface properties (e.g. topography, friction, hardness, etc.) to generate an image.

AFM combines an ease of use with unparalleled ability to image surface features, and to do so **without extensive sample preparation and under ambient conditions.**

**Atomic force microscopy can magnify surface features by as much as x100,000,000. As a result, single atoms and molecules on surfaces are directly observable.**

**Atomic Force Microscopy** utilizes a sharp probe to scan across the surface of a sample. A laser is focused on the tip and the beam is reflected to the split photodiode detector. As the cantilever is deflected over the sample surface, the photodiode monitors the changes of the laser beam. The changes are recorded in a computer and are used to produce a topographic image of the surface.

### AFM Plot Charts



These two images are both the same sample. The left contains data displayed as a series of cross sections in an oblique view, with color coded height information.

The image on the right transforms the data into a three-dimensional topographical image.

### Reblown Glass Surfaces are Optimal for Polarization Cells

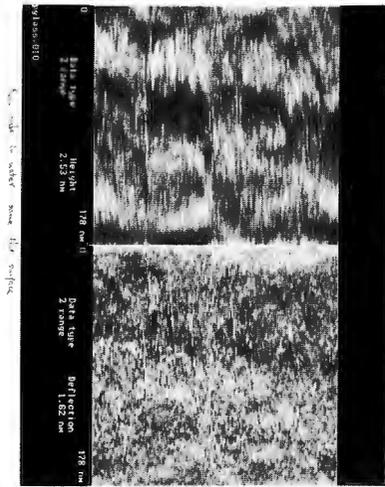
As researchers in Princeton's Physics Department accumulated polarization cells of various designs, intuition led them to believe that glass that was "worked or reblown" may lend a better surface for polarized atoms. **Since these atoms depend on spin for their alignment, it was felt that the smoother the surface, the better.** A decision was made to resize or reblow all glass tubing to required sizes as part of fabricating cells. As a consequence, there was a significant improvement in the lifetime of all our cells.

To gain a better understanding of this phenomena, a comparison test was undertaken by the glass shop which utilized Atomic Force Microscopy for an analysis between reblown glass tubing and manufactured raw tubing. To accomplish this, a sample tube of 15mm od 1720 aluminosilicate glass was used. Half of the tube was blown to a 21mm od and the remainder was left unaffected. Specimens from each section were cut away from the tube and given for AFM scans.

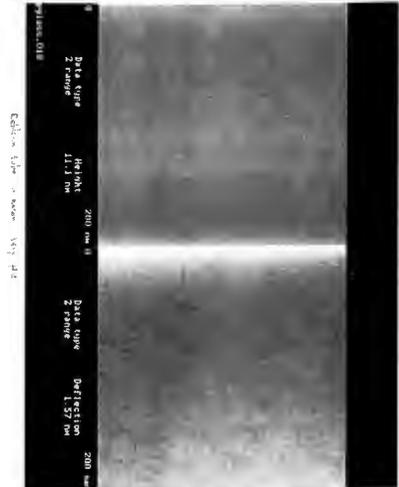
**The results as shown clearly depict that the raw tubing appears to have a rougher surface than does the reblown section of the tube.** A likely source for the contrast may

have to do somewhat with the manufacturing process of drawing tubing. In addition, it should be pointed out that almost all of our 1720 aluminosilicate tubing is at least 25 to 30 years old and as such may have accumulated flaws over that period of time.

## Raw Glass Tubing



## Reblown Tubing



### Observations in Cell Material

An additional comparison test was done on a used polarization cell (*identical to the one pictured in the polariscope slide*). The cell material studied came from one of a dozen <sup>3</sup>He Polarization Cells<sup>3</sup> that had been used as nuclear targets for high energy experiments conducted at CLAC (Stanford Linear Accelerator).

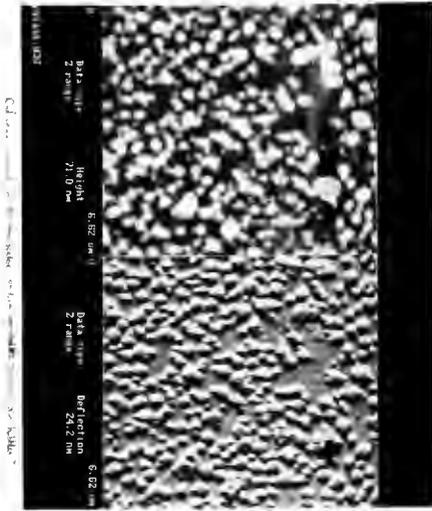
During this experiment, the cell is polarized and enormous amounts of energy are scattered through the thin windows. Under these conditions, the cells start to turn brown in a matter of hours.

The most likely source for this occurrence was thought to be the effects of millions of volts being passed through the cell. However, when one observes the AFM scan of the inverted thin window where the scattering occurs and compares it to the scan of the cell body, it is obvious something else is influencing the breakdown of the glass.

A prime suspect is pressure and Rubidium. Under extreme pressure conditions, glass will "flex" open and will fatigue as it collects flaws. (As a criterion for the experiment, these cells would achieve pressures of nearly 200psi.) Conversely, the window was inverted and as such it was under compression. Thus, we now suspect that 1720 aluminosilicate glass may not be as resistant to Rb as previously thought, especially under conditions as rigorous as what these cells must undertake.

<sup>3</sup> Michael J. Souza, "Super Thin Windows for High Density <sup>3</sup>He Target Cells," *Fusion* 42 (November 1995): 22-27.

## Cell Body



## Cell Window



### Conclusion

- Atomic Force Microscopy is an invaluable analytical tool for glass.
- It requires very little preparation.
- Glass is an ideal material for this type of microscopy.
- More investigation into the effects of pressure should be carried out.
- AFM comparisons on cells with high percentages of polarization and long lifetimes (T1's) versus ones that do not meet standards should be done.

The results of this study are still inconclusive. We still have very little insight as to what causes cells to vary in quality. This is despite the fact that every procedure and process that produces good cells are recorded, re-enacted and carried through the fabrication process.

The goal in producing high-quality polarization cells is extremely important. The difference between 30% polarization and 40% is almost exponential in experiments and lifetimes of the cells need to be extended as long as possible.

### Acknowledgments:

- Digital Incorporated, Division of Material Science for their sample graphics
- Princeton Materials Institute at Princeton University
- Princeton University Department of Chemistry

# **An Economical and Easy Way to Build A Thin-layer Spectroelectrochemical Cell**

**by**  
**Randolph B. Wilkin**  
**University of Houston**  
**Chemistry Department**  
**Houston, TX 77204-5641**

A vacuum-tight all-glass thin-layer cell with a platinum working electrode was constructed for spectroelectrochemical studies. The cell body can be made from borosilicate glass for studies in the UV-visible region or from quartz for studies in the ultraviolet and visible regions. The rectangular thin-layer chamber forms a sandwich configuration with platinum gauze inserted. The chamber is open to the bulk solution at all four edges and has a thickness between 0.1 mm to 0.3 mm depending on the thickness of the platinum gauze used.

The cell is directly manufactured from a borosilicate or quartz tube of 20 mm in diameter. A piece of stainless steel or graphite model is formed in the shape of a bathtub (bottom: 1.0 x 0.5 cm) and is placed inside and supported by a graphite block underneath. The tube is sealed at one end and connected to an ordinary lab vacuum (15 mm Hg). The glass is slowly melted with a hand torch to press tightly against the model. This forms the one side of the cell window. The form is then withdrawn and a piece of graphite tape is cut 2.0 x 1.0 cm and placed on the top of the inside window. The same technique is used to collapse the other side of the glass tube to form the sandwiched jacket. The cell path length is thus determined by the thickness of the graphite tape used. The Pt working and auxiliary electrodes are connected to the cell body with cobalt-glass for borosilicate or Epoxy glue for quartz.

This design of a spectroelectrochemical cell has the advantages of minimum IR drops, durable mechanical strength, good experimental reproducibility, and is free of contamination.

This work was done for and in conjunction with the research of Dr. Xiangqin Lin, Dr. Xiang Gao and Professor Karl Kadish.

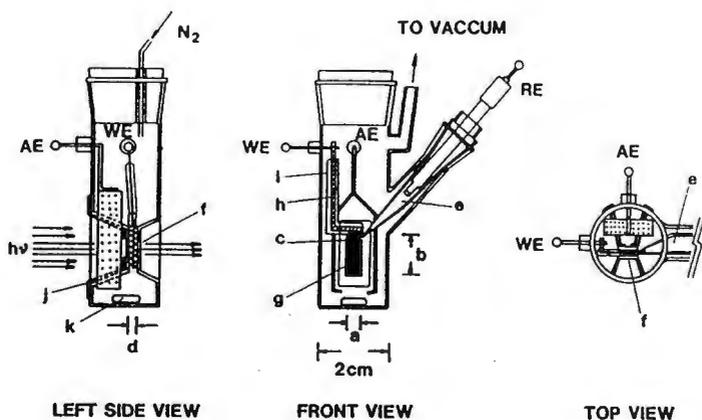
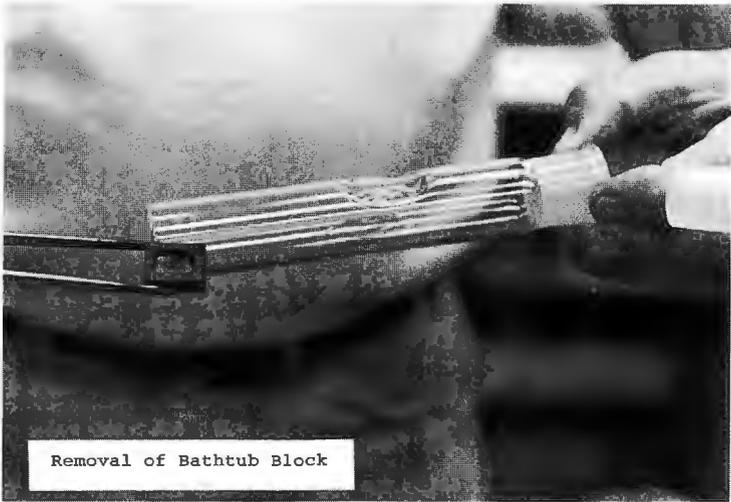
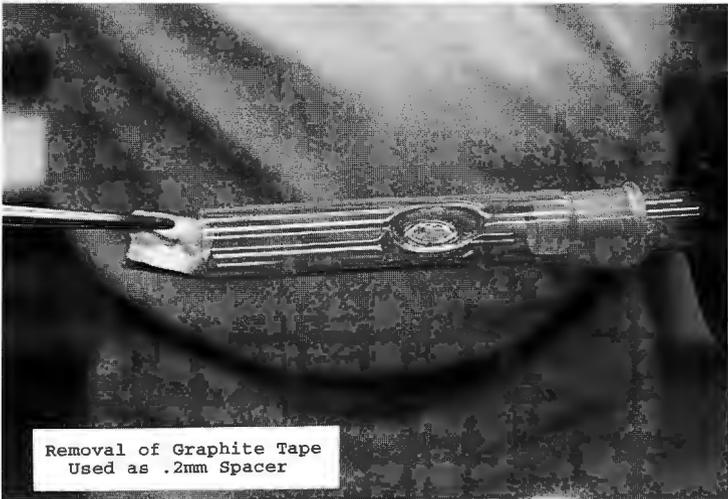


Figure 1. Schematic illustration of the vacuum-tight thin-layer spectroelectrochemical cell with a doublet platinum gauze working electrode. The front view shows the vacuum operation while the side view shows the cell with regular nitrogen deoxygenation. The frit arm is not shown in the left side view. Parts of the cell are as follows: (a) width and (b) length of gauze working electrode; (c) thin-layer chamber; (d) thickness of the thin-layer chamber; (e) glass frit (Pt tipped for vacuum operation, asbestos tipped for regular operation); (f) photo window; (g) gauze working electrode; (h) stainless steel foil; (i) Teflon film protection; (j) light shelter for photo window; (k) magnetic stirrer.





Removal of Bathtub Block



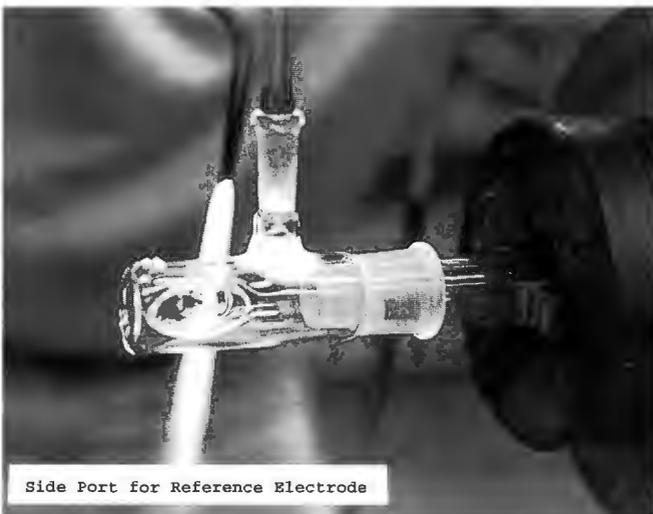
Removal of Graphite Tape  
Used as .2mm Spacer



Bathtub Block



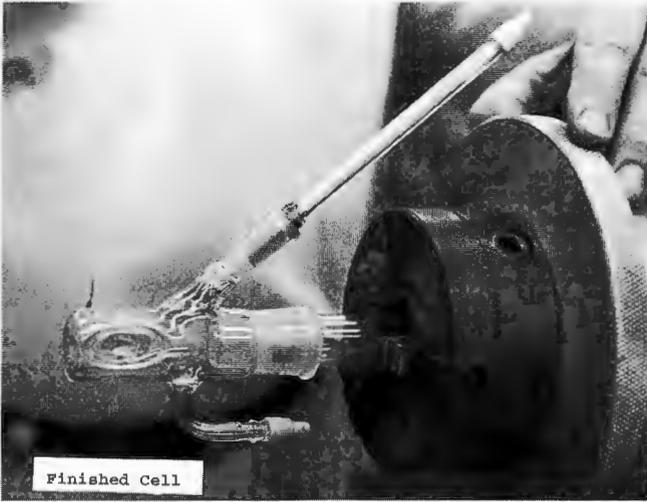
Auxiliary Electrode



Side Port for Reference Electrode



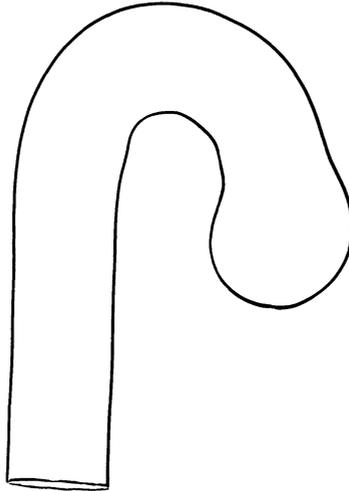
Vacuum Connection



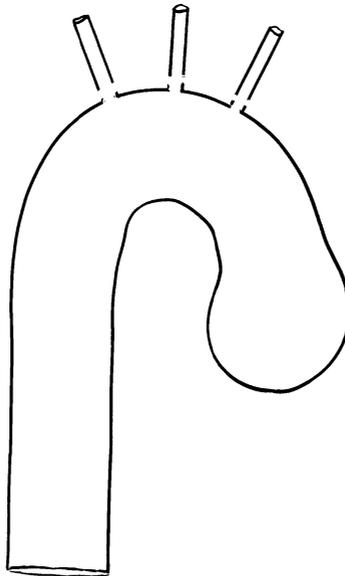
# Arterial-Vascua System

by  
Hans A. Florell  
University of Minnesota  
Minneapolis, Minnesota 55455

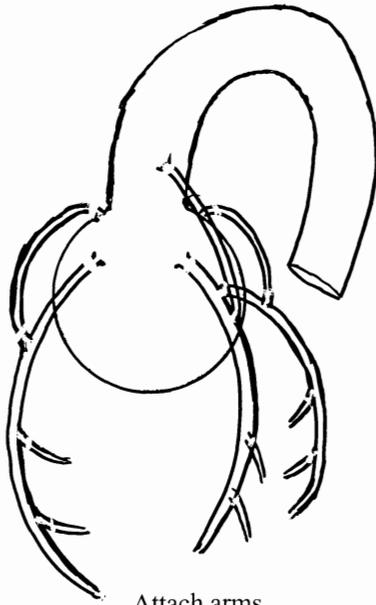
The Arterial-Vascua System is used for training of the insertion of catheters for blockage and stents.



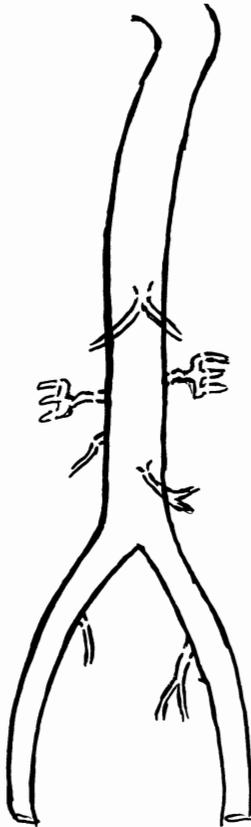
Bend and blow tube as shown.



Attach arms.



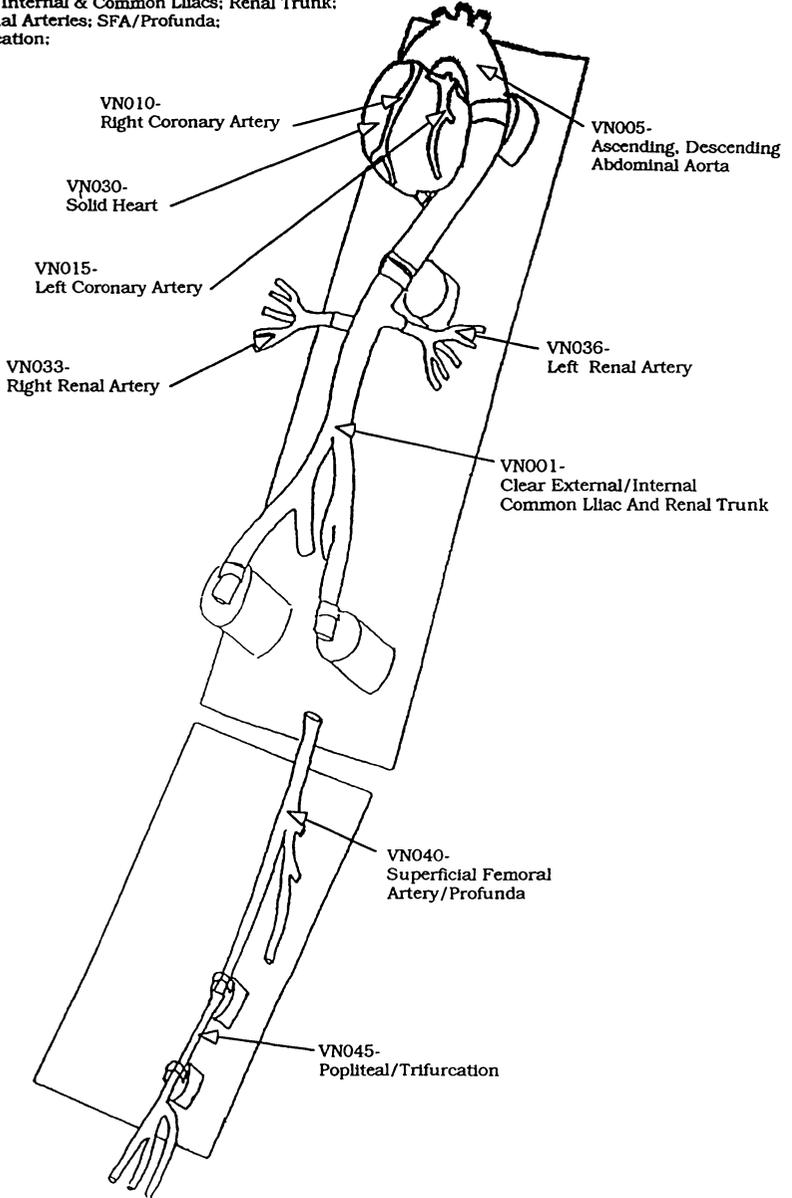
Attach arms.



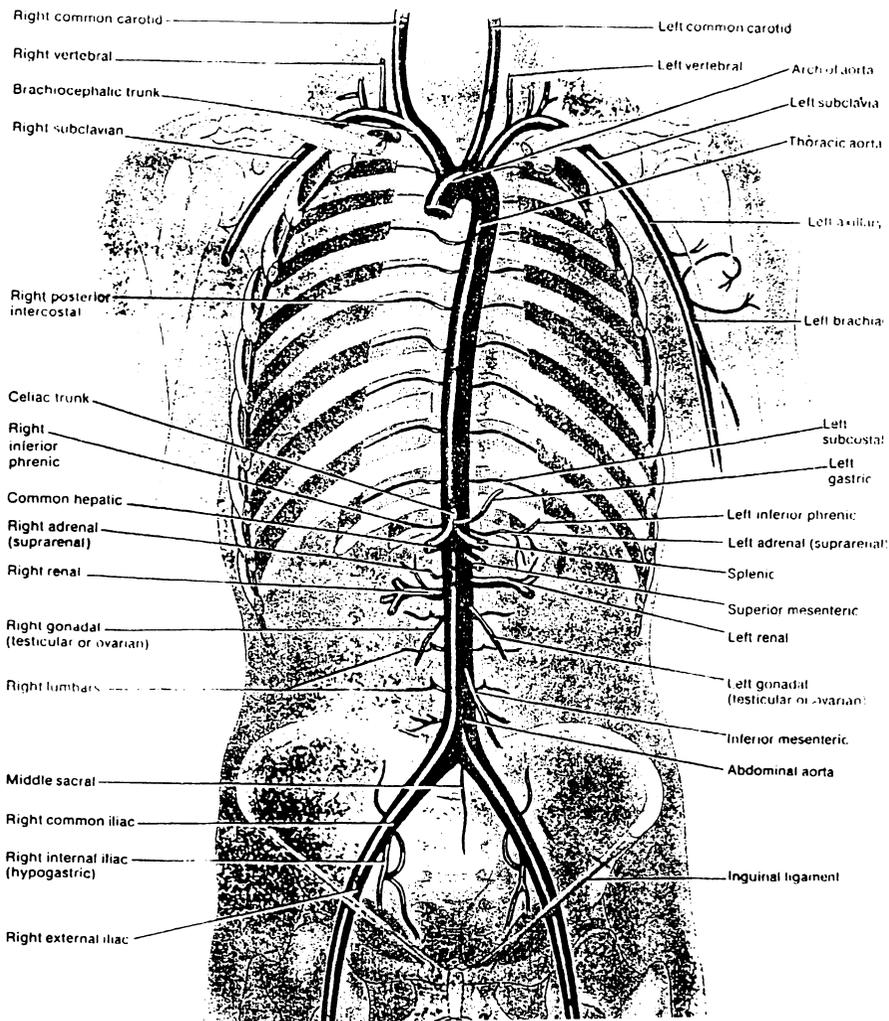
Attach arms.

**ARTERIAL SYSTEM**

Heart, Right Coronary Artery,  
Left Coronary Tree, Ascending, Descending & Abdominal  
Aorta; External, Internal & Common Iliacs; Renal Trunk;  
Right & Left Renal Arteries; SFA/Profunda;  
Popliteal/Trifurcation;



# Adult Anatomy & Physiology



# Flat Bottom Quartz Dewar For Use As A High Temperature Camera Housing

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

A 1" x 1/8" quartz disc is sealed to a Pyrex tube of desired size with Apiezon black wax. A disk of 18mm and 25mm od was needed. The disc is ground to size on a wet belt sander using a 400 grit diamond belt.



A vacuum is used to hold the disc on a 12mm od Quartz tube. The inner jaws of a Litton 6-jaw chuck are used to hold the vacuum chuck. The outer jaws are used to chuck the 20mm x 18mm tubing in the head stock. A scrollchuck is used to secure the tubing in the tail stock.



With the disc positioned in the proper location, it can now be sealed. The small diameter of the tubing allows one the option of using gas or hydrogen to seal the disc. A sharp flame is focused on the disc and the tubing is tooled down onto the disc. As soon as contact is made with the disc, remove the vacuum chuck and continue to tool down the tubing until the ground edge of the disc appears clear. The disc is now sealed.



After the tube is cooled, the excess tubing is cut off with a wet cut off saw. Take care not to cut too close to the disc and scar the surface. Any excess tubing can be ground off with a wet belt sander. Clean the tube and allow it to air dry. This step is repeated after the second disc is sealed.



Corrugated cardboard is used to hold the inner tube, 20mm x 18mm, on center of the outer tube, 28mm x 25mm. The inner tube is tooled up to make the dewar seal, so allow the extra length for this. Work the seal in, making sure to maintain the inner and outer diameters of the tubing.



The evacuation port, 4mm od, is sealed to the top of the dewar seal and should not interfere with the inner or outer diameters of the dewar. The 4mm tubing is very fragile and care must be taken in the remaining steps not to break it. After the dewar has cooled, carefully remove the cardboard and thoroughly clean the dewar. Allow to air dry.





The second disc is sealed in the same manner as the first. The overall length of the dewar is critical so this disc is positioned to provide the proper finished dimension. The disc is sealed, the dewar allowed to cool, and the excess tubing cut off as in step four. After cleaning and air drying, the dewar is ready for evacuation.



The dewar is sealed to a vacuum system which has an outlet in the annealing oven. A graded seal is needed to seal the quartz dewar to the Pyrex manifold.



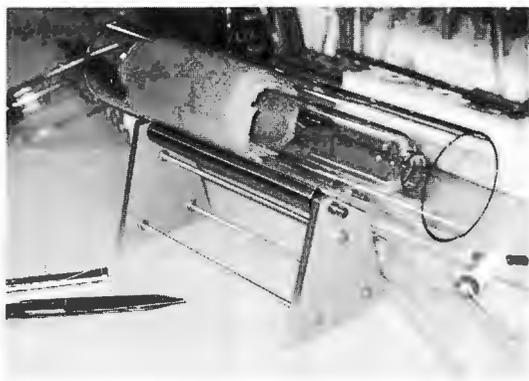
Once the dewar is sealed, the roughing pump is turned on and after a few minutes the dewar is checked for leaks with a Tesla coil. With no leaks, continue to rough pump. At approximately  $10^{-3}$  torr, the diffusion pump is engaged and the dewar is allowed to bake out at  $565^{\circ}\text{C}$ . Optimal vacuum for this particular set up has been  $5 \times 10^{-7}$  torr. Once this pressure is reached, continue to pump for several hours. Now the dewar is allowed to cool to around  $300^{\circ}\text{C}$  and sealed off as close to the dewar seal as possible.



*Photographs by Patrick Westover*

# Jacketed Dropping Funnel with Pressure Equalizing Arm

by  
Steven H. Anderson  
University of Nebraska/Lincoln  
Lincoln, NE 68588-0304



Inner reservoir is wrapped with packing paper until held securely inside the 75mm outer jacket.



Outer jacket is carboned down onto the 28mm end of inner reservoir.



24/40 outer joint is sealed into place.



A hole is blown in outer jacket and a side arm is attached.



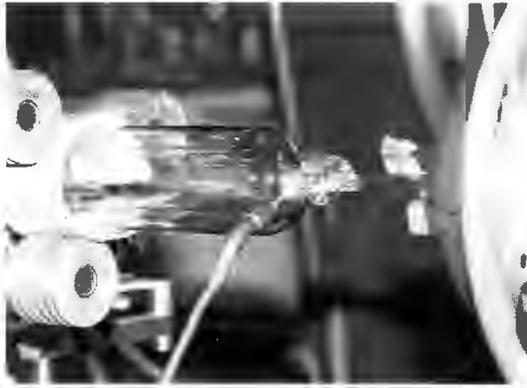
The upper portion of the pressure equalizing arm is added...



and bent to a 90 degree angle.



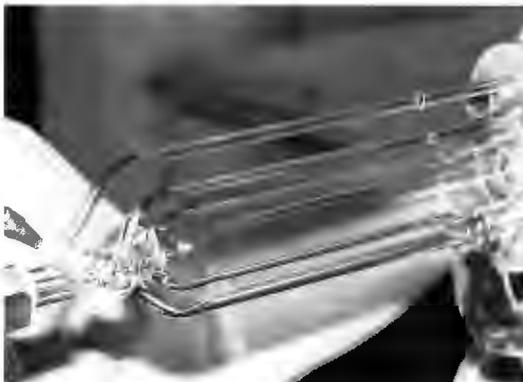
This blow hose assembly allows one to blow through both the inner reservoir and outer jacket simultaneously.



After the outer jacket and inner reservoir are joined in a ring seal, the 4mm stopcock is attached...



and a 24/40 inner joint with drip tube is added.

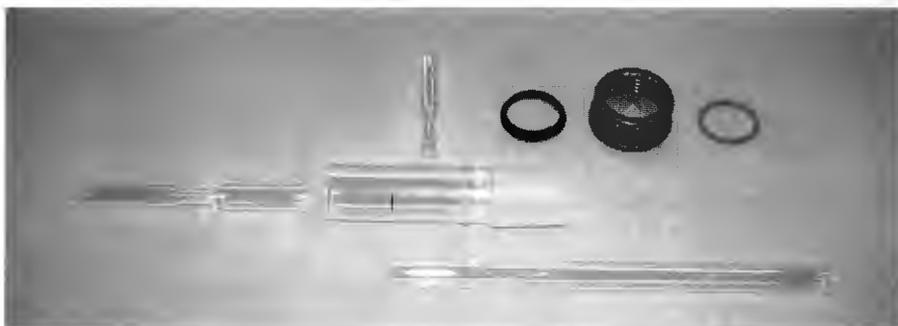


The lower portion of the pressure equalizing arm is first cold sealed to the upper portion and then worked into the 24/40 inner.

# Lathe Bends

by  
Tracy Drier  
Aldrich Chemical Company  
Milwaukee, WI 53225

The following is an outline of how to produce a 90-degree glass bend for our cold trap tops.



Sequence for producing cold trap top, up to the point of bending.



### Step One

- Produce a gradual uniform taper from the shoulder to the hose connection.
- Thicken an area 2-3 tubing diameters in length from the shoulder. This will assure uniform bend wall weight as the bend is produced. In this particular case, the bend will be as close as possible to the shoulder.



## Step Two

- Heat area to be bent until cherry red, while maintaining uniform taper.
- Shut off torch and let lathe rotate 1 or 2 revolutions. Glass will cool slightly.
- Stop the lathe:
  - ⇒ If glass is too hot, the tubing will droop.
  - ⇒ If glass is too cool, the bend will kink.
- With gloved left hand, reach across and grab the cold end of the hose connection while simultaneously unchucking the tail stock with right hand and wheeling it away.
- Slightly lift the glass and draw the tubing into an exaggerated upward arc. Pump the glass up and down (Y-plane) and in and out (X-plane) as necessary to bring the hose connection around to 90° in good form.



### Step Three

- Looking into the head stock while still holding the hose connection, twist clockwise or counter-clockwise as necessary to align in the Z-plane.



### Step Four

At this point, one of three things will have occurred:

- **Bend is perfect.** No additional action required.
- **Slight imperfections** (kink, frost). If so, hold the hose connection while supporting your arm on the head-stock chuck and heat as needed.
- **Major disaster.**  
Depending upon the severity, you have 2 options:
  - ⇒ Re-straighten the bend, re-chuck the tail stock, re-taper and re-bend.
  - ⇒ Attach glass rod from tail stock to tip of hose connection. The bend is held rigid. Work on the bend until completed.

## Step Five

- Complete top with side hose connection.



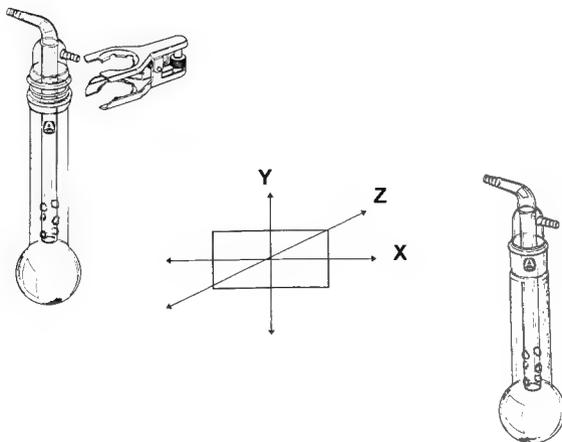
A variation on this assembly procedure is to seal on the side hose connection first.

### Advantages:

- The shoulder will have just been pulled down and sealed to the center tube. Everything will be very hot.
- You can put the side hose connection wherever you please.
- When going to make the 90° bend, after you have shut off your torch, waiting to stop the lathe with the side hose connection at the 6 o'clock position will allow the glass to cool slightly before making the bend.
- You can get absolutely perfect 180° alignment between the two hose connections.

### Disadvantages:

- You need to be comfortable with lathe bends or you may have one-too-many variables to keep track of.



# Sealing Tapered Quartz Windows

by

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## Introduction

A method for sealing Quartz windows yielding minimal optical degradation is presented. This method uses relatively low temperatures and does not require the critical lapping of previously reported techniques.<sup>1-4</sup>

## Preparation

A. The window is purchased slightly oversized, and is mounted to the required diameter tube with a resilient adhesive. I use Aremco's Crystalbond 509.<sup>5</sup> The window is then ground at a 30-45° angle. The final diameter should be about 0.5-1mm larger than the tube od (fig 1). Debond and clean the window.

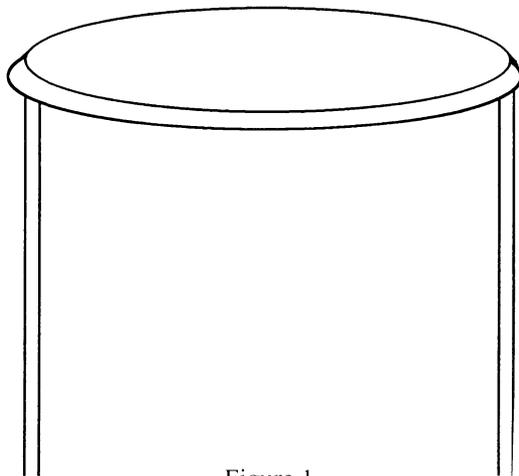


Figure 1

B. The tube is carefully cut square. If any high spots exist, they should be ground flat. Grit polishing is not required. Thoroughly clean and etch to remove contaminants, and lightly firepolish. Do not distort or bead the tube end during firepolishing; it is better to under rather than over polish.

## Set Up

I prefer to work on a ring stand as this allows access to the tube interior. The window is held in place with a light pressure as shown (fig. 2).

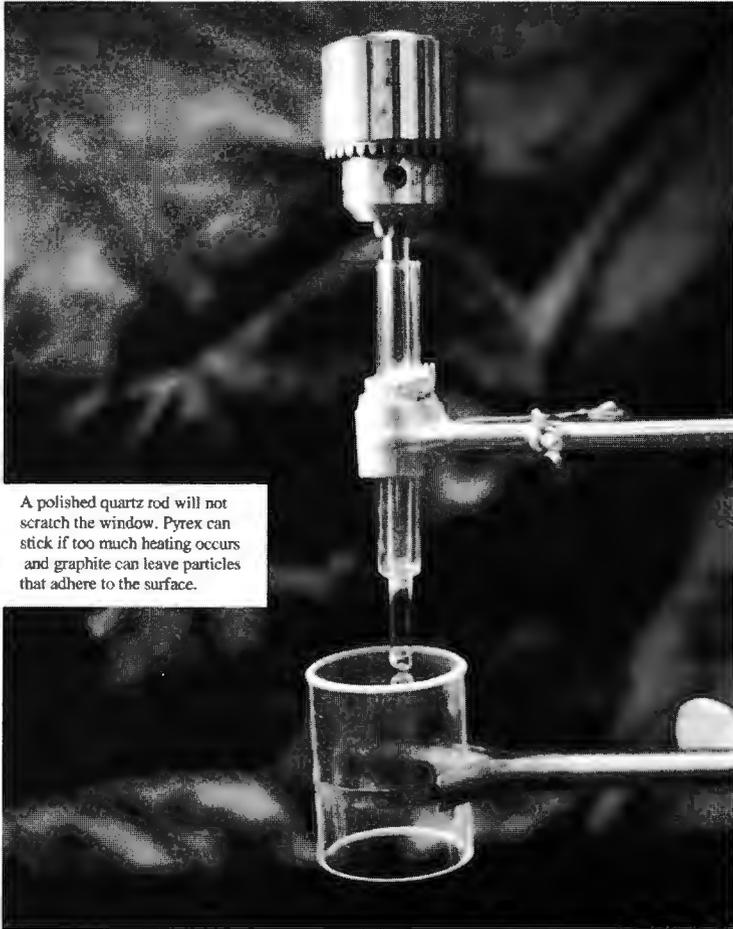


Figure 2

Additional items used are:

- 1) a National torch with a #2 or #3 tip
- 2) a National torch with a syringe tip or any 'micro torch'
- 3) a clean 3/8" or 1/2" graphite rod
- 4) split lens Quartz/Pyrex glasses (preferred)

**Method**

After lightly warming the window, focus the flame on the tapered edge (pointing away from the window) (fig. 3). As the feathered edge of the window starts to glow, roll the

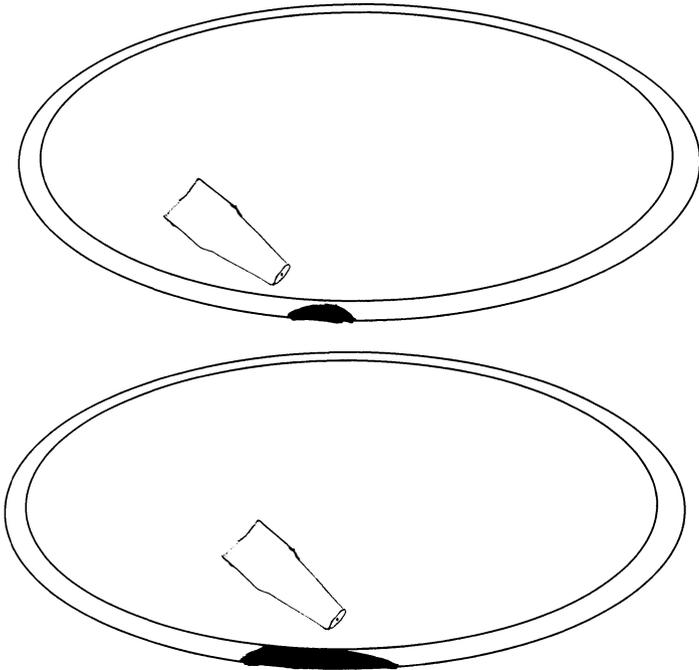


Figure 3

carbon rod down along the taper to force good contact of the bonding surfaces and smear the thin overhang down onto the tube. If the temperature is right, the interface will go clear indicating bond formation. This clearing will start with the outer edge and proceed inward. Repeat this process adjacent to the freshly bonded area until about one inch is sealed.

Go back to the start of the seal and fuse in the bead or seam to yield a clean smooth seal (fig. 4).

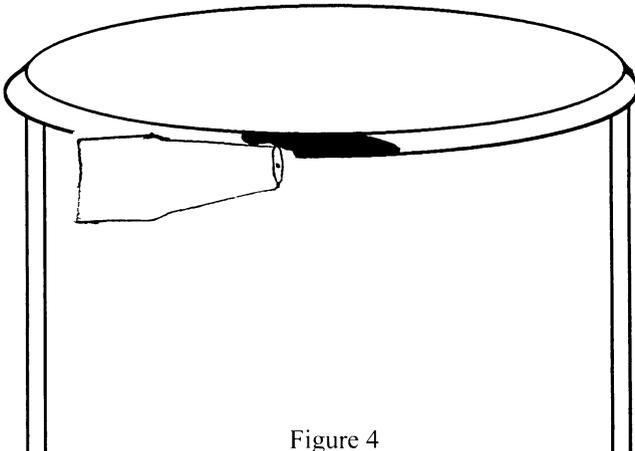


Figure 4

The interior of the seal can also be fused in at this time by using the micro torch inside and the larger torch outside (optional).

Repeat these steps until you have worked the full circumference.

### **Conclusions**

A method for sealing Quartz windows is presented. The high heat required for typical fusing is avoided, thus minimizing optical distortions. The fine lapping and polishing steps previously reported are sidestepped.

This method is especially useful when dealing with Brewster angle windows and optically flat windows. Care must be taken to protect the surface from handling damage, but distortions from the bonding process can be kept to a minimum.

Windows as large as 10" have been sealed with two glassblowers simultaneously using this technique.<sup>6</sup>

### **References**

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5. Aremco Products, (509) 762-0685.
6. Midrivers Glassblowing, (314) 939-9003

# Keeping Grad Students Happy or A Simple NMR Tube Cleaner

by  
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Kansas State University  
Manhattan, Kansas 66506

For a scientific glassblower in a university setting, many times graduate students are your main customers. Good business practice dictates making and keeping your customers happy. A complimentary NMR tube cleaner is a good introduction to the glass shop and some of its capabilities.

Students, aware of how the glass shop can help them with their research, will be better and more informed customers, making their advisors happier and your job easier and more rewarding. With a few simple techniques, putting this apparatus together need not be time-consuming and it is a nice little job to do at the bench.

## Nuclear Magnetic Resonance (NMR) Spectroscopy

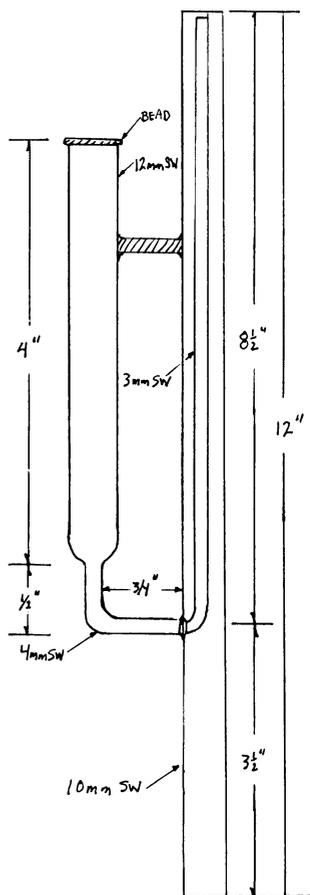
NMR Spectroscopy is used by researchers to help determine those NMR-active atoms which are present in a molecule and to aid in deducing the structure of that molecule. High resolution NMR spectra yield the necessary data for this analysis. These spectra are recorded on low viscosity liquids contained in thin wall glass tubes made to very precise tolerances. The highest grade of tube can cost over \$40, and, since they are rather fragile, a safe, efficient way of cleaning them is necessary.

The NMR tube cleaner described here is for 5mm by 7" tubes although the dimensions could be adjusted for other sizes.

## NMR Tube Cleaner

This design of NMR Tube Cleaner is meant to be used with a rubber stopper and a heavy wall filter flask.

The NMR tube is placed in the cleaner and the plastic cap normally provided with the tube is placed over the bottom to provide a good vacuum seal. The reservoir is filled with solvent and a vacuum is applied to the filter flask. This draws the solvent out of the reservoir and up through the inner tube, washing the inside of the NMR tube. This is usually followed with an acetone rinse, and finally just a stream of air is drawn through the NMR tube for drying.



## Materials

|                |          |
|----------------|----------|
| 12mm SW Tubing | (8")     |
| 10mm SW Tubing | (12")    |
| 4mm SW Tubing  | (12")    |
| 3mm SW Tubing  | (9 5/8") |
| 4mm Rod        | (1/2")   |

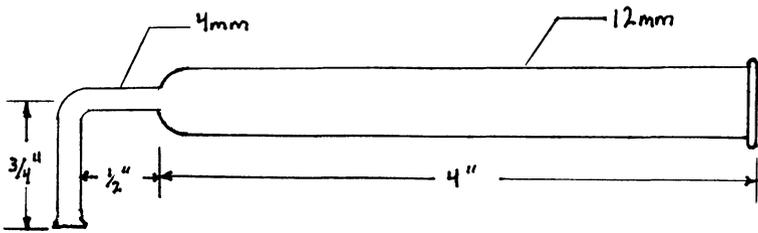
## Tools

- Centering holder for inside tube
- Small clamp
- Corks for 12mm and 10mm tubing
- Blowhose with swivel and tube to fit over end of 10mm tubing

## Parts Fabrication

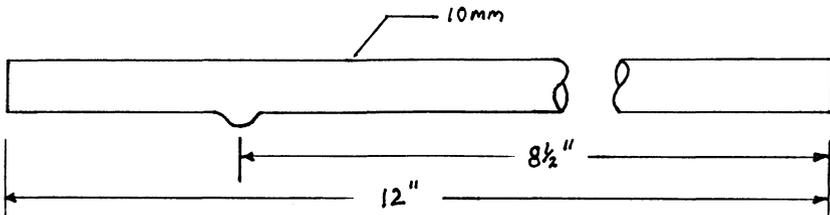
### Solvent Reservoir

1. Straight Seal 4mm to 12mm
2. Cut 12mm at 4" and bead end
3. Bend 4mm 1/2" from 12mm
4. Cut 4mm 3/4" from bend and slightly flare



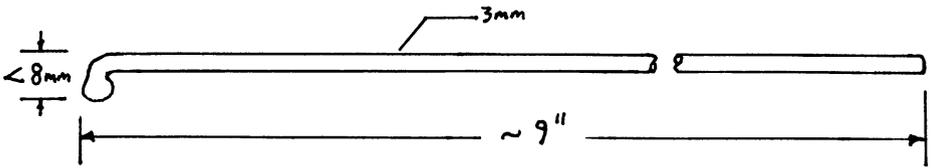
### Body Tube

1. Select the squarest end of the 10mm for the top and blow a small bulge 8 1/2" below this end.

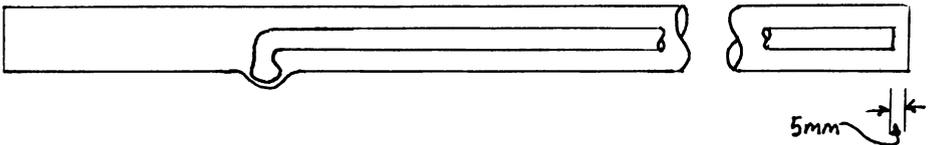


### Inner Tube

1. Bend about 20mm of 3mm tube a little past 90 degrees.
2. Pull off short end about 8mm from end and blow a small ball.

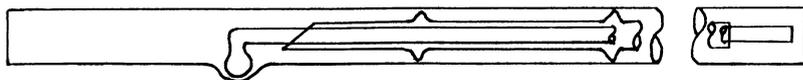


3. Check the fit. The 3mm tube with ball should be able to slide inside the 10mm tubing.
4. Slide the ball end into the bulge on the body tube. Mark the 3mm tube where it exits the 10mm body tube. Remove the 3mm tube and cut it 5mm below your mark.



### Assembly of Apparatus

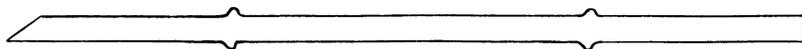
1. Slide the 3mm tube into the 10mm body tube until the ball drops into the bulge.
2. Slide the slanted end of the centering holder over the 3mm tube until it is entirely inside the 10mm body tube and holds the 3mm tube centered.



3. Cork the bottom end and attach your blowhose to the top. Fuse the bulge on the body tube to the ball on the 3mm. Blow out this seal for attachment of solvent reservoir.
4. Cork the solvent reservoir and clamp to body tube. Check alignment. Seal reservoir to body tube and flame anneal.
5. Remove clamp. Carefully insert brace rod and seal to reservoir and body tube. Flame anneal.
6. Remove blowhose and centering holder. Check for perfect centering on 3mm tube. If not centered, heat on triple seal and center.
7. Over fire polish top of body tube until it collapses to about 6mm. Check with vernier. Flame anneal and you are done!

### **Notes on Techniques**

1. The centering holder is made by pushing up two marias (slightly less than 8mm) on a length of 5mm tubing. When complete, the overall length should be around 7 inches. Curing one end at a slant makes it easy to slip over the 3mm tubing and keeps it away from the seal area. Unlike tapes or paper, it will not burn up or outgas at the most inconvenient times.



2. By blowing a small ball at the end of the inner tube, it is easy to fuse the triple seal and there is no chance of an unsealed area such as might occur if you just flared the end. The ball also tends to make the area of the seal slightly stronger than just a bent tube.
3. When attaching the brace, direct a small sharp flame mainly on the rod and very slightly on the body tube and reservoir tube. The heavier weight of the

rod will transmit plenty of heat to the walls of the tubing for a beautiful, undistorted seal. Blow hard at the last to make a slight indent up into the rod, thereby avoiding an abrupt change in wall weight from tubing to rod.

4. Putting the clamp on and aligning the reservoir before the seal is made avoids the maddening occurrence of your clamp "moving" the glass when you are making the seal. It is easy to pivot the reservoir up slightly to heat both sides just before making the seal.

### **Acknowledgments:**

I would like to thank the Chemistry Department at Kansas State University for supporting me in my endeavors to increase my skills in glassblowing. I would also like to thank my teachers, and the glassblower or researcher responsible for the first design of this NMR tube cleaner.

# Triple Wall Reactor

by  
Doni Hatz  
The Procter & Gamble Company  
Loveland, OH 45140

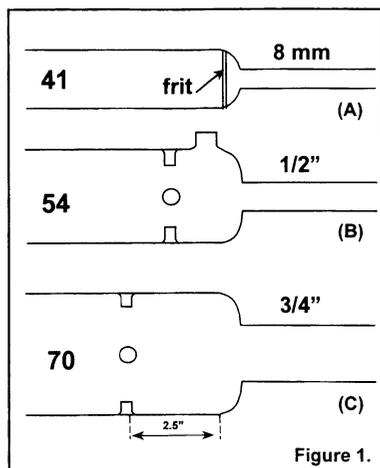
Demonstrated in this poster is a vacuum jacketed reactor for an automated chemical reaction system. The inside chamber holds the chemicals for organic synthesis. The chamber between the inner and middle walls are where the heat transfer fluid flows. The chamber between the middle and outside walls are evacuated to provide insulation. The advantage of this vessel compared to a metal reactor is that you can visually see what's happening. There will be more modifications made to this design, but this is a starting point for the initial work to begin.

The reactor has a 100ml capacity that connects to an adapter of inlet tubes. A 10mm stir rod and bearing connect to the adapter that has a customized glass fin on the bottom. A 41mm od tube is jacketed by a 54mm od tube, and the 54mm od tube is jacketed by a 70mm od tube. The top connector is a 45/50 inner Rodaviss ground glass joint. A 25mm extra coarse frit is sealed in the bottom portion of the 41mm tube (to catch sediment). Only the reactor body will be demonstrated in this poster.



## Preparation

To begin, a point is pulled on a 41 mm outside diameter od tube with the shoulder tapered to seal in a 25 mm od extra coarse frit. Once the frit is sealed, slightly taper the shoulders and seal on an 8 mm medium wall (MW) od; make sure this tube is perfectly centered. Flame anneal and set aside (figure 1a).



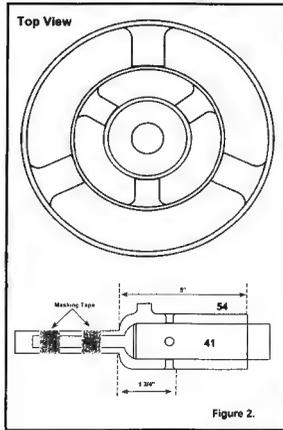
Once it is cool, trim the 41 mm od tube to 5 inches from the shoulders and acid etch the saw cut. Heat and flare the trimmed end of the 41 mm od tube to 54 mm od tube.

Prepare the next tube by pulling down 54 mm od tubing (in the lathe or bench) and seal on 1/2" MW od tubing; make sure this tube is perfectly centered. The bottom water connection is made by adding some rod and blowing it out; as with the side seal, trim to 6 mm. (The rod is used to increase the mass of glass so you do not need to seal on another tube; a one shot, short seal.) Flame anneal and set aside. Once the tube is cooled, trim to 5 inches from the shoulders and acid etch the saw cut. Heat and form a bead on the end of the 54 mm od tube, flame anneal and set aside (Figure 1b).

To support the 41 mm od tube into the 54 mm od tube, two-inch masking tape is wrapped in two sections. Wrap the tape around the 8 mm tube to hold the tube tightly enough inside the 1/2" tube to keep it from flopping around. Cut a small channel lengthwise into the masking tape for air to flow freely through the tubes. Once the 41 mm is centered in the 54 mm od by the supports, I found extra support is needed to guarantee that the ring seal will stay centered. Since a stir rod will be supported and suspended by a bearing, held in an adapter above the reactor, all pieces have to be perfectly centered.

A simple method to keep the 41 mm od centered is to make indents in the 54 mm od tube. A 1/4" graphite rod is used to make 3 indents that should be spaced evenly apart. The tube is heated and indents are pressed into the 54 mm od tube (with the square end of the graphite rod). To avoid fusing the tubes together, press the indents in but leave the slightest gap so as to not touch the inside tube. Then heat the indents again to finalize the support of the inside tube (the indent tip has a chance to cool and the heat is only on the outside of the indent). Adjust the indents to support and center the inside tube perfectly.

Make the indents about 1.75 inches from the shoulder (this is to avoid being too close to the lower water connection during fabrication). I usually do this at the bench, checking with the support rollers so as to see that the 41 mm od tube will not go out of center (figure 2).



Prepare the 70mm od tubing by pulling it down and sealing on 3/4" MW od tube. Trim the 70mm od tube to a reasonable length (about 18 inches) to support both ends of the tubing in the lathe (one end in the headstock and the other end in the tailstock). Support the 54mm od tube (like the 41mm od tube) with two-inch masking tape in two sections to fit inside the 70mm od tube. Heat and press in the indents on the 70mm tube as described before for the 54mm tube, 2.5 inches above the shoulder (to avoid hitting the indents in the 54mm od tube) (figure 1c). Flame anneal and support in the lathe.

The side arm connecting tubes are made of 10mm MW od tubing. One tube is heated and bent at a 90 degree angle and the other tube is constricted (for evacuation) and bent at 90 degrees. A 45/50 outer ground glass joint holder is prepared with two side arms. This is needed to connect rubber tubing to the upper two side arms for air pressure when making the bottom ringseals (shown in photo 1).

**Fabrication:** Support the 54mm od tube inside the 70mm od tube. Place in the lathe, heat and pull down the tubing and form the ring seal.



Once the ringseal is flowed in, blow out the inside tubing and trim as much glass off the end as possible. The next step is to make the upper water connection.



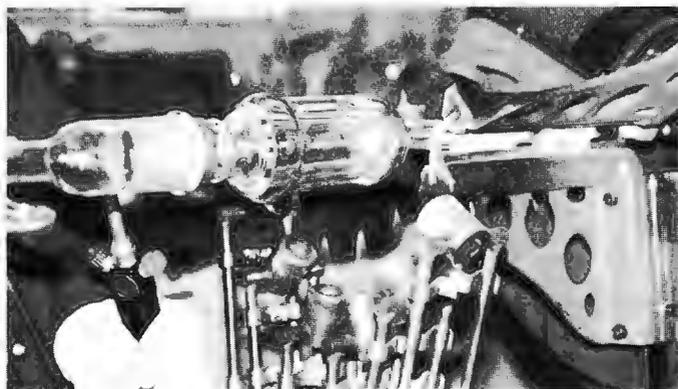
Heat the outside tube with the hand torch and begin forming the top water connection. Using a small tip on the torch, collapse the outside and inside tubes until they touch. Open the connection with a tungsten pick and ream it open to fit 10mm od tube. Once the port is sealed, close the port off with a spare piece of tubing.



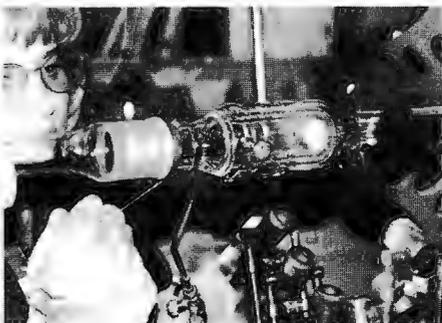
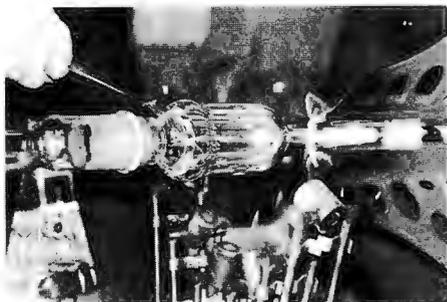
Insert the 41mm od tube (prepared earlier), and cork the 8mm tube to hold the air pressure. Heat up the tubing and make a dewar seal, sealing the flare of the 41mm tube to the 54mm od tube. Flame anneal and keep the reactor body warm.



Trim down a 45/50 Rodaviss inner ground glass joint and seal to the reactor body.



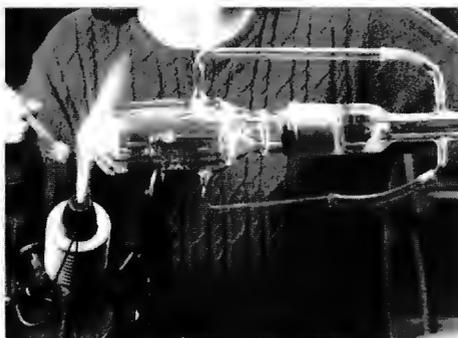
Heat and blow out a hole to insert the 6mm od tubing thermowell (trimmed at 75mm, to fit inside the 41mm tubing, flared at an angle). Once the 6mm od tube is sealed, adjust it to lay on the side of the tube. Flame anneal and prepare for the next seals.



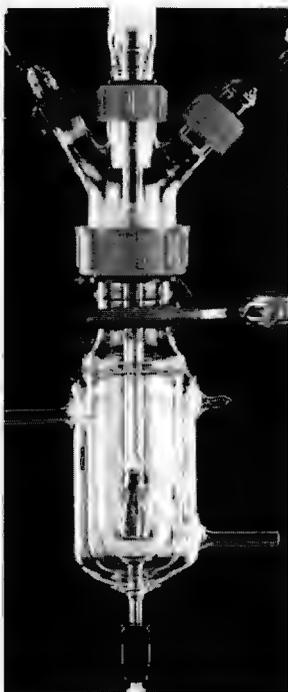
Heat the upper water connection and blow out the seal made earlier; seal on a 10mm od tube, then blow out and seal on the next 10mm od tube with the constriction. Flame anneal and hook up flexible tubing to the 45/50 outer ground glass holder.



Begin heating the shoulders of the bottom of the reactor and pull off the 3/4" tubing and other tubes (quickly so as to avoid heating the masking tape). Heat the bottom water connection and seal on the 10mm od. Remove as much glass as possible between the two ringseals and seal on a 2mm id and 6mm od capillary altec fitting. Flame anneal and throw into a preheated annealing oven.



The final reactor set up before installing into rack.



Another reactor with a 150ml capacity, in the laboratory.



# Water Jacketed Electrochemical Cell

by

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This simple water jacketed Electrochemical cell utilizes threaded glass connectors for air tight interface of electrodes. The experiments conducted with this cell must be completely free of oxygen. Because of the oxygen-absorbing tendency of Teflon™, a glass plugged high vacuum stopcock was incorporated into this cell. Both the threaded connectors and stopcock assembly were purchased from Chemglass, Inc. (Photo 1)



Pieces required to build apparatus. (Photo 2)

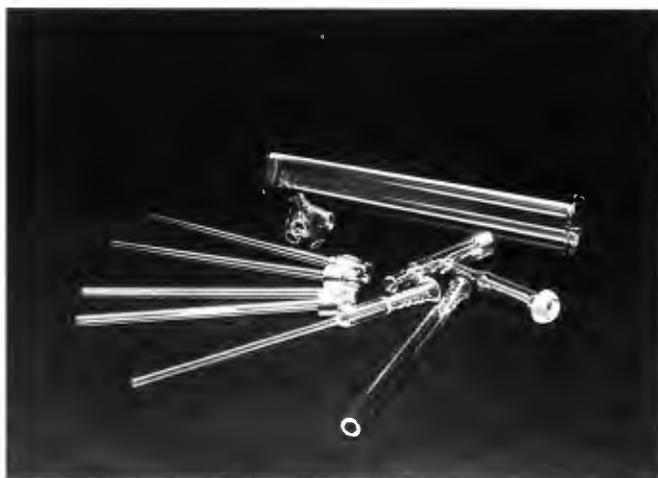


Photo 2

Construction of the inner cavity was accomplished with 10mm standard wall tubing. The seals were worked out with a Micro-Torch and a #5 tip to ensure sharp corners and to minimize volume. A side seal was made at 90 degrees to accommodate the stopcock assembly. (Photo 3)



Photo 3

Flat bottom a piece of 38mm standard wall tubing and allow to cool. Cut the inner cavity to snugly fit in the 38mm. Place inner cavity into the 38mm with the side stopcock port toward the flat bottom. A Pluro stopper with a long piece of tubing cut to the proper length is used to hold the inner cavity in place. (Photo 4)

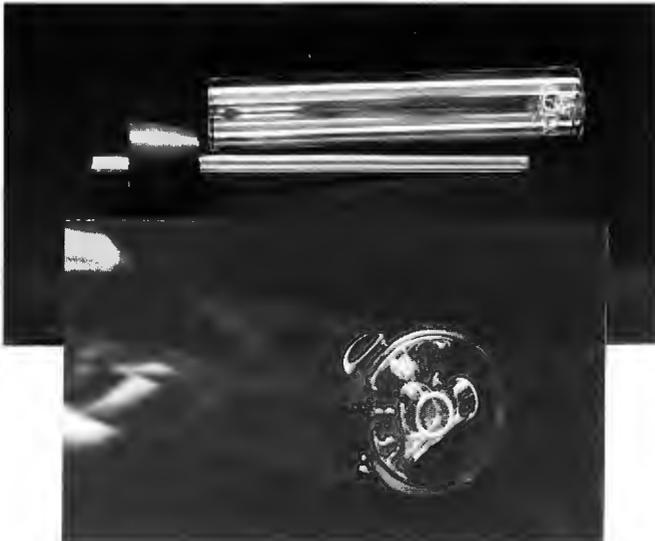


Photo 4

Seal the side stopcock port to the flat bottom. Do not blow open the ring seal. This will hold the inner cavity while you add the remaining ports. You should keep the piece hot at all times now and complete the cell without allowing it to cool. Remove the support tube from the Pluro stopper. (Photo 5)



Photo 5

Add three of the four threaded connectors and thoroughly work out all seals. (Photo 6)



Photo 6

Connect the stopcock to the cell and add a second blow hose from the stopcock side. (Photo 7)



Photo 7

Blowing from both sides, add the fourth threaded connector. (Photo 8)



Photo 8

Remove the blow hose from the stopcock. Add the water inlet and outlet, leaving one outlet with a long tube connected to the end. Bend this toward the stopcock. You will use this to blow through to close the water jacket. (Photo 9)



Photo 9

Close the water jacket by putting on a flat bottom. Flame anneal or place in a hot oven. (Photo 10)



Photo 10

# Technical Workshops 1998

Peter Brooks - *Oxford University*  
**"Lathe Tool Post for Shaping Glass"**

Dan Edwards - *Northern Illinois University*  
**"Fabrication of Lab Apparatus"**

Mike Greico - *V.M. Glass Company*  
**"Lathe Tooling"**

Eri Maraine  
**"One-Gallon Glory Hole"**

Mike Souza - *Princeton University*  
**"Large Ring Seals for Jacketed Reactors"**

Tom Stefanek - *University of Minnesota*  
**"Double Electrode"**

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Giessen D-35331  
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Hellertown, PA 18055-  
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Lab Glass/WilmadGlass  
Scientific Group  
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Buena, NJ 08310-0688

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Redwood, CA 96064

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200 Litton Dr.  
Grass Valley, CA 95945

Lunzer Inc.  
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Saddlebrook, NJ 07663

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