PROCEEDINGS

THE FORTY-SEVENTH AND
THE FORTY-EIGHTH ANNUAL

SYMPOSIUMS
ON THE
ART OF SCIENTIFIC GLASSBLOWING

2002 & 2003

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Proceedings

The Forty-seventh Annual
Symposium
on the
Art of Scientific Glassblowing

Sponsored by
The American Scientific
Glassblowers Society

Marriott Grand Hotel
Point Clear, Alabama
May 28-June 2, 2002

THE
AMERICAN SCIENTIFIC GLASSBLOWERS SOCIETY
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Papers

Working with Borosilicate Color

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ABSTRACT

Working with colored borosilicate glass can bring many hours of enjoyment or be a very painful and humbling experience. It is not unlike welding in the sense that it is important to understand the properties of the materials with which you are working. Once you understand the colorant constituents, how they respond to heat, flame atmosphere and annealing, it is quite easy to work with borosilicate colors. The following paper explores the primary colorants used to color borosilicate today.

THE NEUTRAL FLAME

Prior to the pioneering work at Glass Alchemy, most of the information that was available for working borosilicate colors was nothing more than myth. Some people could not get their silver colors to strike, others could but could not explain how to another person, colors would turn "muddy," rubies would become livery and greens would turn red or crack. The stock answer for everything was "you did it wrong," "go take a class," or "your flame needs to be oxidizing or reducing or...." To make matters worse, nobody had a method for adjusting the flame to neutral position and verifying it.

Today, for a successful and rewarding experience with colored borosilicate, we understand the importance of the flame chemistry. It is imperative that the artist work in a neutral flame calling upon a reducing or oxidizing flame when needed. After exploring flames and torch set-ups in hundreds of studios, it has become clear that the setup of the torch often turns out to be more influential on the final outcome of the piece than the original choice of torches or any adjustments made at the knobs during construction of the piece. For these reasons, the lampworker should be familiar with some basic principles of flow dynamics so that they can set their torch up to maximize its performance.

Supply line architecture is of extreme importance for a proper setup. In an attempt to save money, one may choose a smaller diameter hose/pipe thinking that "20 pounds of pressure is 20 pounds of pressure," not considering the pressure drops associated with the various diameters of supply lines or the volumes that can actually be transported through these long constrictions. The symptoms of this problem vary. Often there is not enough oxygen to fully combust the propane and a reducing flame results. This type of flame can inject carbon into the glass and shift the refractive index such that the color is not bright; the carbon can reduce certain metals used as colorants (such as cobalt turning gray) and, in some cases, cause the colors to go "muddy". The common solution is to increase the pressure to increase the volume, which introduces many other problems but does not increase the volume. Increased pressures are a primary cause of turbulent flow and often result in a cooler, reducing flame.

To achieve laminar flow at the torch, the basic goals when setting up the torch are to know that enough volume of gas is being delivered and to be able to stabilize the flow. It is important to know the demand of the torch for gas and air in an all-out situation. The regulators need to be capable of supplying this demand. Due to pressure drop issues, supply lines should be short and of significant enough bore to deliver the demand volumes. Simply increasing the pressure to get more juice is very similar to over-pumping the beer keg…foam. The same thing happens in the torch: the laminar flow becomes turbulent and the flame consists of hot and cold spots. It takes longer to pull points, build sculpture and the colors become dull.

When the torch is set up properly and the correct propane and oxygen settings are chosen, the colors will perform as they are designed to. To test for a neutral flame Glass Alchemy, Ltd. recommends that you use a stick of 987 Amazon Night and heating to a warm orange glow and cooling. If the stick is a light sky blue or has a metallic sheen, the flame is reducing and needs to be adjusted. Reduce the propane content. If the stick is sky blue, it is very reducing and can only be adjusted by reducing the propane pressure at the regulator. If the rod is metallic, adjustments of the regulator of 1/4 pounds at the regulator should result in a neutral flame.

WORKING WITH COLORS

When creating a piece, many elements go into the design. Consideration is given to the overall size, what colors, the order of attachment and so forth. Often the color choices may include some of the more serendipitous ones such as 786 Triple Passion. When using one of the "color changing" types, the preferred method is to build the complete piece in a hot, neutral flame. After the piece is assembled, then go back and bring out the properties of the colors.

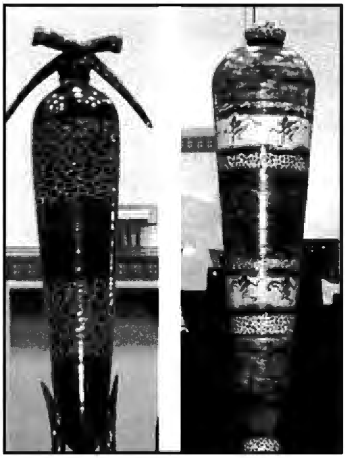
CADMIUM COLORS:

(such as 104, 106, 204, 206, 301, 302, 304, 403, 406, 804 crayon colors.) *Health Warning: Cadmium is a heavy metal that sublimes at a temperature below the optimal flame working temperature. Acute toxicity from inhalation of cadmium fume or ingestion of cadmium compounds can produce serious illness, particularly in the lungs and GI tract respectively. There are several large on going studies of cadmium-exposed cohorts which are shedding light on cancers related to cadmium. Chronic cadmium exposure causes a pulmonary fibrosis and bronchitis, producing both restrictive and obstructive changes. The disease progresses, apparently even after exposure ceases, leading to shortness of breath. Therefore, when working with cadmium colors, always work in a well ventilated area (mechanical; positive or negative).*

Cadmium colors sublime (similar to evaporate) at temperatures near 1750-1800° F. To prevent cadmium colors from subliming into a gas, work the rod further out in the flame, encase it or adjust the flame to a cooler setting. You can adjust to a bushier flame which is cooler because not all of the propane burns or to an oxidizing flame because the flame tends to be more turbulent and is rich in oxygen which cools the flame.

The 104 red is the color richest in unbound cadmium and therefore requires the greatest skill. The 106 is a darker, "iron oxide" red and is bound with selenium and much easier to work. The 106 is closely related to 804 Chocolate which is the easiest cadmium to work.

COPPER COLORS:



Reduced copper (reds) (such as 132, 135, 138, 139). *Health Warning: Copper also puts off toxic fumes when melted (in the reduced red form the melting point is about 1984° F) so use only in a well ventilated area.*

Copper is an ionic colorant. In different valence stages, copper provides different colors due to the distortion in the shape of the molecule. To obtain the red color, a percentage of the copper is reduced to the native state. Not all of the copper in the rod is reduced, therefore working the color with a reducing flame can deepen a red color or working with an oxidizing flame can introduce dark (green, even blue) lines into the red color. The difference between the 138 and 139 is the percent of copper that has been reduced at the factory. The 139 Cherrywood is an opal ruby (more copper has been reduced, but not enough to provide a metallic sheen as is found in the 132 Jasper Red which is a similar formula with additional reduction) while the 138 Ruby Strike is a transparent ruby (with less reduction).

All reduced copper colors (copper ruby family) should be worked in a neutral to oxidizing flame. The opal rubies can be worked in a variety of flames to add "character" to their final appearance. All rubies need to be oven struck to bring out their red color otherwise they will have a salmon hue to them. GA colors are designed to strike at 1075° F for one hour +/- 20 minutes. Shorter striking will yield less red while additional striking will darken the color. Flame striking or oven striking at higher temperatures is not recommended for transparent rubies.

Unreduced Copper (Greens) (such as 421, 431, 521, 531). *Health Warning: Copper puts off toxic fumes when melted (in the un-reduced green form the melting point is about 2418°F). Use adequate ventilation.*

These are beautiful, pleasing transparents that bring out the best in glass. They both transmit and reflect light which makes glass a unique medium. While they can create great dots, they make spectacular sculptural colors.

Rare Earth Colors (such as 161, 163, 263, 461, 761). *Health Warning: Similar to colorless glass. Use only in a well ventilated area*

These colors are not affected by the torch atmosphere and so a neutral flame is recommended. These tint colors can be used over a white to create a pastel color, over other opals as an encasing to change the reflected color, or gathered into large masses such as in marbles or sculptural shapes to yield a vibrant transparent color. The rare earths also exhibit florescence and glow different colors under UV light. The UV bulb must be of the BLB (black light blue) type and should be less than one year old.

Silver Colors (such as 182, 287, 381, 382, 383, 385, 386, 388, 481, 485, 487, 489). *Silver is a heavy metal and it can accumulate in your body. "Overloading the body's natural eliminative systems with silver causes the body to store some excess silver in the face; this over time can result in a pronounced gray complexion. Argyria is strictly a nontoxic, cosmetic condition. However, argyria is quite serious in that it is thought to be permanent, much like a tattoo." Sited from www.silversolutions.com*

Certain silver colors are heavily saturated and can have silver wire running through it. Treat this color as you would treat fuming. In addition to ventilation, you should use a shield or HEPA respirator.

Silver is a colloidal colorant and is reduced by heat as well as by carbon. At elevated temperatures, if nuclei are present, crystals will start to grow on them. This is similar to a rain drop needing a speck of dust to form. When silver is not mixed with another colorant, it is placed in the yellow category due to the fact that the smallest visible silver crystal creates a yellow color. Silver crystals always grow in the same sequence, from yellow to orange, red, red-purple, purple, blue and finally green. Areas that are hotter will grow crystals faster than cooler spots. An area that appears purple on the surface will be ruby color below the surface when you cold work it. Because the color change is dependent upon temperature, and, knowing that glass is an insulator, frit pick-ups or "dots" will create different colors under the glass. Touching the silver colors with tools such as needle nose pliers will leave patterns of colors.

Some colors have crystal growth inhibitors to impede the change of color. For many applications, the color will remain yellow; however, hard working or prolonged kiln work will cause the crystals to grow. Many do this to achieve purple where another choice may yield a blue because of the working conditions and time.

Some colors have no nuclei. These colors can be worked very hard but require cooling to a slight orange glow to create nuclei on which to grow crystals if you want to create a rainbow of color. After creating nuclei, raise the temperature of the flame to grow the crystals.

Some colors are loaded with nuclei and are very "playful." These colors will always yield different results. With a modest amount of nuclei, the colors strike easily and can be controlled so that matching pieces can routinely be produced.

Some colors have additives that cause the glass to always transmit orange, red or some other color of light. Hobnails on beads or bobbles catch the light and create dazzling patterns on the walls. These colors should hang in every window.

Silver colors can also luster. Lusters can range from very modest to a bright mirror effect. The luster is achieved with a reducing flame; this removes the oxygen from the surface of the glass, thus yielding metallic sheens. Use a buff wheel and silver polish and the piece will become very reflective and the metallic sheen will be enhanced. Prolonged kiln striking intensifies the metallic sheen.

Work pieces containing silver at hot temperatures to create, form and assemble all of the elements. Use reduced (lower) heats, just above the annealing temperatures (1075°F to 1125°F), to heat treat the entire piece causing growth in all of the silver crystals. If you want to bring a luster to the surface, raise the temperature of the flame about 75 degrees (remember that it is easy to burn a fume off of the surface) and treat the surface about 20 seconds in a busy, reducing flame. This flame will strip oxygen from the silver oxide (silver is reduced with heat, un-burned carbon will transport liberated oxygen away) leaving metallic silver on the surface causing the "sheen". If it is necessary to create nuclei (you cannot get the silver color to strike), lower the temperature to a slight orange glow or just above the strain point, about 975-1000° F.

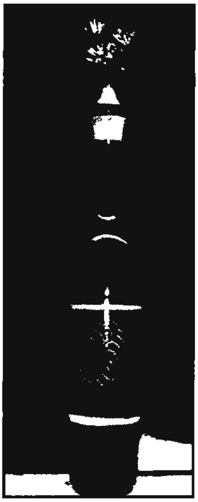
Chrome colors (such as 441, 442, 444, 445, 446, 546, 548). *Health Warning: Chrome is a heavy metal and is on a lot of lists. In most forms, it is a poison, in some forms cancer causing. These colors should always be worked only in well ventilated areas. In addition, chrome puts off a very bright white flare and excellent eye protection is required.*

Chrome based colors tend to be opal (opaque); if worked incorrectly, this can be problematic. To avoid problems, the torch must be set up properly to achieve a neutral to oxidizing flame. A reducing flame can cause cracking in chrome colors. If the color is going to be exposed to prolonged periods of a reducing flame, consider encasing first.

If you are holding chrome colors in the kiln for an extended period, it is best to "garage" at 975°F rather than at the annealing temperature of 1050° F. You should not hold these colors (or strike other colors in the same piece) at temperatures above 1075° F due to the risk of creating aventurine at elevated temperatures.

Aventurine colors, produced from precipitated chrome, should be worked in a neutral to oxidizing flame. While much has been done to stabilize the chrome to prevent unwanted aventurine growth and cracking, it is the artist's responsibility to mitigate the known issues when working with chrome colors. "Tugs" to stretch this color will align the platelets to improve the level of sparkle. Also, it is known that when working with clay, it is necessary to align the platelets. Not much is written about the use of aventurine glass as it relates to flameworking. Our experience indicates when adding on glass to use a wipe-on/(wipe-off) technique rather than straight seals at 45/90 degrees. End-to-End seals should be quite hot and pressed together and then pulled/stretched to align the platelets. GA makes this recommendation because it has noticed that Mother Nature tends to be consistent in her rules. The sparkle is most pronounced when thinned, covered in clear or used for inside work.

Cobalt Colors (such as 510, 512, 514, 515, 516, 517, 592). *Cobalt is an ionic colorant, therefore the higher the cobalt content, the denser the color. Also, the thicker the application, the darker the color becomes. Health Warning: Cobalt is a heavy metal and inhalation of cobalt fumes can cause shortness of breath, coughing and pneumonitis. Hypersensitivity appears to be involved because lung changes occur at low incidence and are varied in intensity and time of onset. In most cases, the symptoms disappear. Cobalt is listed by The International Agency for Research on Cancer (IARC) as Category 2B possibly carcinogenic to humans. Cobalt is listed by ACGIH as an animal carcinogen. Cobalt is known to the State of California to cause* *cancer. Use only in a well ventilated area.*



Cobalt blue has always been one of the most appealing colors in glass. Cobalts are infra-red emitters; because of this they require more energy to melt and they cool much faster so that they work "stiff." In some heavily saturated formulations, a flux can be added to soften the glass in the temperature range above 1800° F. A typical working technique is to apply more heat to cobalt than other colors or clear.

Cobalt can produce grays in a reducing flame. This can be pronounced when the glass cools to a very light orange and then placed back into a reducing flame. If you experience gray cobalt, you should test your flame for neutrality. If a reducing flame is required, then keep the work hot rather than allowing it to cool below 1400°F.

Manganese colors (such as 672, 683, 773, 775, 860, 864, 974). *Health Warning: Symptoms of manganese poisoning range from sleepiness and weakness in the legs to difficulty in walking and uncontrolled laughter. Health surveys of employees exposed to manganese fumes have demonstrated a high incidence of pneumonia in these workers (OSHA). Work in a well ventilated area.*

These are very easy working colors. A neutral flame is recommended. These colors coil pot very well with no visible lines.

Manganese is an ionic colorant, commonly producing transparent browns that can be used as tints or used as solid colors. In a reducing flame, they can be shifted to a smoky black color.

SUMMARY

1. Work crayon colors in a cooler flame, either by moving out 1.5 inches or cooling the flame. You can also encase the crayon colors.
2. Generally use a neutral or an oxidizing flame. Only use a reducing flame to bring silver metal to the surface.
3. Chrome colors can crack if worked in a reducing flame.
4. Avoid using colors with heavy colorant content next to other colors with heavy colorant content.
5. Avoid using colors with heavy colorant content with dichro, especially high Manganese colors.
6. Use a neutral to oxidizing flame with cobalt colors; keep them hot.

Reference

Excerpts reprinted with permission from Drew Fritts. *Torchworked Marbles, Vol. 1 - Beginner to Intermediate Techniques*. Springfield, MO: Dichro-Moon Publishing, 2004.

Glassware Innovations at the University of Wisconsin - Madison

by
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ABSTRACT

A number of original glassware innovations have been designed by our faculty and students. These ideas are then prototyped and modified as necessary in the department's Glass Shop. This paper will review four products that have been built between November 2000 and February 2001.

INTRODUCTION

Progress and innovation are the norm in a university environment. The Chemistry Department at the University of Wisconsin in Madison is recognized as a leader in chemical research. Original research in many cases requires custom designed equipment. A number of original glassware innovations have been designed by our faculty and students. These are then prototyped and modified as necessary in the glass shop. This paper will review, in chronological order, the designers and the products that have recently come out of the UW-Madison Chemistry Department. Acknowledge these designers if you use their designs.

1. Dry Solids Addition Funnel

Figure 1. *Dry Solids Addition Funnel.*

Student Lei Jiang designed a powder addition funnel for air-sensitive compounds. This idea was brought to the Glass Shop to be built in November of 2000 and is shown in Figure 1. This is a novel approach that is simple to use, easy to clean and provides a clean addition of chemical into the reaction flask. This is important when the amounts to be added are critical.

The unit consists of two parts: the spoon which holds the solid, and the funnel which seals the reaction system. The funnel was made from a 250 ml flask with a bottom inner joint, a top outer joint, and an outer joint in the middle of the flask. A spoon attached to an inner joint swivels for the powder addition. The spoon runs through the vertical centerline of the flask when the spoon joint is seated.

Operation:

If the weight of the chemical to be added is critical, the chemical and glassware is weighed in a glovebox. The spoon is flame dried over a Bunsen burner to prevent the chemical from sticking.

The funnel is placed on a round-bottom reaction flask. Addition can be achieved in a number of ways. These include:

- A reflux condenser in the top of the funnel will wash out the solids. Slowly turn spoon to add in or reflux will dilute solids back to the flask.
- Replace the reflux condenser with a septum. Use a syringe to dissolve solids.
- A septum or nitrogen adapter lets you introduce an inert blanket (e.g., Nitrogen). Dump solids.

A prototype has been made for a spoon with two chambers. This allows for two separate chemicals to be added independently with one spoon.

2. Preparative Electrophoresis Chromatography Apparatus

This apparatus was designed by student Lizheng Zhang and was brought to the Glass Shop in January 2001. The intended use was for purification of RNA and was highlighted in a paper at last year's annual ASGS symposium. The design went through three modifications with the final design shown here in Figure 2.

The design consists of a water-cooled gel chamber, buffer solution chambers, positive and negative electrodes, Teflon stopcocks, and a quartz tubing section.

Operation:

A gel column is saturated in a buffer solution between two electrodes. The negatively charged RNA is placed on the gel. The stopcocks are positioned to create an open path between the two electrodes and 2000 volts is passed between the electrodes. Only the RNA is moving through the system. The negative RNA ions travel through the gel towards the positive electrode. Traveling through the gel, the impure RNA sample separates into discreet horizontal bands depending upon their size and shape.

At this point there is no way to visually see where the RNA is in the system. Here we use the fact

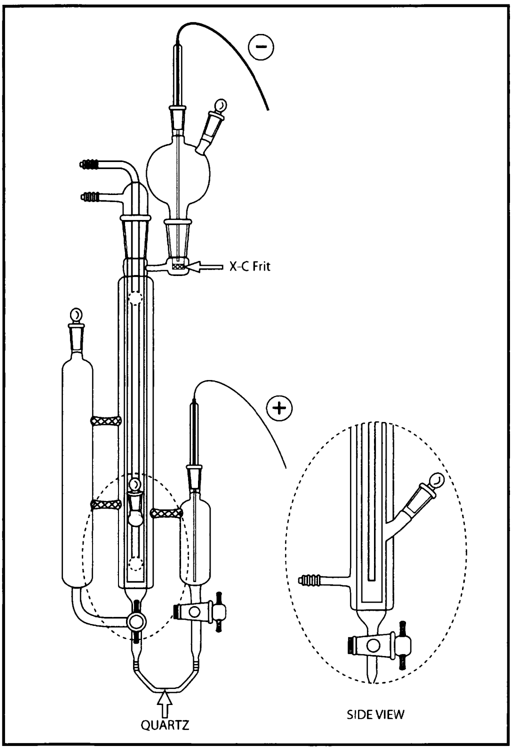

Figure 2.
Preparative Electrophoresis Chromatography Apparatus.

that RNA will absorb ultraviolet light and quartz glass will not. Using a short wave UV light in front of the quartz tubing section and a fluorescent plate behind, a dark outline will appear on the plate if RNA is present. Turn off the power source. Reconfigure the stopcocks to flush the sample into a collection flask. Continue the process until RNA is present again. These collected RNA samples are then characterized for further use.

3. Gas Washer

Thorsteinn Adalsteinsson designed a gas washing apparatus for cleaning an inert gas stream such as nitrogen or argon. The nitrogen is passed through the washer before using to remove impurities prior to its use in an experiment. Typical contaminants in a nitrogen stream include water and oxygen.

The washer is shown in Figure 3. The gas stream travels from left to right. Each of the U-bends has a blind seal in the main connecting tube to force the gas to pass through the U itself. The 100 ml flask in each bend acts as a surge protector and splash guard. The volume of each bend up to the flask is less than 100 ml. On top, there is an inner joint with cap used for filling each bend and there is a stopcock on the bottom for draining. Each bend also has a bypass stopcock. This design is compact and easily accessible for filling/draining/cleaning.

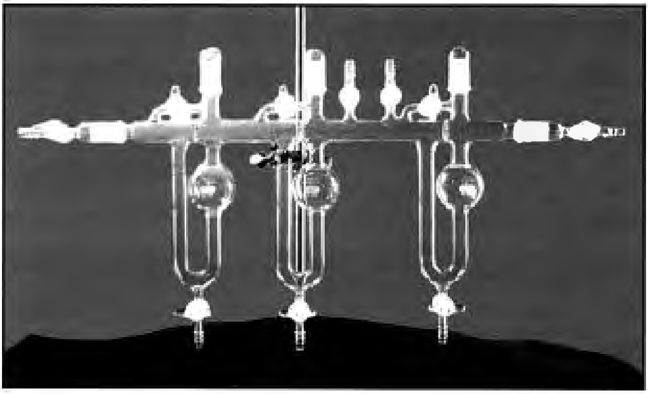

Figure 3. *Gas Washer.*

Operation:

This device can be used to treat any gas stream. The following is one example of how this would operate to purifying house nitrogen.

The first bend is filled with concentrated sulfuric acid. This removes water. The second bend contains concentrated potassium hydroxide and removes any trace acid and oxygen. The third bend is filled with mineral or silicon oil and is used as a flow meter/bubbler. It creates a pressure differential between the incoming and outgoing stream. The stream can be taken off prior to the oil through one of two stopcocks between the second and third bends. Any of the bends can be bypassed with stopcocks to selectively treat the gas stream as dictated by the experiment.

4. The Wisconsin Schlenk Line

This greaseless, rotary-valve manifold was designed by Assistant Professor Shannon Stahl and is shown in Figure 4. It was built in February 2001. This gas/vacuum manifold incorporates a new robust, ergonomic, and greaseless three-way valve design.

Key features:

- Valves are ergonomically positioned to provide easy accessibility.
- Unobstructed visual access to the PTFE/glass seal.
- All valves avoid the requirements for greased seals.
- All valve seats are positioned towards the vacuum source to prevent out-gassing.
- Rugged construction minimizes down-time for Schlenk line repair.

A detailed write-up of this design can be found in the November 2002 issue of *Fusion*, pages 25-27.

Figure 4. *Wisconsin Schlenk Line.*

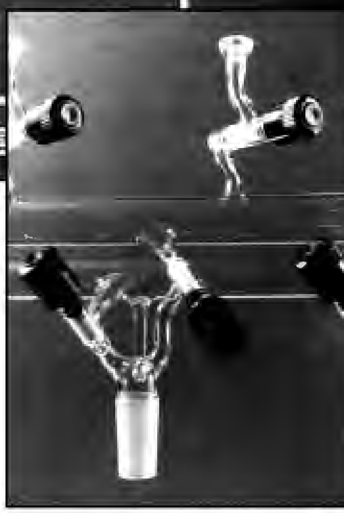

Figure 5. *Wisconsin Schlenk Line – Detail - Valve Arrangement.*

Acknowledgements

I would like to thank all of the above designers for allowing me to highlight their contributions to chemistry and to the Chemistry Department. Anyone who uses theses designs should acknowledge them as well.

I would like to thank the University of Wisconsin – Madison's Chemistry Department for their support and encouragement.

I would like to thank Dr. Thomas Stringfellow/UW School of Pharmacy, for the addition funnel and gas washer photographs.

A Large Modular Hi-Vacuum System for Air Sensitive Manipulation

by

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ABSTRACT

This paper will describe some useful design modifications that can be used in the fabrication of very large hi-vacuum systems. The use of O-ring joints for connecting vacuum lines and their components is not a novel idea; they have been used for years. I like to use the term "modular" to describe this type of hi-vacuum system. The major problem with a system using O-ring joints and hi-vacuum valves this extensively is that it can take many days to completely outgas. If you can live with this compromise, this system design offers some very attractive benefits.

BACKGROUND

In March of 2001, I was introduced to a newly-hired professor who intended to start work on his research in July. After giving Professor Paul Chirik a tour of the glass shop, he made a simple request: "Could you make some hi-vacuum lines for our group?" Professor Chirik showed me several drawings of hi-vacuum manifolds, cold traps, and mercury manometers commonly used in most research labs. He also produced the drawings that illustrated these smaller manifolds connected via # 9 O-ring joints to a 17-foot long, 56mm o.d. main manifold. He told me that he would like to be able to work in the 10^{-5} to 10^{-6} torr range and that he would need a good diffusion pump to supply the vacuum for this system. Little did I know that when the system was completed, it would look like figure 2.

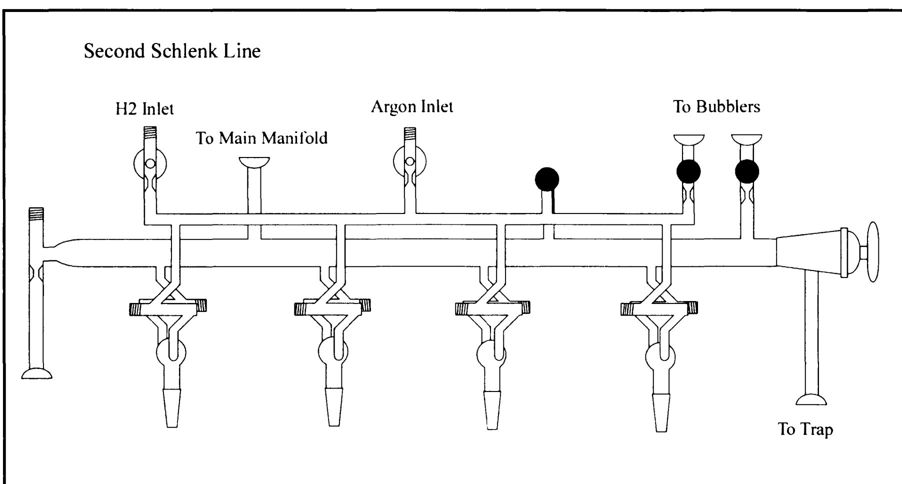

Figure 1. *The first of many project drawings supplied to me by Professor Paul Chink.*

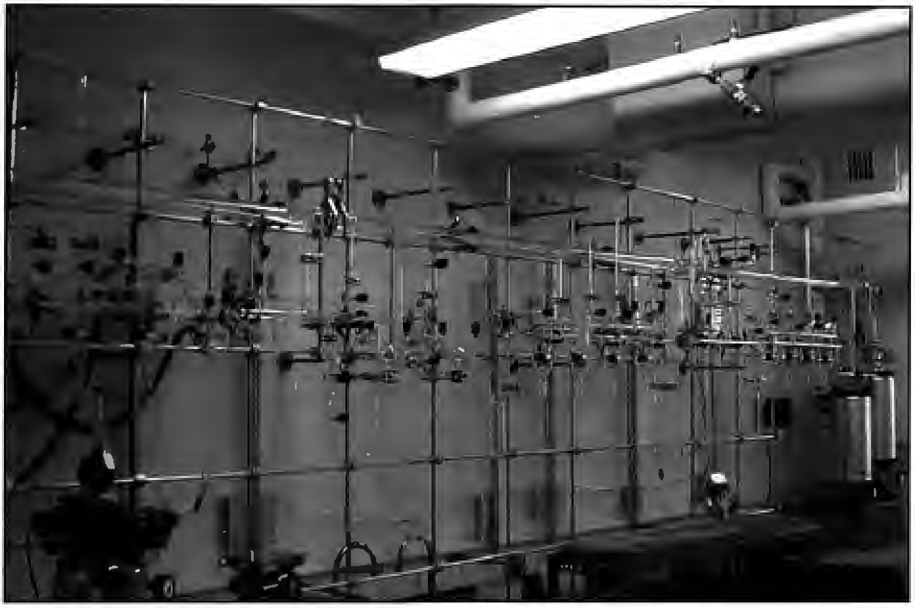

Figure 2. *Photo of the new modular hi-vacuum line at work in the lab.*

The main advantage of using this system design is found when damage occurs and repair is needed on a vacuum system using a modular design. The damaged section of the vacuum system can be isolated and removed, and repair is usually accomplished without disrupting other ongoing experiments. A vacuum system that does not use a modular design is likely to be completely shut down for several days or longer while the repairs are made. Research groups, who must share the use of one hi-vacuum system, do not like to be down for days. This project was becoming very interesting and the professor had my complete attention.

AREA PREPARATION

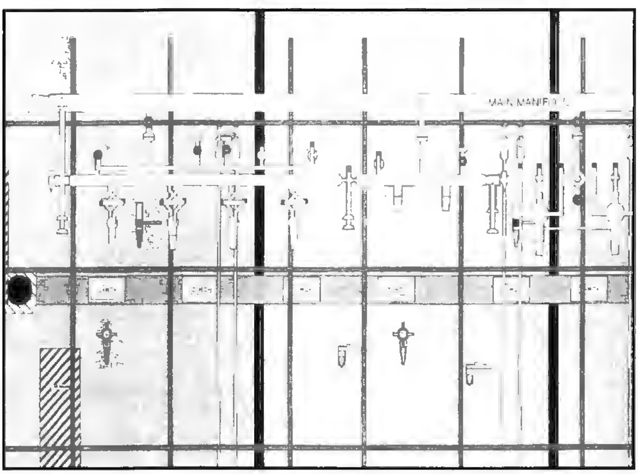

Figure 3. *Original drawing shows the main manifold without #40 O-ring joint connections.*

I asked Professor Chirik to show me the lab that he and his research group would occupy. After arriving at the lab, I was shown the intended location for the hi-vacuum system. There was approximately 22 feet of open wall space available where we would mount the new system. The main problem, however, was that the building of the lattice grid framework could not begin until all utility updates to the room had been completed. There were many electricians, carpenters, pipe fitters, and others involved in the lab renovation project and I was informed that access to the laboratory would be limited for the next three months! The lab renovation issues were thus going to affect my timeline for fabrication of the vacuum system. There is usually a construction sequence that I like to follow when making most of my glassware; I obviously needed to adjust this sequence and be creative in selecting which components to fabricate first.

Diffusion pump

The vacuum line to be built for Professor Chirik would require a diffusion pump that offers fast pump down speed of a large volume into the high vacuum range. My first thought was to build a 3-stage mercury diffusion pump. There are several diffusion pumps of this type being used in our department and they have very good performance. I was hesitant to suggest using this type of pump in the new lab for one major reason. There is always the potential problem of a mercury diffusion pump being broken while in operation. Anyone in the lab at the time of breakage would be exposed to the very harmful vapors produced by the hot mercury. This could turn the usually small problem of breaking a piece of glass, into a major health hazard. There is also the problem of cleaning up a mercury spill. This cleanup would involve many safety personnel and call for the usual investigation and paperwork associated with such incidents.

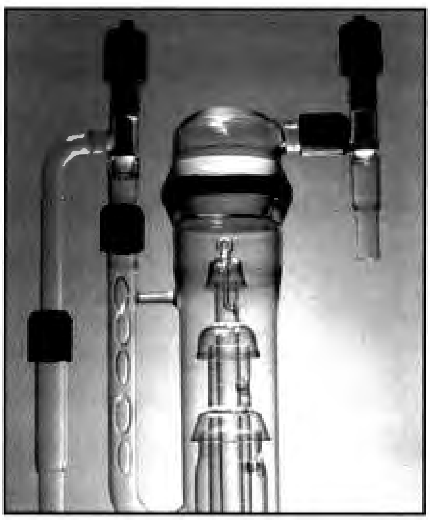

Figure 4. *4" Oil Diffusion Pump as designed by Michael Wheeler.*

While searching for an optimal solution, I started thinking of a technical paper that was published in the 1995 ASGS *Proceedings*. The article, written by Michael Wheeler, described the building of a 3-stage oil diffusion pump capable of working in the hi-vacuum region.[1] As a bonus, all the dimensions and step-by-step instructions were included in the article. The modular design that was used helps to make the oil diffusion pump easier to fabricate than the mercury pumps that I have seen in use. The traps for the pump use standard # 40 O-ring joints and the bypass valves are connected using 1 inch threaded O-ring joints. This design helps to make using, cleaning, and maintaining the pump very easy. I also like the fact that the jet assembly could be repaired or replaced if somehow damaged. The most important feature was that, according to the article, the unit could pump at a rate of 188 liters per second. Through modifications to the jet assembly that were developed by Michael

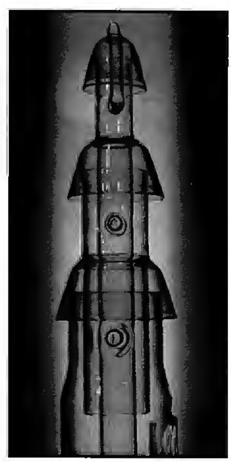

Figure 5. *Through modifications made to the jet assembly, the pumping speed can be increased to 375 liters per second.*

Wheeler, the pumping speed can be increased to 375 liters per second. That is many times faster than the best mercury pumps we have in the department. The following points made the choice to use this oil diffusion pump design an easy one:

- This pump is able to fit into a relatively small space and small hi-speed fans may be used for cooling instead of water jackets if desired.
- If the vacuum is compromised, there will be no mercury spill cleanup or hazardous vapors to address.
- The unit is easy to operate, easy to maintain, and reliable. By replacing the jet assembly, pumping speeds of 375 liters per second can be achieved.
- Total cost of the oil diffusion pump is much less than a comparable size mercury diffusion pump.
- Safety: we will make a safer work environment for the researcher by using an oil diffusion pump. I also do not make the safety people on campus uneasy.

The pumping station uses a large, 1 hp. Welch Duoseal® belt drive roughing pump set on the floor under the diffusion pump location. A double trap system is used that incorporates bypass and relief valves. This allows you to isolate and clean or empty either of the traps without disrupting the vacuum in the system. The use of one-inch threaded O-ring connectors for the bypass valve section would allow us to place the pump into the small area available that we had to work with. Although the pump was one of the first items that I made for the system, it ended up being one of the last items connected. The next photo shows how we squeezed all of the glass into the corner to protect and support the glassware (Figure 6).

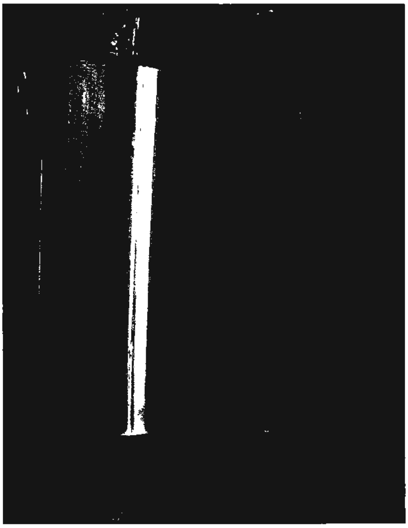

Figure 6. *View of the oil diffusion pump and bypass valve.*

CONSTRUCTION

Manometers

Although I was able to keep a lot of mercury out of the new lab, the manometers used on the system did require its use. The vacuum system needed four double and four single manometers to be mounted behind the lattice grid. I fabricated these and mounted all of them to their Plexiglas supports using small plastic tie wraps. After these were fabricated, I stored them, without the mercury of course, in the glass shop until the lattice grid was finished. You should always consider how to contain any potential mercury spill if the manometers were to be broken during use; I prefer to use the bottoms of one-gallon plastic containers for this purpose. I put these containers under the manometers before they are mounted to the wall. After mounting them in back of the grid, I will fill the containers about 2/3 full with wax. This will provide weight and support for the bottom of the manometers and will serve to catch any spilled mercury.

Next, a 17-foot manifold constructed of 3/4 inch PVC pipe was mounted to the wall in back of the vacuum system. This manifold was connected via a "T" connection to the hood vent system for the room. After the manometers were permanently mounted to the wall, the vent tubes were connected to this PVC manifold. These connections allow the mercury vapors to be carried away safely into the vent system. This can be clearly seen in the next slide (See figure 7). To connect the manometers to the working

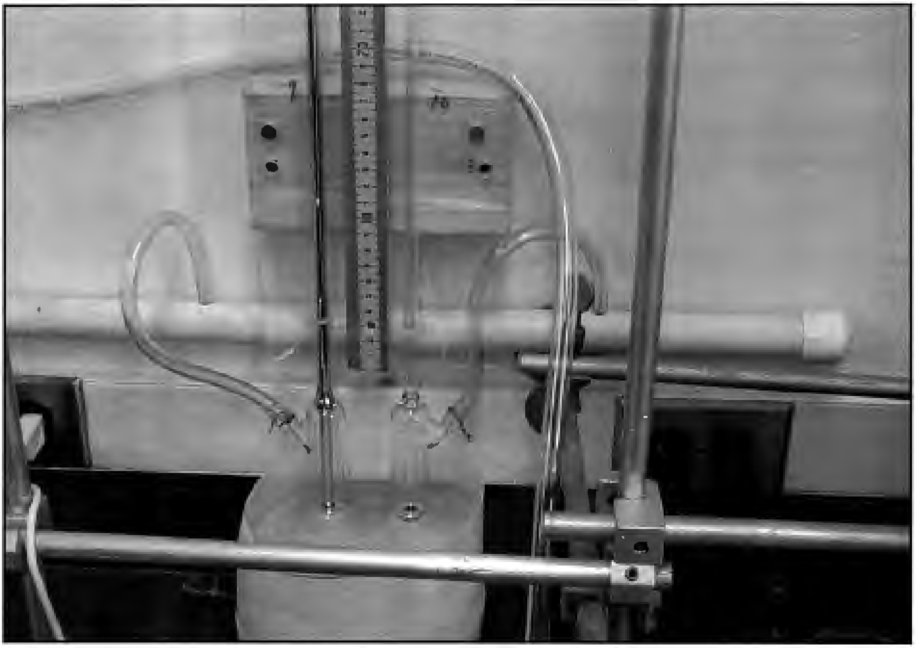

Figure 7. *Close up of the manometers shows how mercury vapors were vented safely away.*

manifolds, I use # 9 O-ring joints. The use of O-ring joints makes it easier for me to remove the manometers for repair or replacement. I guess you could consider these manometers, modular components.

Working manifolds

The next items to fabricate were what I will call the working manifolds. On this system there are three 4 port, one 2 port, and one 3 port manifolds. The 4 port manifolds are very similar to products that can be purchased commercially but with some small and important differences. The gas section has valve connections for introducing

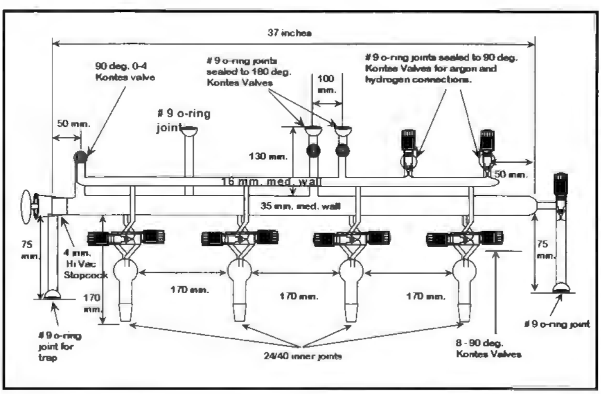

Figure 8. *Drawing of working manifold, note connections for argon and hydrogen.*

two different gasses; Professor Chirik is using argon and hydrogen. We also use 90-degree stopcocks or hi-vacuum valves, with O-ring joint connections, on both ends of the vacuum side of the manifold. We connect each of the different working sections together using LN_2 traps that use # 9 O-ring joints.

Assembly of the working manifolds is not difficult so I will not go into a step-by-step detail. The point I am trying to make is that again we have a design that can be called modular. Each section can be isolated and removed from the main manifold due to the use of valves on each O-ring connection. If one section is broken and in need of repair, it is possible to close all of the valves on that section, remove the traps, and remove it from the main manifold. There is thus little interruption to other group members who may be working at a different section of the vacuum line.

Specialty gas section

Professor Chirik wanted the ability, at a later date, to add gases other than argon or hydrogen to the system. This is why we have a specialty gas section located in the middle of the vacuum system. The opening or closing of the proper set of valves will allow gases to be mixed or introduced into the selected manifold. He may also choose to direct gases into any apparatus connected to one of the 24/40 joints. This section is also modular as it can be isolated or removed from the vacuum line and not disrupt work in progress.

Figure 9. *Close up of the gas transfer section.*

Main Manifold

The grid assembly was still weeks away from completion, but I needed to start work on the main manifold right away. Although the manifold was drawn as one long piece, I suggested to professor Chirik that we could make the main manifold modular by using # 40 O-ring joints. This approach would allow me to make two shorter manifold sections on the lattice grid that I have set up in the glass shop. By using this simple solution to our timing problem, I could transport these sections to the lab at a later date when the grid was complete. We would only be adding one more O-ring joint connection to the original design; he quickly agreed to let me try this idea.

Figure 10. *Photo of work in progress shows #40 O-ring joint connection on main manifold.*

There were other added benefits in using this design that occurred to me as I started to work on the manifolds. After completing the first section in my lathe, I realized that I could use my 5-foot oven to fully anneal each of the sections. My oven happens to have 2.5-inch slots cut on each end of the base frame; these slots allow long pieces of glass to hang outside of the oven while the part inside the oven is being annealed. Using this method, I was able to anneal a 5-foot section, turn the piece 180 degrees, and then anneal the second section. I caution you that the end of the tube that is outside of the oven must be supported to prevent sagging. Although I took care to provide the needed support, I still was able to see a small bit of sagging. I have some ideas on how to prevent this problem in the future, but I will have to wait and try these ideas myself before recommending them to anyone else. Since the sagging was minimal and deemed acceptable, I continued to work on the manifold. Now I had to carefully measure and seal all drop connections, stopcocks, and valves for the working manifolds.

When I annealed these sections, I found that I had to use firebrick, covered with ceramic blanket material to help keep the manifold drop connections facing vertical inside the oven. I also had to use firebrick as a spacer around the border of the oven. This spacer gave me the clearance that was needed for the long drop connections on the manifold; without these spacers, I would not be able to lower the oven completely. This sounds like a lot of work just to anneal a couple of pieces of glass but consider the alternative. Try as I might, I will never get all of the stress out of glass when flame annealing with my torch. Considering the number of seals I needed to make on each section, I feel that I actually saved time overall. I am also sure that better annealing is accomplished using the oven and I do not have to worry about the integrity of the manifold.

In the event a modular main manifold section is broken, the diffusion pump can be temporarily reconnected to the good section of the manifold. If I have to remove the section on the right, I can reconnect the diffusion pump to the left side manifold. For this I can use a # 40 O-ring joint adapter and a length of heavy wall vacuum tubing. If the section on the left is broken, the pump connection remains as is, and I can close off the open end with an O-ring joint cap. If breakage occurs on a solid, one-piece manifold, the whole system would be unusable until repairs were made. If a replacement part needed for the repair is not on hand, the system could be down for too long. This problem can become critical for the research group that may be in the middle of important research activities.

Figure 11. *Photo of # 40 O-ring joints used on LN_2 traps and main manifold.*

Final assembly and start up

At last the laboratory renovation was complete. The room was turned over to me and the installation of the vacuum system components began. I started the installation by temporarily placing the manometers behind the lattice grid. Next I installed the main manifolds and clamped them together using the #40 O-ring joints. I then hung the working manifolds and specialty gas sections exactly as shown on the drawing the professor had provided. I next fabricated the seven traps that would connect each working manifold section to its neighbor. I then had to find room to mount the diffusion pump, LN_2 traps, and the bypass valves. Due to the limited space for these components, a bit of re-design was called for (Figure 12).

Figure 12. *Photo of hi-speed fan that cools the jet assembly area of the diffusion pump.*

Part of what I call final assembly is the fabrication of the argon and hydrogen manifolds (See figure 13). All of the vacuum systems that I have previously made have included only inert gas manifolds, never have I included a hydrogen manifold. The professor assured me that we had a good design so I continued with the installation. I did, however, use my Tesla coil on every inch of both manifolds to make sure that we would not have any leaks. I took extra time to check the hydrogen manifold for obvious reasons.

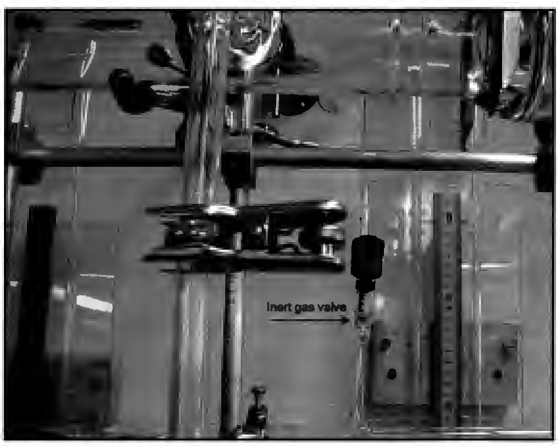

Figure 13. *Photo showing inert gas manifold inlet valve.*

After fitting all of the necessary pieces into their new location and double-checking all of the connections, the system was started for the first time. For the first week, we used only the roughing pump. The vacuum was not what we expected at first so we checked for any undetected leaks with the Tesla coil. I found and repaired two pinholes that were in the system and the vacuum improved to the anticipated level. One of the pinholes was located on the bottom of the ground joint connection used for mounting the McLeod gauge. The second pinhole was on the side arm of a 0-8 hi-vacuum valve used for a drop connection.

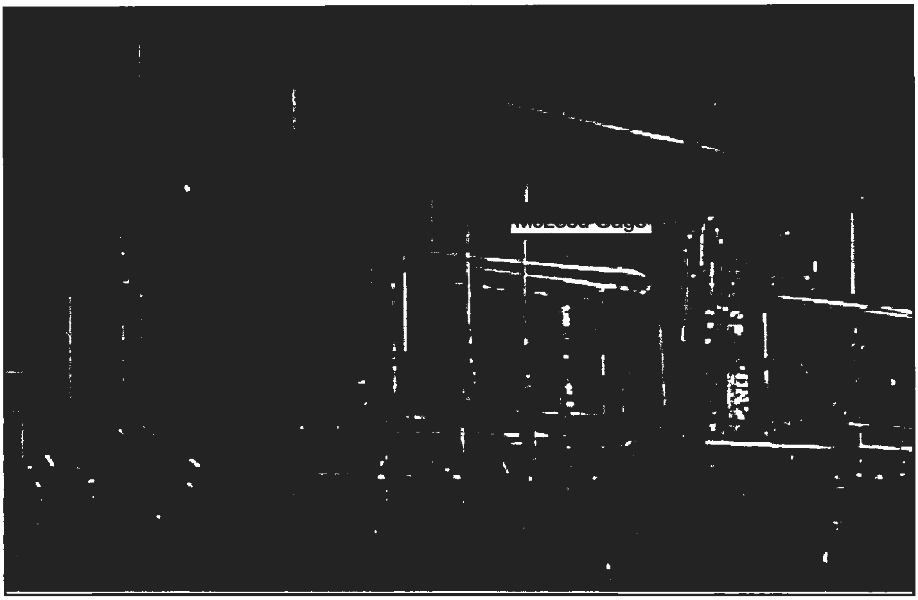

Figure 14. *Photo shows location of McLeod gauge where a leak was detected and repaired.*

This could be a lesson for many of us: do not be complacent when building glassware. I did not check all of my components with my Tesla coil before using them on this system, and for a professional, this was a very embarrassing situation that could have been prevented. Even though you may have made an item a hundred times, a professional should take the time to check every component before using it on any apparatus. Checking glass component quality is a subject for another paper.

After all of this work, we turned on the heater to the diffusion pump, opened the bypass valves, and put the system to work. The pump worked as advertised and the professor was delighted to finally have the system completed and running smoothly. The system now is in daily use and proving to be an asset to the group's ongoing research.

CONCLUSION

Through the use of O-ring joints, Professor Chirik's original design for this vacuum system was already very modular. When the original main manifold design was changed to include the use of the # 40 O-ring joints, I was able to work around many scheduling difficulties. The use of Michael Wheeler's oil diffusion pump and bypass valve design ideas completed the modular concept. Without these changes in design, I would have been forced to completely stop fabrication many times.

Remember that the use of this many O-ring joints and hi-vacuum valves will cause this vacuum system to take a number of days to completely outgas. This vacuum system, however, will only be open to atmosphere by accidental breakage or by not isolating a section that is being removed for cleaning. The professor and his research group live with this compromise and they continue to enjoy the benefits of working

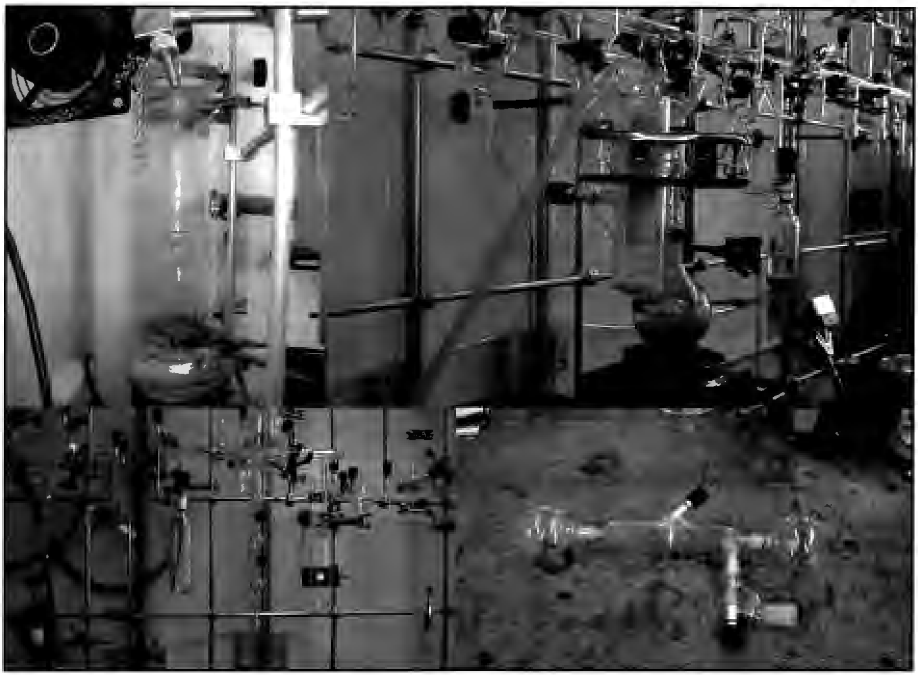

Figure 15. *The Chirik Group is now putting the modular vacuum system to good use.*

with a modular vacuum system:

- **Multiple working manifolds:** There can be several people doing multiple experiments on the system at the same time.
- **Modular design using O-ring joints:** The major components can be taken down for cleaning or repair without the need, in most cases, to shut down the entire system.
- **Two gas manifolds and a specialty gas section:** Provides many combinations of gases to be used.
- **"Wheeler" Oil Diffusion Pump:** High performance, fast pumping speed, modular design, and the safety of using an oil diffusion pump.

Acknowledgements

I would like to thank Professor Paul Chirik, for whom this vacuum system was built, for allowing me to share this information with you. I would also like to thank the Department of Chemistry and Chemical Biology at Cornell University for making this presentation possible. I would like to thank Michael D. Wheeler for allowing the use of photos and references in regards to his oil diffusion pump. And finally I would like to thank my wife Margie and my children, Amelia, Kevin, and Heidi for their help and patience while I wrote this paper.

End note

[1]Michael D. Wheeler, "4" Oil Diffusion Pump," *Proceedings of the Fortieth Symposium on the Art of Scientific Glassblowing* (Seattle, WA, 1995): 24-30.

Sealing a Sapphire Window to Schott Code 8250

by
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ABSTRACT

This paper will discuss how to seal a sapphire window to a 8250 tube for production purposes. While there are other possible methods and techniques for sealing a sapphire window to a 8250 tube, this will explain how to do the procedure for production purposes and with the materials supplied. This paper will also outline some of the problems encountered in this project and the solutions used to overcome them.

In this paper, I will explain the method we use to seal a sapphire window to an envelope made out of Schott Code 8250 using an induction heater. When our company was asked to do this project, we never realized how little information was available and how much experimentation we would have to do. One of the biggest problems was that the customer was providing the materials and was insistent upon Schott Code 8250 tubing, which is not a good match as far as expansions are concerned: Sapphire's expansion is $65x10^{-7}$ and the 8250's expansion is $50x10^{-7}$. Admittedly, the best match for direct sealing to a sapphire window is Corning Code 7520 or Schott Code 8436, but this was not possible due to the customer's requests. I will discuss some of the other issues we came across during this paper.

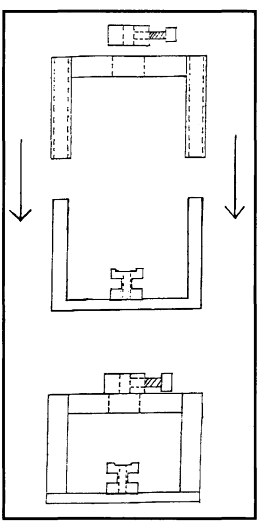

Figure 1

One of the things we had to keep in mind when planning the fixtures and procedures for this job was that this is a high production job, so durability and simplicity were important. We came up with a slide design for a holder for the envelope as illustrated in Figure 1. This allowed for good centering of the tubing over the sapphire window on an easy and consistent basis. A problem we ran into was the material of the holder for the window shown in Figure 2. We first tried graphite and it deteriorated after five windows. Then we used Molybdenum which did not conduct heat effectively. We also tried several different grades of stainless steel which held up better but still deteriorated con-siderably after about a hundred windows. The best material we have found so far is Inconel which conducts heat exceptionally well with very minimal deterioration after several hundred windows. The window holder needs to be machined no deeper than half the thickness of the window and at least .005" larger diameter than the window. We also found that it is important to hollow out the window holder to ensure that the window and material to be sealed to the window heat up quickly and with as much intensity as possible. This will also allow the holder to decrease in temperature rapidly so the envelope can be removed and placed in the oven as soon as possible.

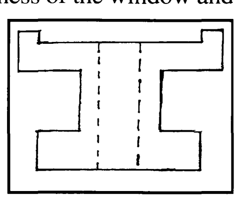

Figure 2

As for the procedure itself, the first step of this procedure is to put the window into the holder. Next, slide the envelope into its holder and bring it down onto the window. A 0.015" spacer was placed on top of the fixture (this number will vary according to the size of the envelope and wall thickness). Next we put a stop onto the tube, engage it with a small thumbscrew, and then remove the spacer; this will allow the proper amount of glass to flow onto the window for a good seal. We then start the 1KW induction heater and allow it to run until the stop comes down to the top of the fixture. Although we have never tested the window fixture within the coil for exact temperature, we must assume that by the time the stop comes down to the top of the fixture, the materials within the coil are approximately 1100°C. When the stop hits the top of the fixture, we set a timer for an additional minute to allow the glass to flow on the window. Without this extra minute, we experienced a large percentage of cracking, because even though the glass was melting down on the window it still had sharp edges as shown in Figure 3. Running the induction heater for the extra minute takes the sharp edges off the glass on the window and eliminates the cracking as illustrated in Figure 4.

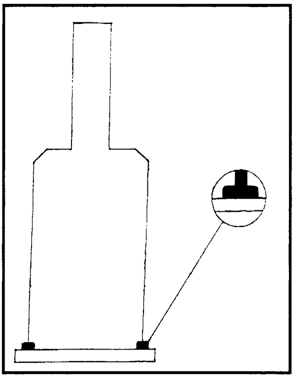

Figure 3

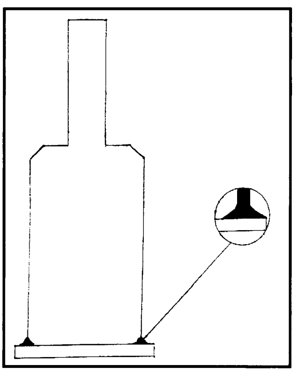

Figure 4

After turning the induction heater off, the window holder is allowed to cool to the point where it is no longer glowing red. The stop is then removed and the slide is pulled up and off the envelope. Next, the envelope is put directly into an oven holding at 480°-490°C, the strain point of the 8250 glass. After all of the envelopes are in the oven, we start their annealing cycle by running the oven up to 510°C, which is just a little higher than Code 8250's annealing temperature, and hold it for ten minutes. We then drop the temperature to 480°C for ten minutes, allowing the envelopes to cool slowly and adjust to the temperature. Next, we drop the oven down to 460°C for 30 minutes, again to allow the envelopes to adjust. Lastly, we drop the temperature down to 400°C for 60 minutes and then turn the oven off, allowing it to cool overnight. This annealing procedure, as compared to our original procedure of running the oven up to 510°C and letting it come right down, has sucessfully stopped the windows from cracking off the envelopes.

I hope that I have helped you understand our procedure for sealing sapphire windows to Code 8250. I have been clear in this paper in admitting that my procedure is based on high production; this procedure could be done less expensively and with less fixtures if you only have a few to do, but in my opinion this procedure is the best way.

References and Acknowledgements

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I would also like to acknowledge and thank Wayne Martin for his help with this paper and procedure.

Small Tank Tetraphenylborate Process Vessel

by
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ABSTRACT

The Glass Apparatus Development Laboratory, of the Savannah River Technology Center, was asked to fabricate a minimum of four thirty-liter glass vessels used to study radioactive salt solutions. During the initial testing, excessive foaming of the solutions was observed in different stages of the experiment, resulting in additional studies to find an acceptable antifoam/defoaming agent. This article is an attempt to describe the steps needed to fabricate the vessel used during testing.

INTRODUCTION

One of the alternatives to processing the highly radioactive salt solutions in the Savannah River Site's Waste Tanks, is to precipitate the highly radioactive cesium with sodium tetraphenylborate concentrate, and wash the precipitate slurry. This process is called the Small Tank Tetraphenylborate Precipitation process (STTP). In the STTP process, soluble ions of cesium, potassium and ammonium are precipitated as insoluble tetraphenylborate (TPB) salts. The resulting slurry, which now contains most of the radionuclides as insoluble solids, is filtered to concentrate the solids. After washing the solids to reduce the concentration of soluble sodium salts in the slurry, the precipitate is processed and incorporated into a glass form in the Defense Waste Processing Facility (DWPF).

To simplify this process, Precipitation Tank #1 is fed continuously with the volume of process water necessary to carry out the precipitation. The precipitate slurry continuously overflows to a second identical Precipitation Tank #2, which serves to increase the residence time for the precipitation process to 16-24 hours. The rate determining step is based on the absorption of the plutonium, uranium, and strontium from the solutions. The slurry exits Precipitation Tank #2 as insoluble solids and is concentrated through a

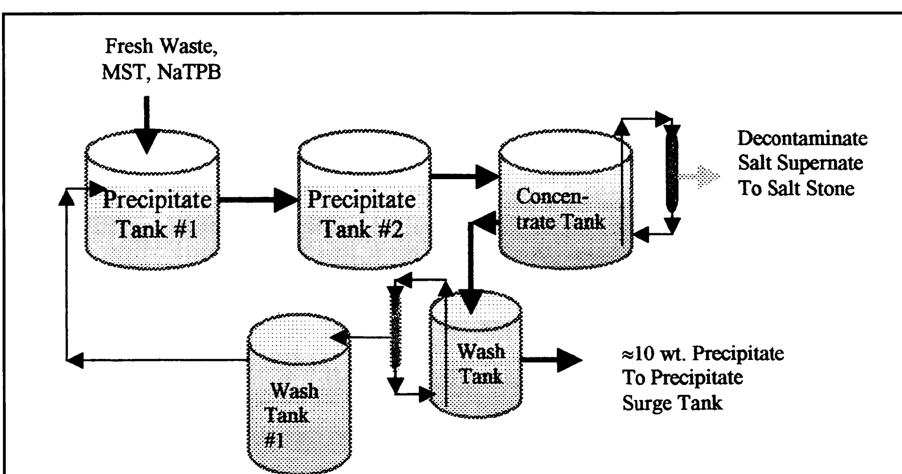

Figure 1

crossflow filter in the Concentrate Tank. The concentrated slurry is then washed with a dilute caustic, using a crossflow filter, to reduce chemical concentrations and to achieve acceptable levels needed for glass production (Figure 1).

During this process excessive foaming was observed in tests conducted using actual Savannah River Site (SRS) radioactive waste. Foaming was also observed during the precipitation, concentration, and washing steps using simulates during testing performed at the Savannah River Technology Center (SRTC). As a result of these observations, an investigation into finding suitable antifoam/defoam agents that could eliminate or mitigate foam generation was conducted by SRTC (Figures 4 and 5). The studies were conducted to develop an antifoam strategy that ensured the maximum and minimum limits needed to prevent foaming during processing of the solutions.

Recently, I was faced with an interesting challenge. It was to make a 30-liter vessel complete with baffles, draft tube, and a large lid containing seven-internal threads for stirring and various instruments readings. Two identical 30-liter vessels shown in Figure I were needed to simulate the precipitation process. One vessel was used to prepare precipitate slurry with antifoam where the other was used to prepare precipitate slurry without antifoam.

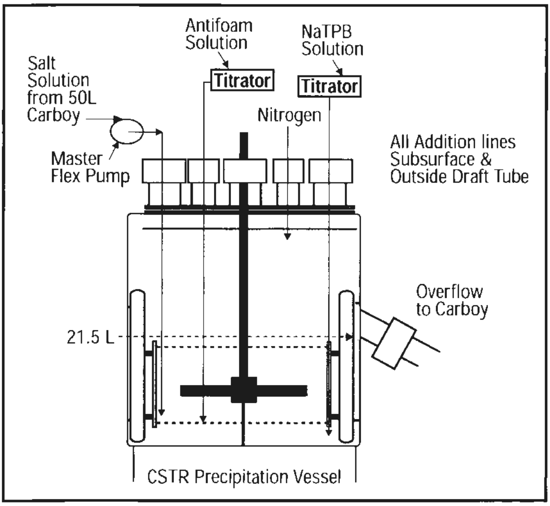

Figure 2

Both vessels were designed with a sidearm sealed at the 21.5-liter level to allow slurry from the Continuous Stirred Tank Reactor (CSTR) to overflow into a collection tank. Good mixing was achieved by using both a four-blade 3.4-inch diameter axial propeller and four baffles (0.94" at 90 degrees to each other) in the tank. The solutions were pumped into the reaction vessels using calibrated peristaltic pumps and flexible tubing. Nitrogen Gas was used to purge the vessel, preventing a flammable mixture from accumulating in the vapor space. This was positioned above the liquid or foam level and was controlled by a calibrated gas flow meter. The antifoam solution was metered into the (CSTR) using an automatic titrator at a concentration of 100ppmV. The agitator was maintained at 750 rpm during the duration of the experiment except for a brief portion of the test, where the agitator speed was increased to 1000 rpm to determine if the slurry would foam. At the end of a twelve-hour shift, the feed pumps, agitator, and nitrogen purge were stopped until the next day.

After the experiments were completed, all the concentrated slurries (with or without antifoam) produced by the experiment were stored and allowed to settle. All the insoluble solids in the concentrate slurries containing the antifoam agent settled to the bottom of the storage container. This observation demonstrated results of the wetting agent on the physical properties of the precipitate slurry.

PROCESS VESSEL

The Glass Apparatus Development Lab of the Savannah River Technology Center was asked to fabricate four identical 30-liter vessels as in Figure 2 for testing of the CSTR Precipitation System. The vessel design provided an airtight seal permitting a continuous nitrogen gas purge during testing. The first step in manufacturing the vessel was to decide the most practical way of sealing the baffles and draft tube into place. The customer requested that the four baffles rest no more than 1/8th inch from the sidewalls and bottom of the cylindrical jar and measure approximately 16 inches overall. The baffles or plate glass were purchased in 4-foot lengths and measured one-inch wide x 3/8 inch thick. These were later cut to 16 inch sections and ground to provide rounded ends using a belt-grinder equipped with a 240 grit diamond belt. The 7mm rods, used to connect to baffles, were cut 3 inches and sealed onto the baffle plates using a Bethlehem bench burner. Each baffle was placed into a hot oven after sealing the top and bottom halves to avoid unnecessary breakage. The draft tube was made from 130mm standard wall tubing measuring six-inches overall. This was used to represent the surface area consumed by the cooling coil in the simulate storage tanks.

Figure 3

To begin assembly of the inner unit, cut the 130mm tube six inches overall and mark 1$^1/_2$ inches from the top and bottom of the draft tube using a chalk marker. The draft tube is then secured onto a holder using Waletex High Temp Glass Tape. Next, seal all eight of the 7mm rods, one at a time, using the marks as a guide and anneal. To obtain the proper height inside the vessel, a spacer is placed under the draft tube and held in place using a smaller inner tube. This is also extremely helpful when aligning the baffle and adds stability to the unit. To provide a flat sealing surface, 7mm rods are attached to the top and bottom of the baffles. The top rods are bent 90 degrees and cut 1/8th inch from the outside edge of the baffles. The bottom rod is bent downward 180 degrees from the previous bend and is cut approximately 1/8th inch, measuring from the end of the baffle (Figure 3). Once all of the baffles are sealed, the entire unit resembling a spaceship, is placed inside the vessel for final adjustments before being returned to the annealing oven for stress removal.

The next step is to begin assembly of the test vessel. Start by securing the jar into the lathe and running the quartz-annealing oven to 850 degrees centigrade. This will allow the temperature of the oven to return to the desired 565 degrees after completing the top or bottom section and placing it into the preheated oven. I chose this temperature at random and it worked great. I used a Litton EE lathe during the assembly of the jar equipped with a Universal Planetary three-jaw chuck with twelve-inch extension arms. To provide ample heating, a 12 fire 7-jet stainless steel, swivel lathe burner was supplied with 15psi of oxygen & hydrogen. Kevlar gloves, sleeves, and a reflective face shield were used as protection equipment during the assembly of the vessel.

Place the baffle unit into the jar end slowly and carefully secure the jar using the twelve-inch extensions. Preheat the bottom section for approximately fifteen minutes. Next, valve off the gas and air burner and begin heating using the lathe burner with the oxygen and hydrogen mixture. Heat the bottom of the jar until it glows a pale pink color. Reduce the intensity of the fires while sealing the four rods into place using a Victor hand torch. Once all of the rods are sealed, raise the oven hood by pressing the remote switch in the upward position. Allow the oven hood to open to the highest point, extinguish the lathe burners, release the vessel from the lathe chucks, and place it in the annealing oven. The temperature in the oven will continue to fall once the vessel is placed inside and the hood lowered. Watch the oven closely until it reaches 565 centigrade and allow the vessel to soak at this temperature for around 45 minutes. Switch the oven to the off position and allow the oven to ramp down slowly until room temperature is reached.

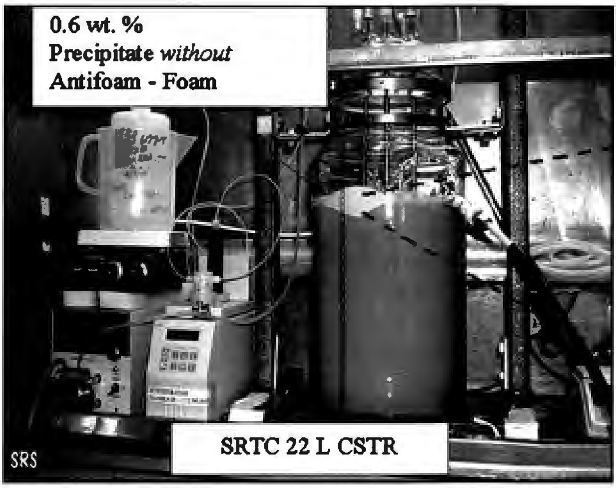

Figure 4

To assemble the top section, the vessel is secured into the headstock side of the lathe using the twelve-inch extension arms as before. Place the 200mm Duran O-ring flat ground flange into the Universal Planetary three-jaw chuck on the tailstock side of the lathe equipped with graphite spools. As before, ramp the quartz oven to 850 degrees centigrade and place it on hold. Preheat both sections at once for a minimum of fifteen minutes before extinguishing the gas and air burner.

Start by shaping the O-ring flange to a 45-degree angle using the lathe burners with the oxygen and hydrogen mix. Roll the top close to the vessel during this operation to permit the radiate heat from the burners to keep the top as hot as possible. Next, shape the vessel in the same manner. Position the O-ring flange so that it is almost touching the vessel and heat until both edges have begun to sag. Lightly touch the two together allowing the lathe burner to continuously heat the seal as you slowly work them together. This allows the vessel and the flange to seal from the inside first thus resulting in a complete seal. Once this is done, reduce the intensity of the burners and seal the rods into place. Alternate between sealing a rod and heating the vessel to assure that the proper temperature is maintained. After sealing all four of the baffle rods, pull a hole in the vessel above the 21.5-liter mark. Seal the #15 Ace thread at a 75 degree angle and reheat the vessel before placing it in the annealing oven. Use the same procedure as before, allowing the completed vessel to soak for forty-five minutes at 565 centigrade.

VESSEL TOP

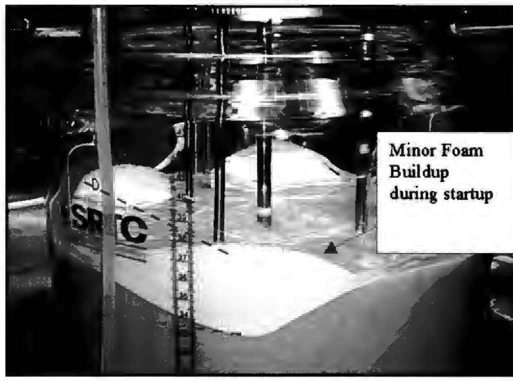

Figure 5

To begin assembly of the kettle top, collect all of the necessary fittings to be used in manufacturing the lid: 1) 6-each #15 internal threads, 2) 3-each #7 internal threads. Always plan for breakage during assembly by prepping one extra of each fitting. This may save you from scurrying for an extra fitting during assembly and possibly prevent a catastrophic break in the flange top. Pull a slight taper on each fitting and place on a heated ceramic hot plate. Next, take a chalk marker and draw the location of the fittings to be sealed on the kettle top. Secure the kettle top into the headstock using the Universal Planetary three-jaw chuck supplied with graphite spools. Begin heating the top using the gas and air Bunsen burner for approximately fifteen minutes. Next, valve off the burner and position an 8-fire, seven-jet lathe burner toward the outside edge of the dome top. Begin by pulling holes, the appropriate size for the fitting, in the dome top using a Victor hand torch. Once the first hole is pulled, heat the top portion of the dome until it becomes a rose color. Alternate between reheating the top and pulling the holes until all of the holes are made. Seal the center thread first to minimize the risk of warping and cracking when attaching the other fittings during assembly. Continue with the same heating sequence as before by heating the top after each seal is made. This should keep the top at a constant temperature and prevent hot spots or excessive strain accumulation in the lid. Once all of the fittings are sealed and aligned, place the completed unit into a hot oven and anneal at 565 centigrade for forty-five minutes.

SUMMARY

The completed laboratory scale units performed very well during testing. This test verified the abilities of the anti-foam agent when used during the STTP process precipitation, concentration, and wash steps, using the KTPB slurry. The vessels enabled researchers to observe the reactions of the solutions during normal and extreme agitation with and without the antifoam agent. One of the most important conclusions reached was the importance of minimizing the introduction of gas into the slurry during processing whenever possible.

Acknowledgments

I would like to thank the Savannah River Technology Center for their continuous support, especially Byron Williams, Mark Lawson, and Jeff Siler for making this presentation possible.

Completed Process Vessel

Solvent Assisted Flavor Extractor

by
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ABSTRACT

The Solvent Assisted Flavor Extractor (SAFE) unit is for the isolation of volatile from non-volatile material. It is composed of five sections that are connected together through a series of steps to yield a small inclusive unit ideal for aroma researchers.

A Solvent Assisted Flavor Extractor (SAFE) is used for the isolation of volatile material from non-volatile material. Essentially the SAFE unit separates the aroma (smell) from taste. In this case, the solvent is water and the product is coffee. At first glance this apparatus appears very interesting to fabricate. But a closer look reveals a complicated assembly of many internal tubes that became a careful game of problem solving the method of fabrication.

- Five sections: 500ml reservoir, dry ice trap, middle section, two receiving flasks legs
- Reservoir: 29/32 outer joint on 60mm tubing with roto-flow valve
- Dry ice trap: 48mm o.d. and 60mm o.d. tubing
- Middle section: #18 GL thread connector, two hose connections, an internal disk (with two 5mm tubes), internal blade assembly
- Bottom legs (2 each): 29/32 inner extended joints, 8mm tubing

Reservoir: 60mm o.d. standard wall tubing
29/32 outer ground glass joint
10mm rod

Figure 1

Seal 29/32 joint onto the 60mm tubing with the neck as short as possible. Seal on a short piece of 10mm rod trimmed at 40mm length (Figure 1). Flame anneal.

Pull off 60mm tubing about 6 1/2" from the bottom of the joint; slump the glass at an angle 6 inches on one side (Figure 2) and 6 1/2" on the other side (the same side as the upper rod seal); then blow out a hole at the bottom side of the tube; seal on valve (Figure 3). Flame anneal and set aside.

Figure 2

Figure 3

Dry Ice Trap: 48mm o.d. standard wall tubing
60mm o.d. standard wall tubing
14/20 rotovis outer ground glass joint, cut at 20mm below joint

Form a round bottom end on the 48mm tube and trim at 7 3/8" total length; flare end slightly to 56mm o.d.

Dewar seal: Support the 48mm tube on the inside using a stainless steel pipe wrapped with high temperature cloth. Seal the 48mm tube to the 60mm tube; heat and flare the 48mm o.d. tube to the 60mm tube (Figures 4 and 5).

Figure 4

Figure 5

Flow in seal and blow out a hole near the top of the dewar seal. Seal on 14/20 rotovis connector, remove strain (flame anneal), and seal on 10mm rod (40mm length). Flame anneal then remove stainless steel support. Pull down 60mm tube about 20mm below the inside 48mm tube round bottom (not shown). Slump the bottom of the tube slightly at an angle towards one side (like the reservoir glass piece). Blow out a hole (aligned with the upper rod seal) and seal on 15mm tubing, 40mm length (to be sealed to the main body later).

Upper Body Construction: Top section: Internal Disk with 5mm tubes (not shown, looks like a funnel with two drip tips): flat bottom on 25mm o.d. standard wall tubing; blow up two bubbles to seal 5mm tubes; blow out one hole, seal 5mm tube, then blow out and seal the next tube; seal two pieces of 5mm tubing to 22mm flat; angle slightly outward. Cut 5mm tubes at 21mm, flare ends slightly, flame anneal. Cut off 25mm tube with 5mm tubes; trim to a disk shape to fit inside the 25mm tubing.

Support the disk inside the 25mm tubing and seal (Figure 6). Fire cut the 25mm tube 20mm above Hopkins seal. Seal on the GL18 threaded connector (Figure 7).

Figure 6

Figure 7

Seal on medium hose connection above the Hopkins seal (Figure 8). Seal on two 10mm knobs on each side of hose connection (Figure 9) and remove the 25mm tube 30mm below sealed disk, flare to 35mm o.d., seal to 48mm o.d. tubing, (see photo under rod supports), seal on the second medium hose connection (below first HC), and flame or oven anneal.

Figure 8

Figure 9

Blade Assembly: Middle Section: Prepare blades: flat wedges using rod ~10mm x 15mm x 3mm thickness. Seal 4 blades on 1/2" medium wall tubing, angle blades right side up and left side down, trim to fit inside 35mm tubing, flame anneal. Move upward on the 1/2" tubing about 33-35mm seal next section of 4 blades, trim. Bend 1/2" tubing above blades about 20mm up from blade section at a slight angle around 45 degrees. Trim and flare slightly (Figure 10) to fit inside 35mm tubing with a round bottom end. Trim other end of 1/2" tubing 15mm below blades and fire polish; anneal.

Figure 10

Support blade section inside 35mm tubing with round bottom; support blades with a 8mm tube holder sealed onto the rubber stopper. Seal in top ringseal blowing up a slight bubble to seal to later (Figure 11). Seal an 8mm tube on the end. Seal in the first set of blades near the top (Figure 12), then the next set (Figure 13).

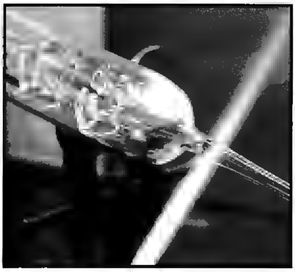

Figure 11

Figure 12

Figure 13

Remove and seal off the 35mm tubing about 35mm below last set of blades and seal on 6mm rod. Bend the rod 10mm at a 90-degree angle, trim rod at 15mm length, to fit inside 48mm o.d. tubing. Reheat 8mm tubes and bend to fit inside the 48mm tubing; anneal (Figure 14).

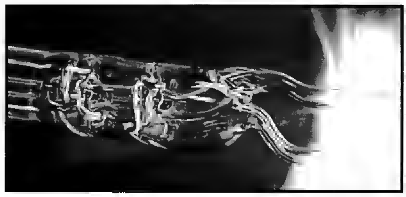

Figure 14

Rod Supports: Support blade section inside the 48mm o.d. tube. It is very important to pay careful attention to the alignment of the 8mm tubes when sealing the top (internal) rod brace (Figure 15). The 8mm tubes are folded inward as shown in the photo but they will be opened up and adjusted later to fit properly inside the lower legs (the 29/32 inner extended joint tubes) that will fit like sleeves over the 8mm tubes. Center the blade assembly, flame anneal and start heating the upper area.

Figure 15

Heat the rod on the reservoir and seal to the middle section (Figure 16). I use a cork as spacer to rest the reservoir while sealing the rods together. Next, heat the dry ice trap and seal to the middle section (Figure 17). Align the pieces and anneal immediately.

Figure 16

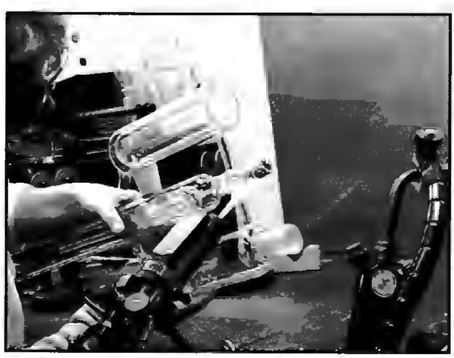

Figure 17

Lower Level Assembly (Bottom Legs): Once cool, trim the 48mm tubing about 6 1/2" down the straight edge of the tubing (Figure 18).

Figure 18

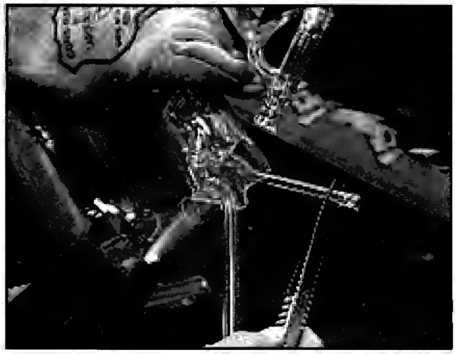

Figure 19

Using an actual size template (blueprint) of the apparatus is necessary to ensure that the tubes are at the proper angles. Heat and bend the 8mm and 9mm tubes in alignment where the 29/32 legs will slip over the tubes (Figure 19). Bend additional 8mm tubes that will be sealed onto the existing 8mm tubes (not shown).

Additional parts: Left leg section: Bend 8mm to template angle; measure 110mm of straight edge and bend slightly. Trim end of 8mm tube about 45mm past 110 mark and flare end slightly. At the 45mm mark from the end, hot stick a rod seal to seal to later. Bend another 8mm tube with ends rounded off and side hole blown out, Total length 80mm with a small rod brace on bottom, rounded end. Measure on template and seal rod brace together. Attach 8mm tube to rod brace where the tubes are practically touching. Check to see that the 29/32 drip joint slips over the tubes.

Right Leg: Bend 8mm tube on middle section aligned with template. Trim at 110mm from the 35mm tube and round the end. Blow out a side hole, flare and add small rod brace. Attach second piece of 8mm tube to template150mm length. This 8mm tube should be rounded on one end with a side hole blown out for the seal and the other end bent slightly towards the center, approximately 100mm from the rounded end. Add the rod seal at the bend and trim. Flare end open for the small ring seal later in fabrication. Attach the second piece to the fixed piece, check outer drip joint and slip over tubes.

Shape the 48mm o.d. tubing before sealing on the 29/32 inner joint sleeves. Contour the outside of the tubing to fit the 29/32 joints (Figure 20). There will be open areas that will be filled in with rod but shape and remove as much glass as possible to minimize extra glass in the seal.

Figure 20

Add the 8mm and 9mm tubes, flame anneal and set up in rack.

Lower receiving flask portion: Outer legs: 29/32 inner joint sealed to 25mm tubing. Prepare two pieces: form a flat bottom on the 25mm tube, measure 120mm down including the joint and trim 30mm above the joint (Figure 21).

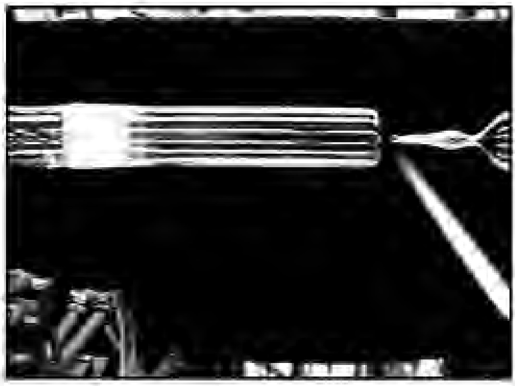

Figure 21

Clamp glassware onto a rack for ease of alignment (Figure 22); support the 48mm tube with leg portion upward, one clamp on the 48mm tube, and clamps on each 29/32 inner extended joints sleeves positioned at the proper angles. Begin heating the 48mm o.d. tube and start filling in the gaps with rod (Figure 23). Once it is air tight, move on to heating the ringseals in the 29/32 joints. Once all the ringseals are done, flame anneal and then anneal in the oven.

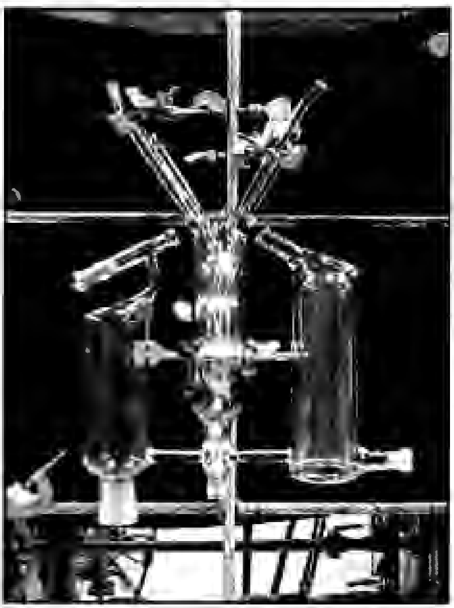

Figure 22

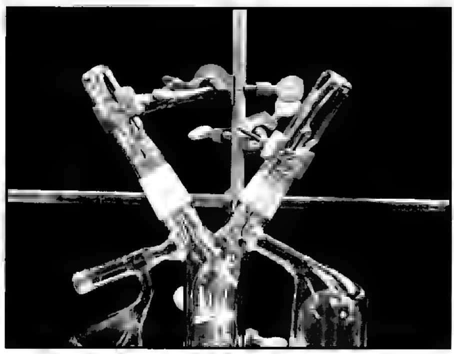

Figure 23

The S.A.F.E. Unit in the Laboratory (Figure 24).

Figure 24

Tools: The Extra Hands of the Glassblower

by

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ABSTRACT

This technical paper presents some of the tools that I have designed and used over the years, notably: use of the side grinding wheel to make custom size disks or windows, some various attachments for the chucks, a quick raising tool holder for hot forming, and a new type of outer ground joint holder. Also illustrated are some practical uses of these tools. This paper demonstrates how, with good tool design and creative thinking, one can save a lot of time while building complicated glassware.

GLASS BLOWING TOOLS

1 - Using the side grinding wheel to make custom size disks or windows.
2 - Various attachments for the chucks.
3 - Tool holder for hot forming.
4 - Joint holders.
5 - Good planning in fabricating glassware to save time with appropriate tools.

GRINDING DISKS

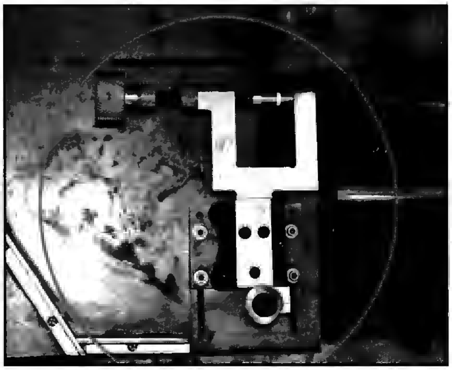
Several types of disks can be ground precisely with the following tool to make windows (make your own from plate glass, any thickness and of any material) or fritted disks (unusual sizes, or special sizes to fit snugly into tubing).

PART 1

Using the side grinding wheel to make custom-size disks or windows

Grinding disks on side grinding wheel
This device will allow the user to precisely grind glass disks or fritted disks to any size necessary.

Table that fits on top of cutting wheel
This tool consists mainly of a fork that holds a device that will keep the piece of glass in place and that permits both rotation and horizontal movement.

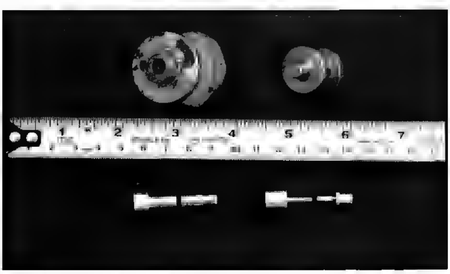


Grinding on side grinding wheel

The glass being ground is held by two aluminum disks that can rotate perpendicular to the side grounding wheel.

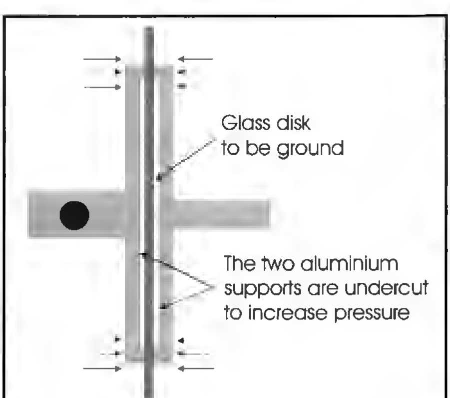
Grinding disks on side grinding wheel

Sets of two aluminum wheels are used to hold the glass disk that will be ground. They range from 2" in diameter to one as small as 1/8".



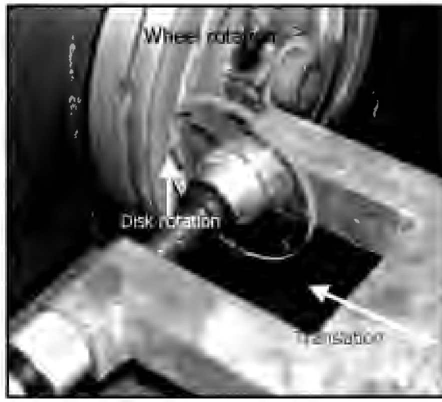
Disk holder

It is only necessary to change the disk holders to an appropriate size, clamp the disk, and start grinding.



Grinding windows

While turning against the rotation of the side grinding wheel, simply turn the calibrated dial to move the assembly towards the wheel, and begin the grinding.

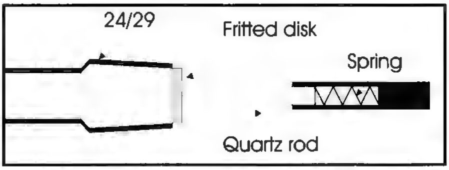


Grinding fritted disk

Use the same technique as for windows.

Special use of ground disks

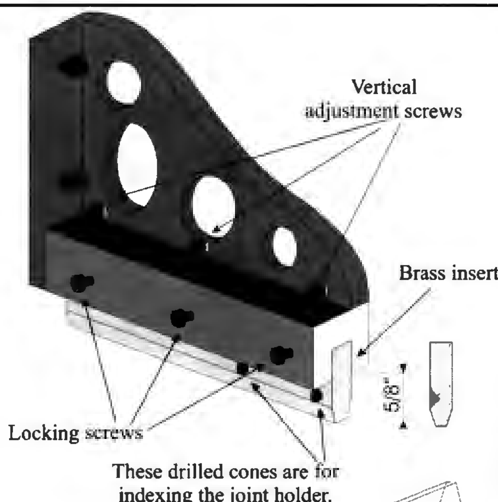
One can attach fritted disks directly at the end of a ground joint. It is particularly useful when making a "rotavap" trap to be used when bumping occurs, or for a vacuum adapter to be used when evacuating powdery material.



PART 2

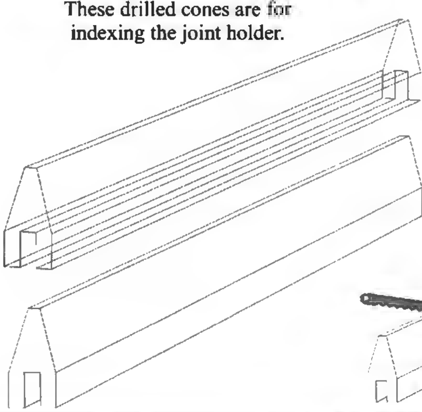
Various attachments for the chucks

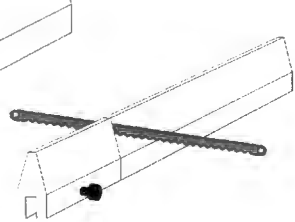
Over the years I have come up with a series of little holders that fit over the insert on the top jaws. This is done both to protect them and mostly to save time. It is faster to clamp in place a small aluminum holder than to wrap layers upon layers of tape.



An aluminum bar is milled from one single piece of metal. It is then cut in three sections of appropriate size. Each piece is fitted with a screw to hold onto the brass insert which has been made longer for that purpose.

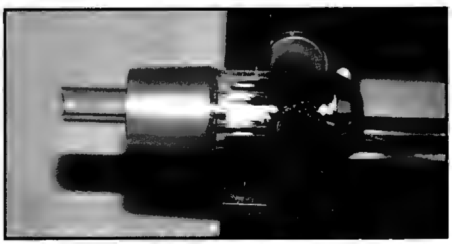
A bar of aluminium is milled in one section of 12" +/- (Drawings 1, 2) then is cut in sets of 3 pieces of same length.
And each piece is fitted with a screw.(Drawing3)

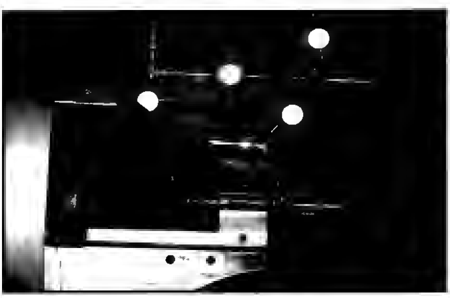




I have had our machine shop make a large array of different size holders. I use the appropriate one for each job.

Handy to repair an otherwise difficult-to-hold piece.

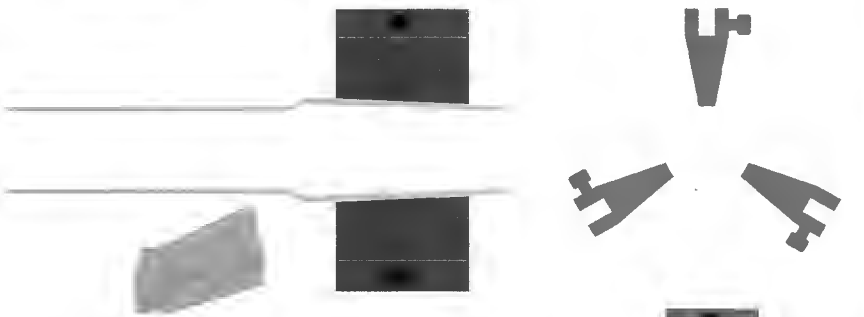




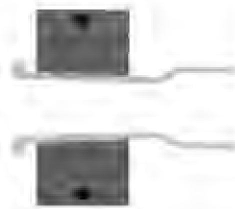
I have made some holders out of wood for a softer contact and thermal security. The wood will eventually become like charcoal.

For very delicate material like NMR tubes, I have made some holders out of teflon too. They simply snap in place.

In this example, I use a very small holder to support a stopcock on one chuck and a modified holder to hold a standard taper joint on the other chuck.

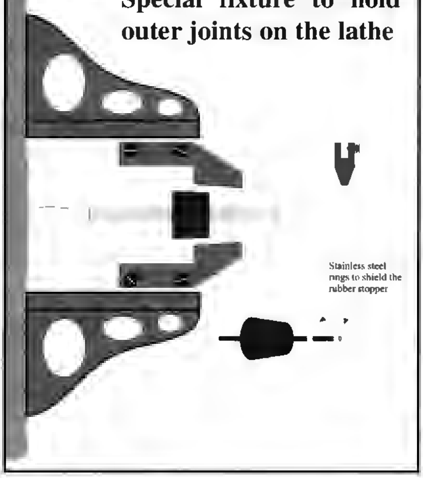


Perspective exaggerated for clarity



If one wants to hold ground joints on the lathe, cut off three sections then cut them tapered to match the angle of a standard taper joint. The same fixture can hold both inner or outer joints (if the outside surface of the outer joint is parallet to the inside surface).

Special fixture to hold outer joints on the lathe



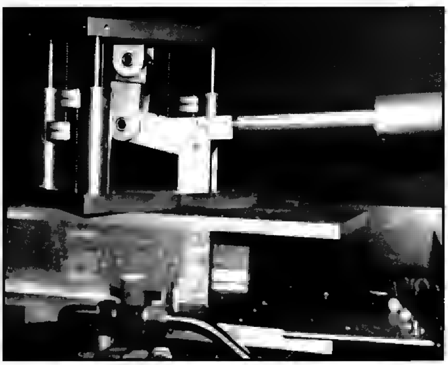
PART 3

Special jack and supports for hot forming on the lathe.

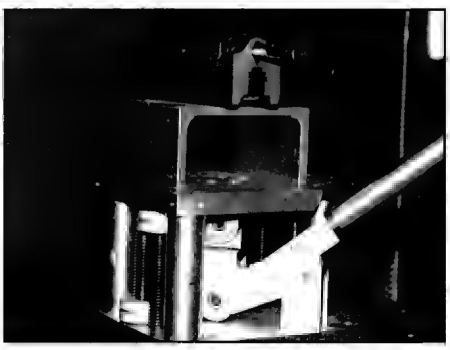
Tool support

Front view of one jack. This one was made by our machine shop with recirculating bearings; it is very smooth. The real nice part is the handle that will lock the tool in position with a slight twist of the handle.

Same tool 3/4 view to show height adjustment screws and detail on handle lock.

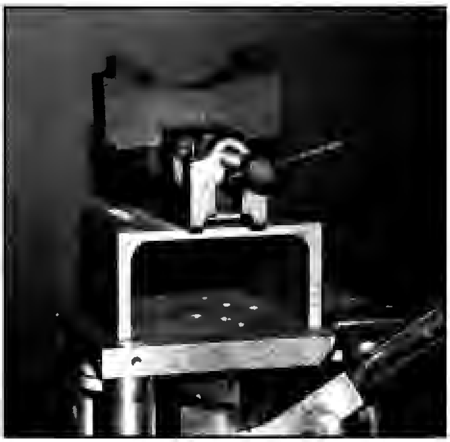


The small vice will hold the graphite tools.

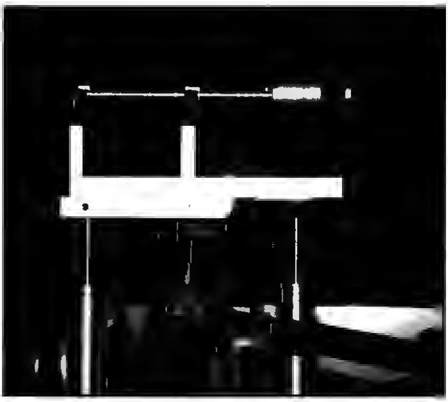


With separatory funnel shaper.

With 100ml round bottom shaper.

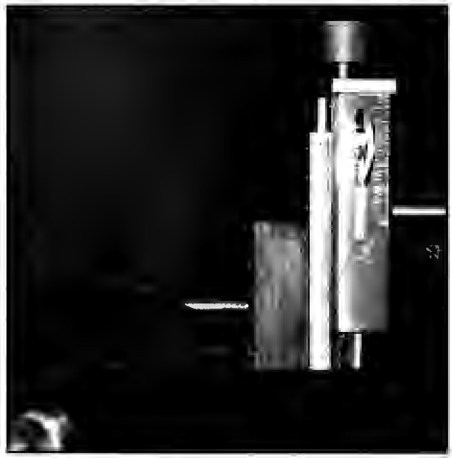


With special shaper to make 100mm flanges with O-ring groove.



Making a 100mm flange with O-ring.
On the right side one can see the special tool that will press the flange against the roller and the small knob that will create the groove for the O-ring.

Enlarged view of flange shaper.
One can see the graphite rod that will open up the inside of the flange, and the small rounded graphite that will make the O-ring groove.

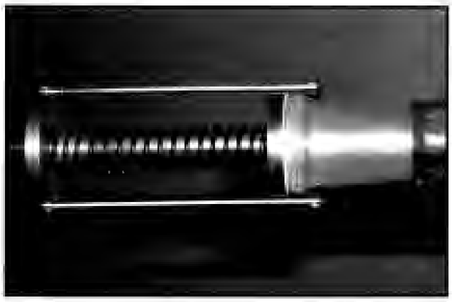


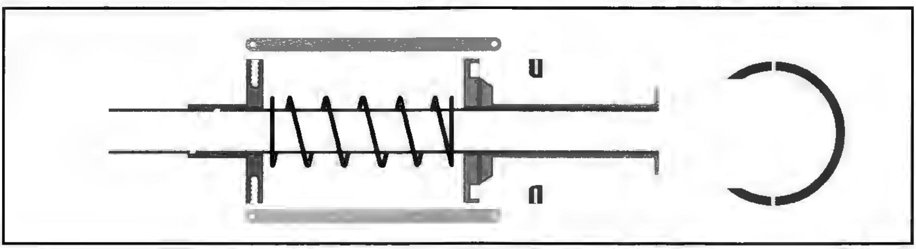
PART 4

Joint holders for the lathe or bench work

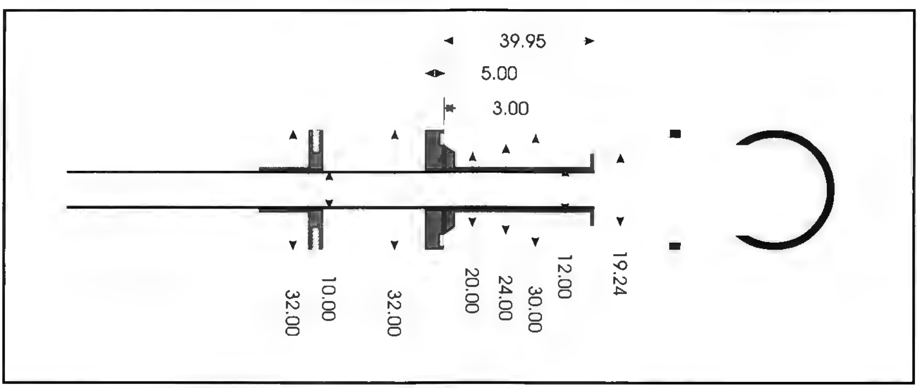
Ground joint holders

I have designed some holders for outer member ground joints.

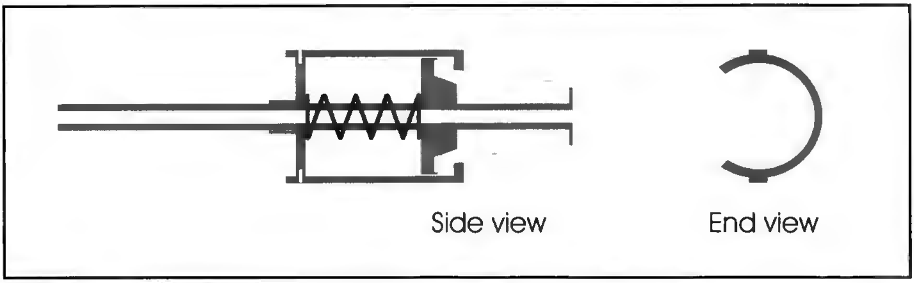


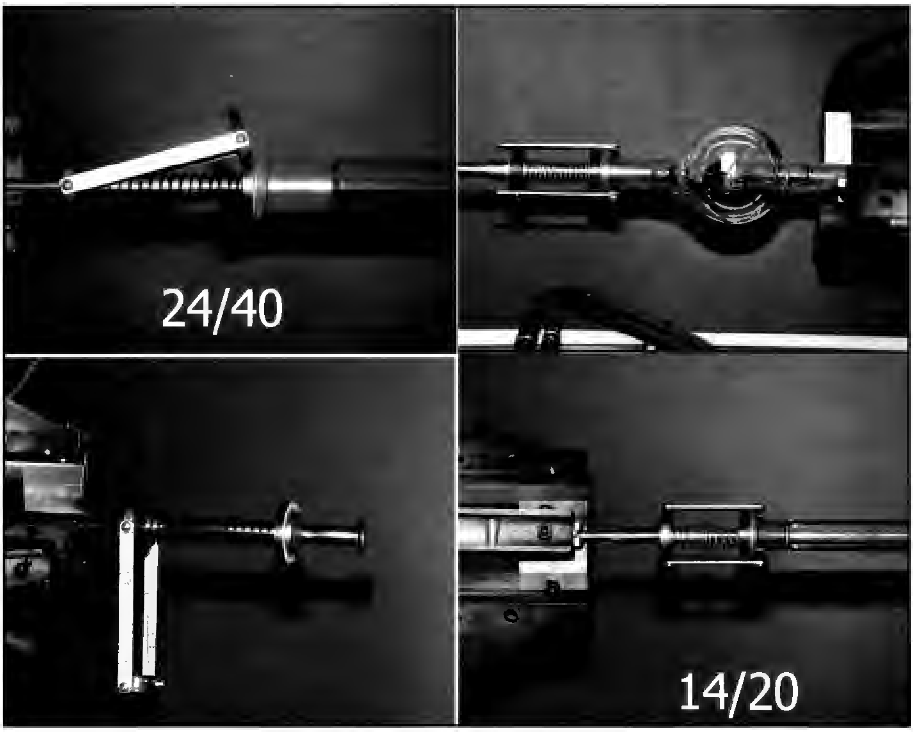


Measurements for 24/40 ground joint holder



One can see from this drawing that the ground joint is held against the teflon washer from the pressure of the spring. The front ring is there only to stop the heat from burning the ground part.

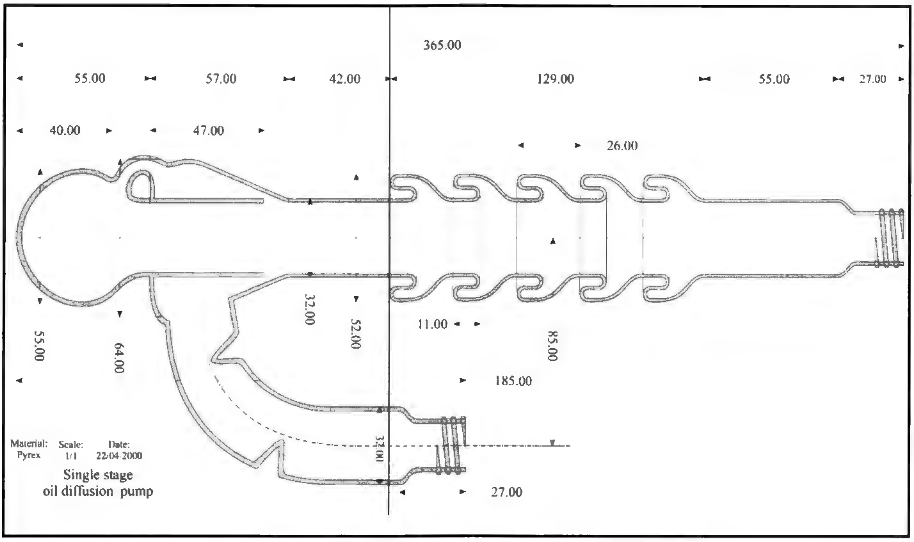




PART 5

In the following series of pictures, I would like to demonstrate how one could save a lot of time while building some complicated apparatus with careful planning and a judicious use of tools. I have chosen the construction of a single stage oil diffusion pump to illustrate the idea.

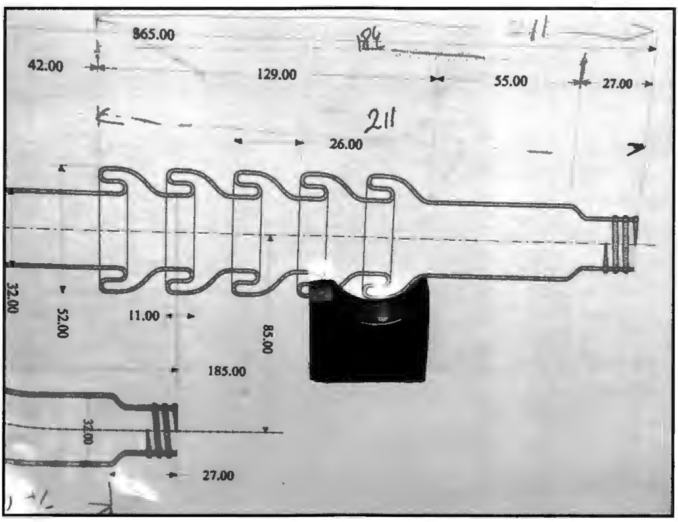
I have decided in this case that the bottom of the lowest bulb would be the center of the reference place. Each mold for the piece will be made from that reference point.



Blowing the fractionating bulbs

Step 1

Using a 1/1 scale drawing, I cut the graphite shaper that will be used to make all the bulbs.



With the first graphite shaper, I will blow the first bulb.

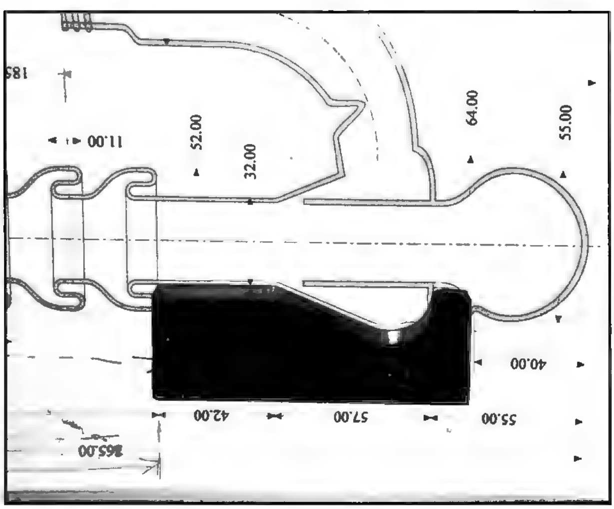
I pushed in the bottom part of the bulb and I **placed the tool against the bottom** of the first bulb; I am now ready for the next one.

Three more bulbs have been formed in the same fashion as previously done.

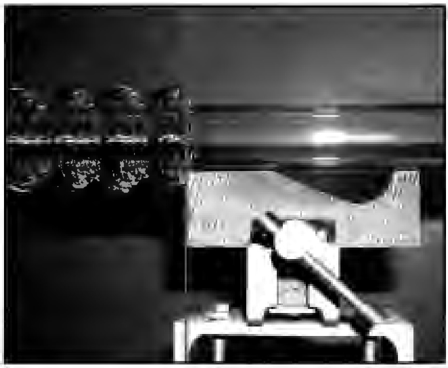
The five bulbs have been made; they are all exactly the same length and the same diameter. I will usually prepare seven sets like this before going to step 2.

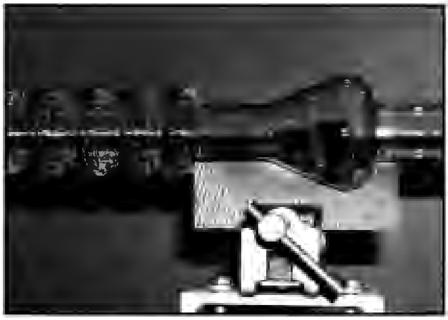
Blowing the main body
Step 2
Making the graphite mold that will shape the main body: note that the graphite tool begins at the bottom of the lowest bulb.

The tool is put in place against the bottom of last bulb.



The main body is shaped, and the opening blown out. Again I will do all the pieces before going to step 3.

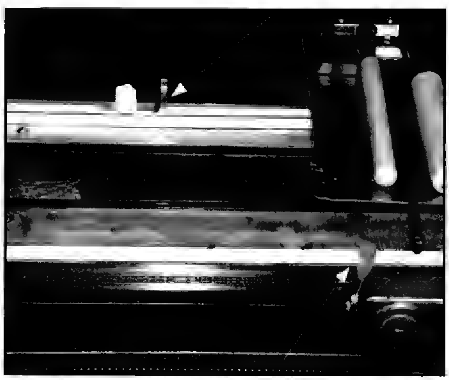




Preparing the top
Step 3
Resting the bottom bulb against the top jaws will give me a repeatable position; a glass marking pen or a diamond tip will indicate where to pull off the excess glass.

The Chemglass Chemthread will then be added. The correct position of the thread is assured by the line on the tool and in respect to a stop on the lathe bed.

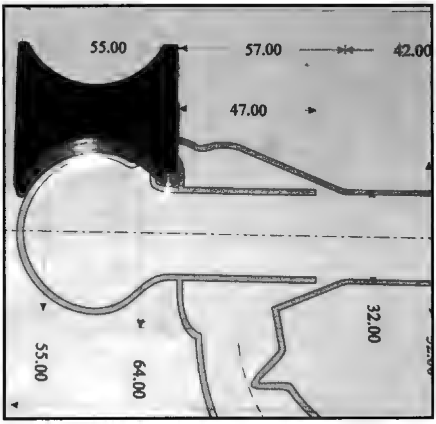
Tail chuck stop
An adjustable stop is installed on the lathe bed with a ruler. It makes repeating precise moves fast and easy.



Making the oil reservoir

Step 4

I shaped the graphite mold that will make the oil reservoir to match the drawing. The graphite is shaped to adjust to the bottom of the boiler, again as a reference.



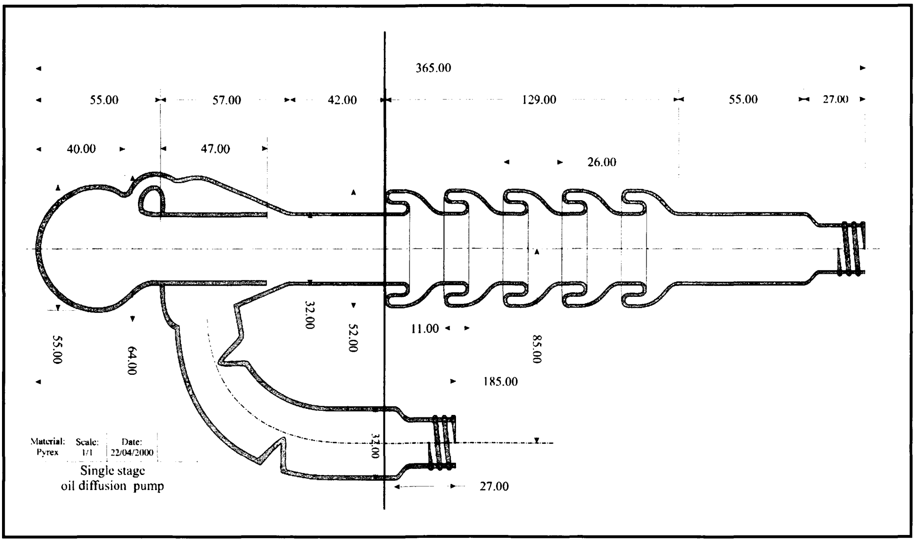
The injector and the reservoir have been blown. The oil return will be added at the bench.

Adding the side arm

A special support was built specifically for this job: it enables you to rotate the pump in the lathe and to attach the side arm exactly and easily every time.

The side arm has been easily welded in place, then bent. The second Chemthread is added, again using the same reference point.

The oil diffusion pumps have been easily made. They are exactly the same size and were done without taking any measurements, only using the appropriately shaped tools.



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