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Papers

How to Construct a Schlenk Vacuum Manifold

by
Joseph S. Gregar*

ABSTRACT

This paper will instruct experienced scientific glassblowers how to easily construct, step by step, a 4-port Schlenk vacuum manifold. It will also give some background and history about Wilhelm Johann Schlenk, its inventor and namesake. There are also many glassblowing techniques shared with the readers.

WHAT IS A SCHLENK MANIFOLD?

The Schlenk line, also known as a vacuum/gas manifold, is a commonly used chemistry apparatus. It consists of dual manifold lines connected together with several ports (valves or stopcocks). One manifold is connected to a source of purified inert gas and the second to a vacuum pump. The inert gas is vented through an oil bubbler and solvent vapors and gaseous reaction products are routed through a cold trap to protect the vacuum pump using liquid nitrogen or dry ice and acetone. Special stopcocks or Teflon® taps allow vacuum or inert gas to be selected without the need for placing the sample on a separate line. Schlenk lines are useful for safely and successfully manipulating air-sensitive compounds, that is, those that could experience rapid oxidation and ignite or explode or at least be decomposed and rendered useless.

WHO INVENTED THE SCHLENK MANIFOLD?

Wilhelm Johann Schlenk (March 22, 1879 – April 29, 1943) was a chemist who was born in Munich, Germany where he also studied chemistry. Schlenk was employed at the University of Berlin in 1919. He was an organic chemist who discovered organolithium compounds around 1917 and he won the Lieben Prize in 1917. He also investigated free radicals and carbanions and discovered that organomagnesium halides are capable of participating in a complex chemical equilibrium, now known as the Schlenk equilibrium. Today, Schlenk is remembered mostly for developing techniques to handle air-sensitive compounds and for his invention of the Schlenk flask and the Schlenk line.

WHY USE A SCHLENK LINE?

Schlenk lines are useful for safely and successfully manipulating air-sensitive compounds. The vacuum is also often used to remove the last traces of solvent from a sample. Vacuum and gas manifolds often have many ports and lines, and with care, it is possible for several reactions or operations to be run simultaneously. When the reagents are highly susceptible to oxidation, traces of oxygen may pose a problem. Then for the removal of oxygen below the ppm level, the inert gas needs to be purified by passing it through a de-oxygenation catalyst. This is usually a column of copper (I) or manganese (II) oxide which reacts with oxygen traces present in the inert gas.

MANY DIFFERENT DESIGNS

The Schlenk manifold incorporates a double manifold system: one manifold for vacuum and a second manifold for an inert gas. These will be interconnected together by high vacuum valves. Various catalogs offer many different designs, styles and sizes of

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Schlenk vacuum manifolds (Photos 1 & 2).

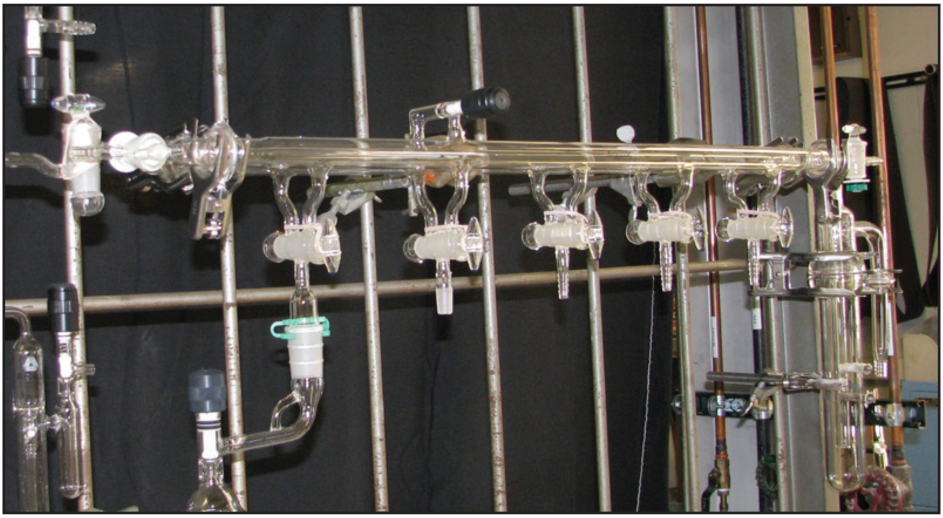

Photo 1

Photo 2

THE GREGAR DESIGN

The Gregar design is very compact and uniform, with all valves in alignment. The manifolds are connected together with Teflon® high vacuum valves with relatively short open lengths in the vacuum spaces (Photo 3).

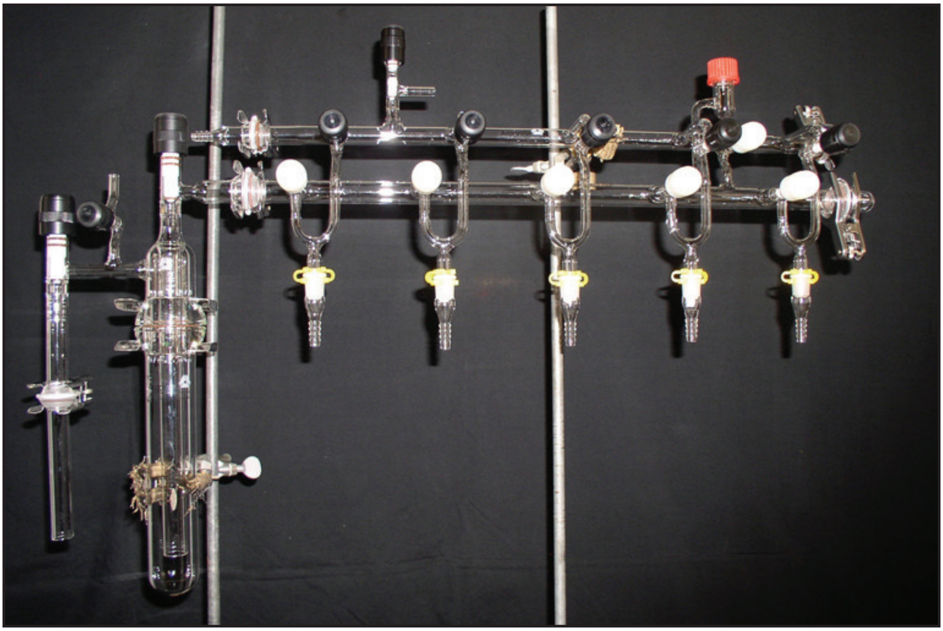

Photo 3

PARTS PREPARATIONS

Prepare all parts before the assembly sequence.

1. Make a full size drawing of both manifolds showing all attachments.
2. All tubing and parts are washed in a detergent and rinsed in deionized water before use.
3. The ends of tubing are scored with a carbide steel knife and snapped apart. The knife marks are then heated in a flame, peeled off, and removed before fire polishing the ends and producing a flare on the end before sealing onto the manifold lines.
4. Lay manifold sections onto the drawing and mark all of the attachment points. I first use a small mark from a permanent marker and then use a small diamond ball scribe in a Dremel® tool for a more permanent marking. This small mark will be removed when making the hole for the tube splice.

START WITH THE INERT GAS MANIFOLD

I prefer O-ring ball joints and clear polished sockets for the ends of my manifolds. Using the ball and socket joints allows the ability to make tight seals on the ends but also allows for a long-handled brush to be inserted for cleaning purposes on a periodic schedule.

Make your manifolds by splicing the polished sockets onto 19 mm medium wall tubing. This is the tubing size commonly used on the 28/25 ball and socket joints. Match the manifold's overall length to the drawing (Photo 4).

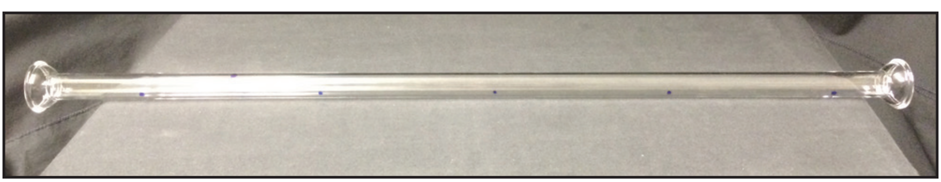

Photo 4

Use a typical 4 mm bore, 180 degree (straight) Teflon® high vacuum valve. Orientation of the valve seat is very important. The valve seat should be installed so that it faces toward the vacuum source; therefore the upper arm on the valve must be changed. Carefully heat with a hand torch and remove the upper arm and splice a new section of 9 mm, 1.5 mm lab special wall tubing. This new tube is then heated and bent in the configuration in the next photo. The two arms are then cut to the length that is on the drawing (Photos 5 & 6).

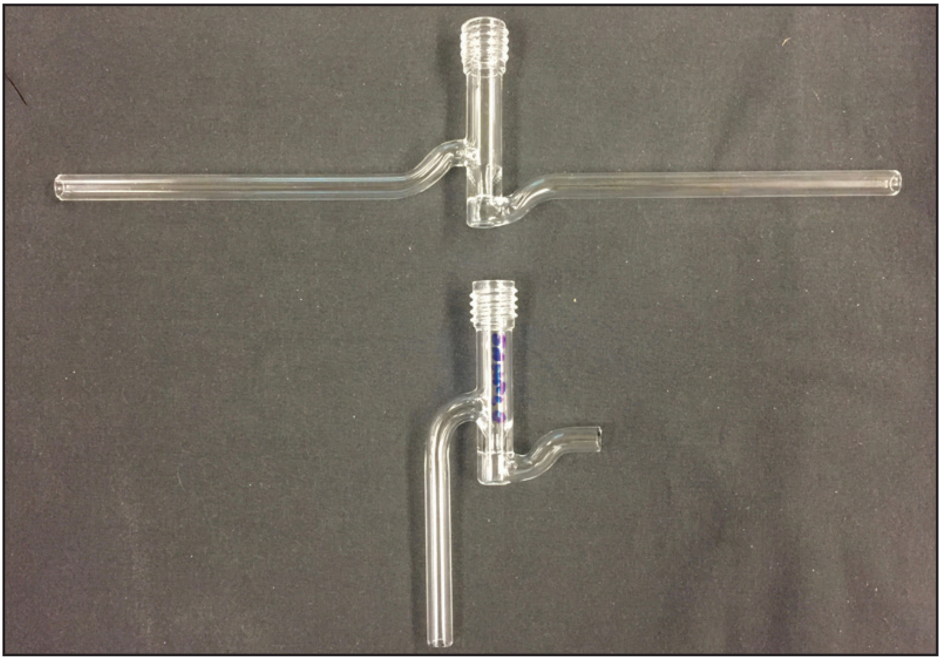

Photo 5

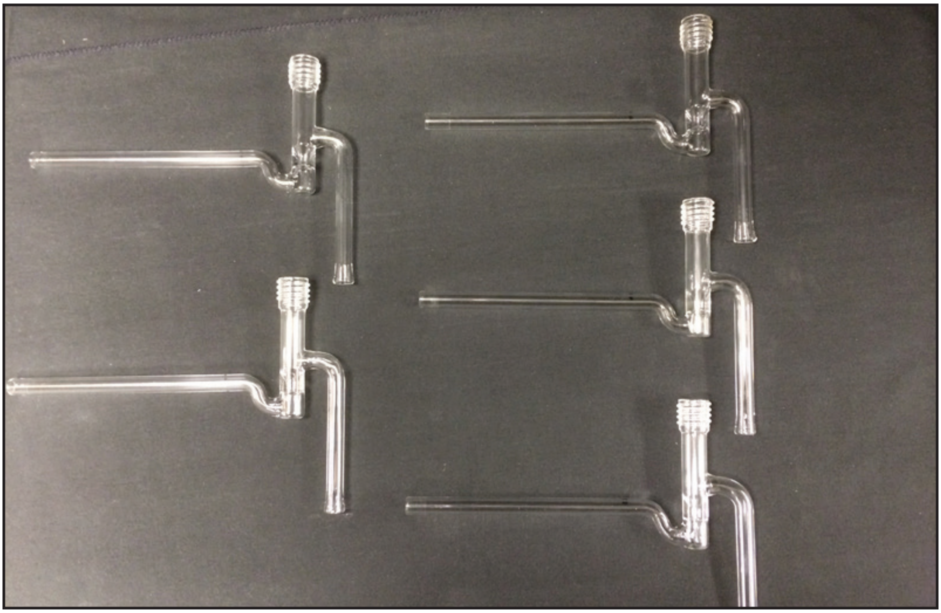

Photo 6

ATTACHING THE VALVES

Photo 7

Support the manifold in a ring stand and seal the valve's modified upper arms onto the front face of the manifold at the scribed marks (these marks were added to the manifold from the drawing). Remember to add the safety pressure relief valve to the top of the manifold, and anneal in oven (Photos 7, 8 & 9).

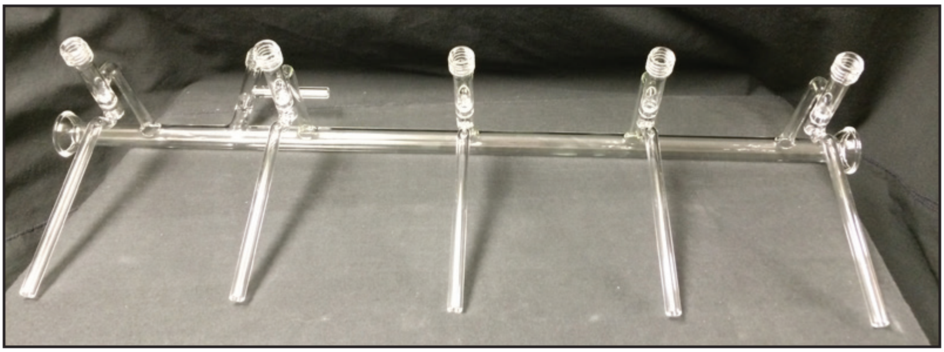

Photo 8

A TECHNIQUE FOR SPLICING THE VALVES ONTO THE MANIFOLD SECTIONS

Photo 9

1. Pre-heat the manifold tube in the area of the right angle splice with a bushy flame on the hand torch.

2. With a fairly sharp gas oxygen flame, heat an oval area on top of the manifold where you want the hole for the valve to be. When that area is warm, gently blow a bubble but do not let it pop open. The size of the heating area should be slightly smaller than the

diameter of the flare that was prepared on the end of the valve tube to be attached. This way, as you blow the bubble, the sidewalls of the bubble will match the flared tube. If not successful, reheat the entire bubble and blow it again slightly larger (Photo 10).

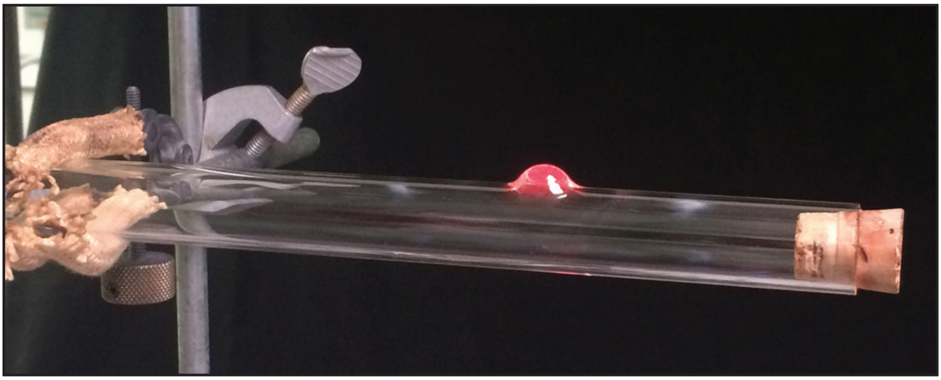

Photo 10

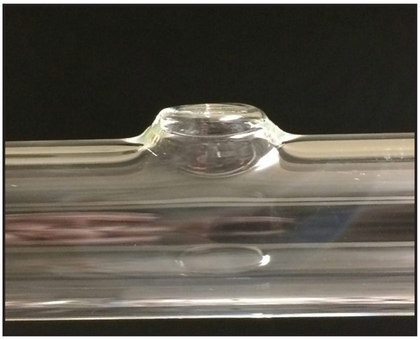

Photo 11

3. Reduce both fuels on the hand torch to have a smaller and cooler flame. Carefully aim the flame tip over the top of the bubble and just heat the top-center surface of the bubble carefully until it flattens (Photo 11).

Stop heating by removing the torch from the area and let the flattened bubble cool. Make a small sharp flame and reheat the top surface of the bubble; when it is soft, pull that flat section out using a small glass rod or tweezers (Photos 12 & 13).

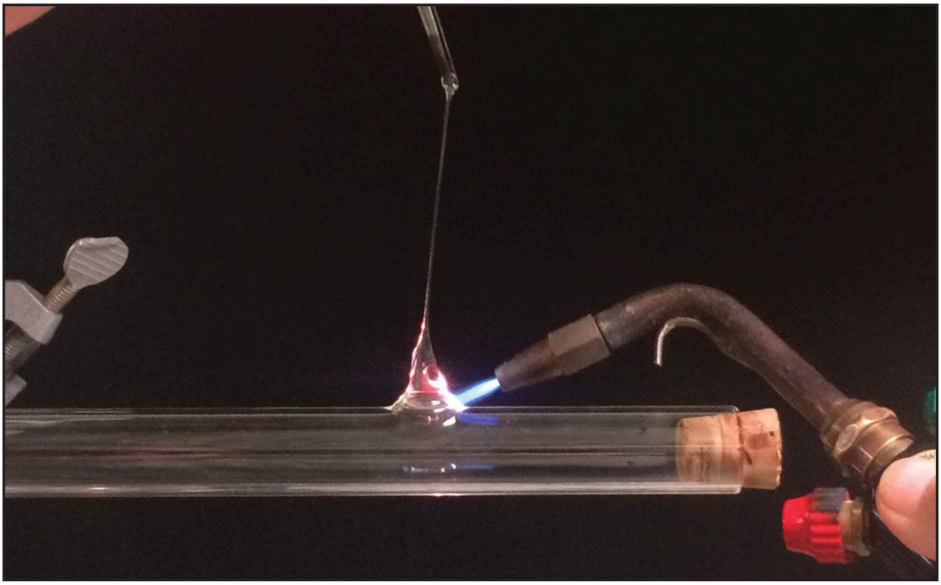

Photo 12

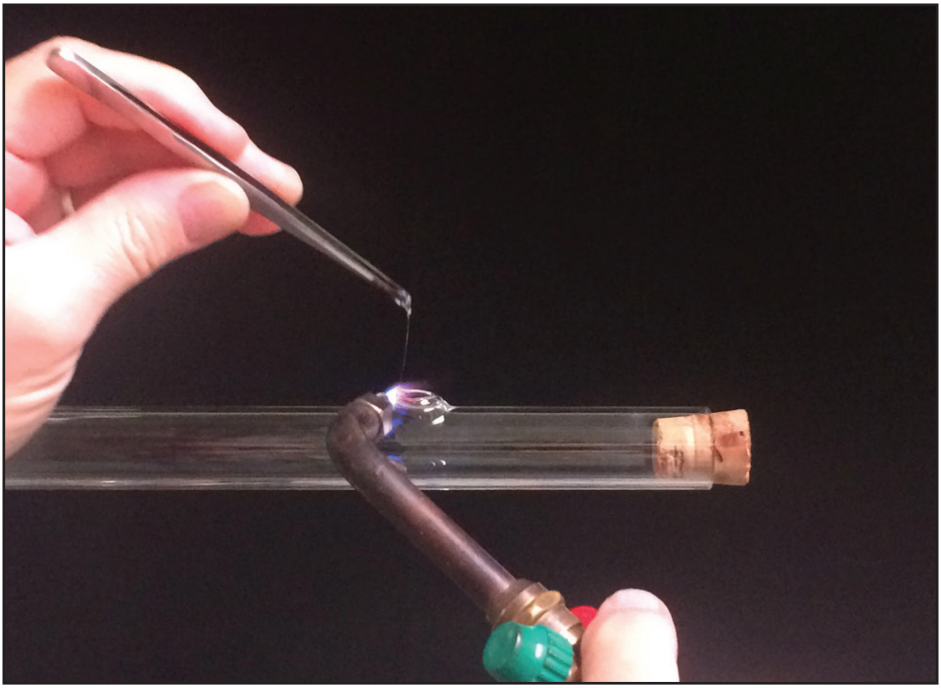

Photo 13

4. If necessary, heat and make the hole round using a thin graphite rod (Photo 14).

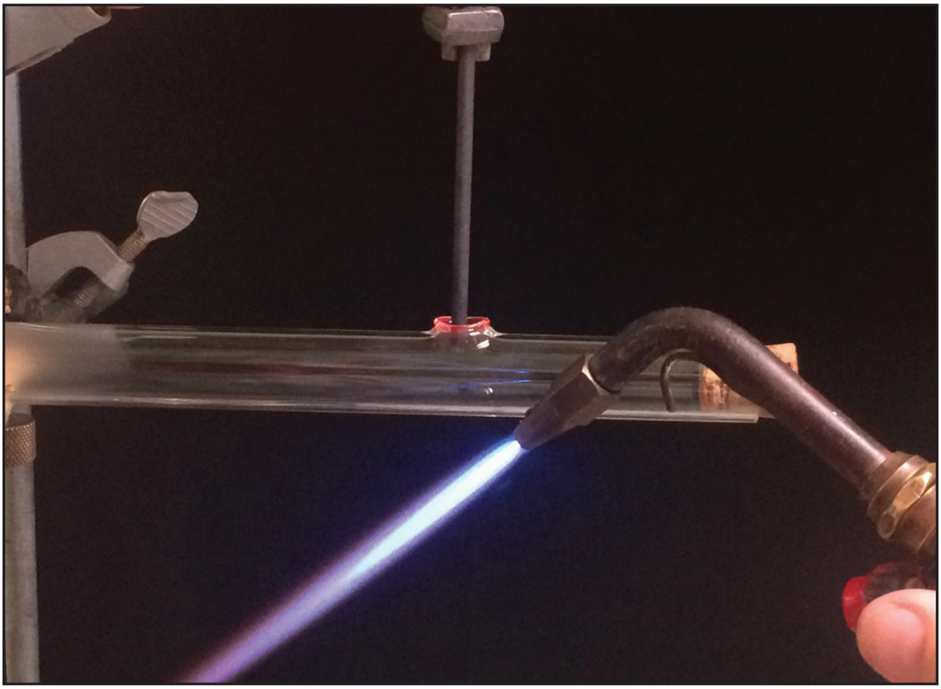

Photo 14

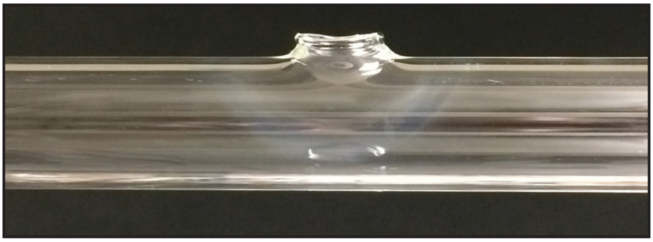

Photo 15

5. Notice the nice sleeve of glass that sticks up from the tube for you to splice onto (Photo 15).

6. Completed 90 degree splice (Photo 16).

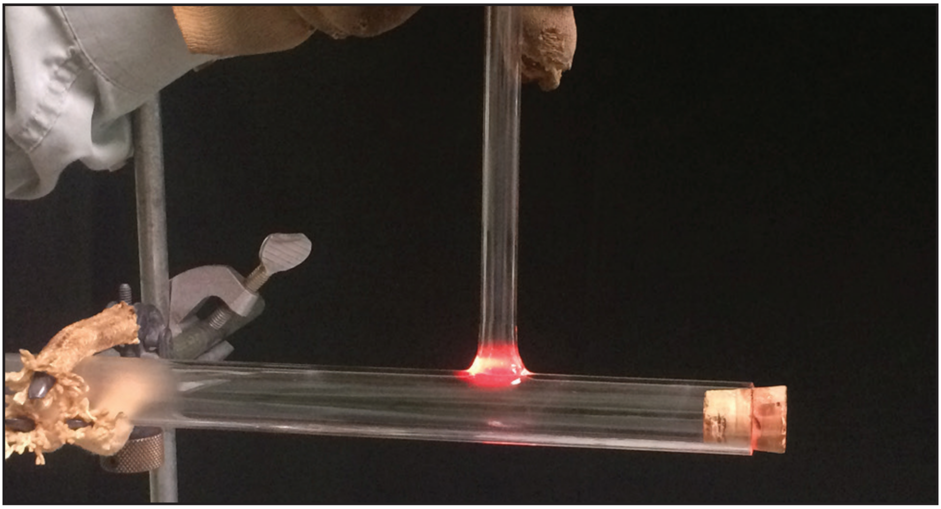

Photo 16

CONSTRUCT THE VACUUM MANIFOLD

1. Prepare the 90-degree valves for the vacuum manifold.

Again the valve seat must face toward the vacuum source (Photo 17).

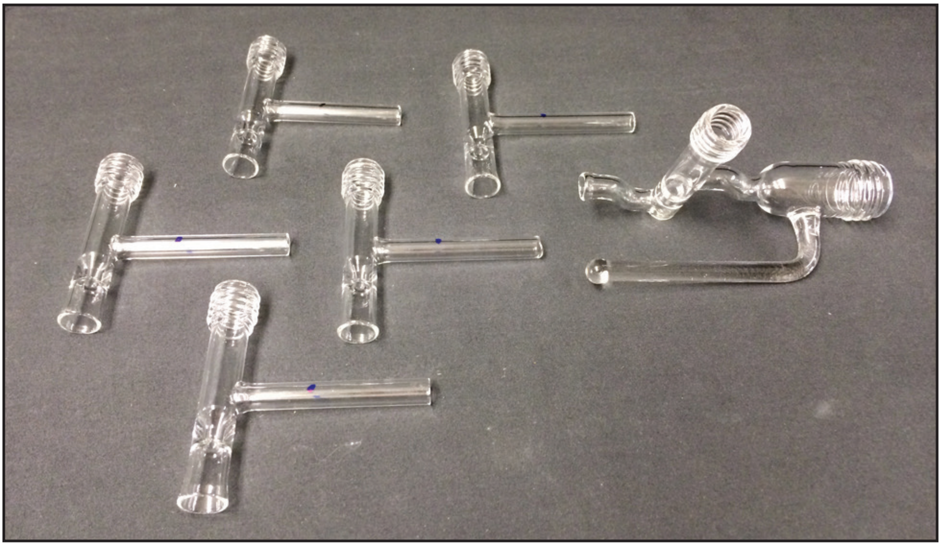

Photo 17

Photo 18

2. Add the threaded vacuum adapter to the top of a 180 degree valve and add a 9 mm rod as a support brace, and anneal in oven (Photo 18).

3. See the completed vacuum manifold (Photos 19 & 20).

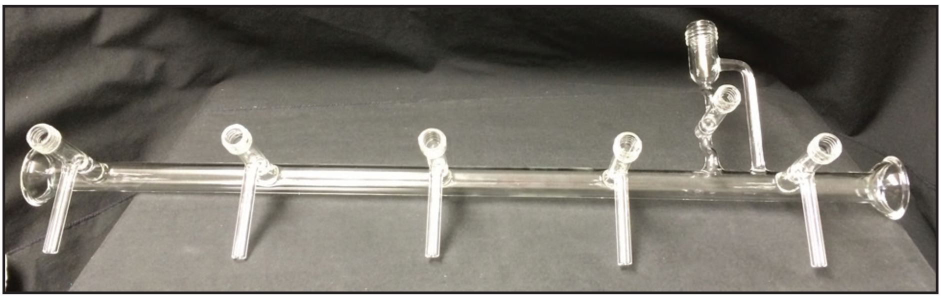

Photo 19

Photo 20

COMPLETED MANIFOLD SECTIONS

Here is a photo of the individual assembled gas and vacuum manifolds (Photo 21).

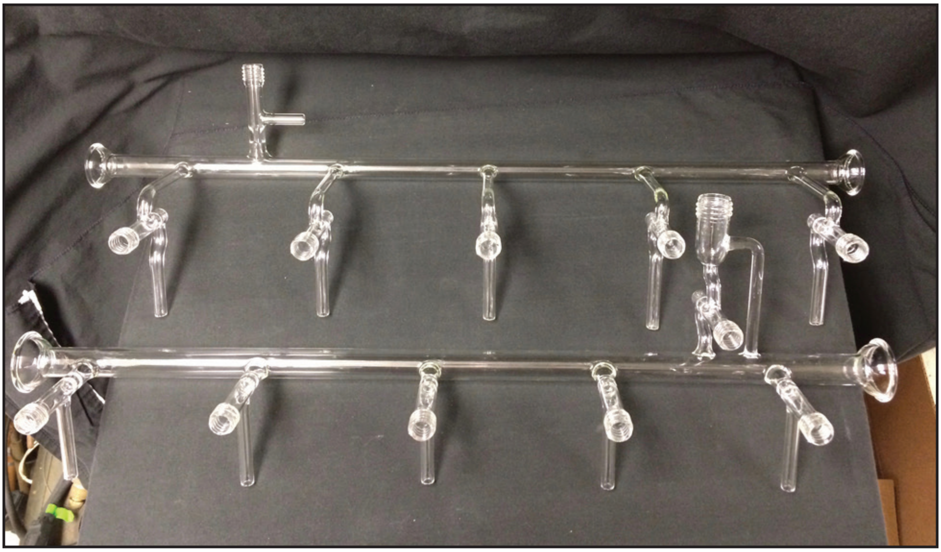

Photo 21

PREPARE FOR ASSEMBLY

Mount the two manifolds on a lattice style vacuum rack or two ring stands. Be very particular in the alignment of the two manifolds before starting the sealing process to join them. Make sure that they are straight and parallel to each other and that the spacing between them gives you the proper alignment for the valve placement and height (Photos 22, 23 & 24).

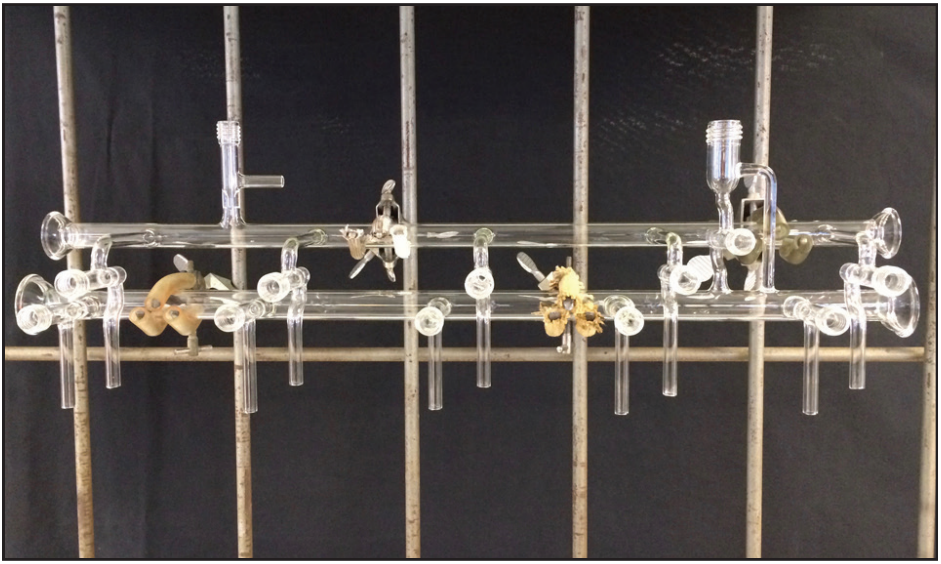

Photo 22

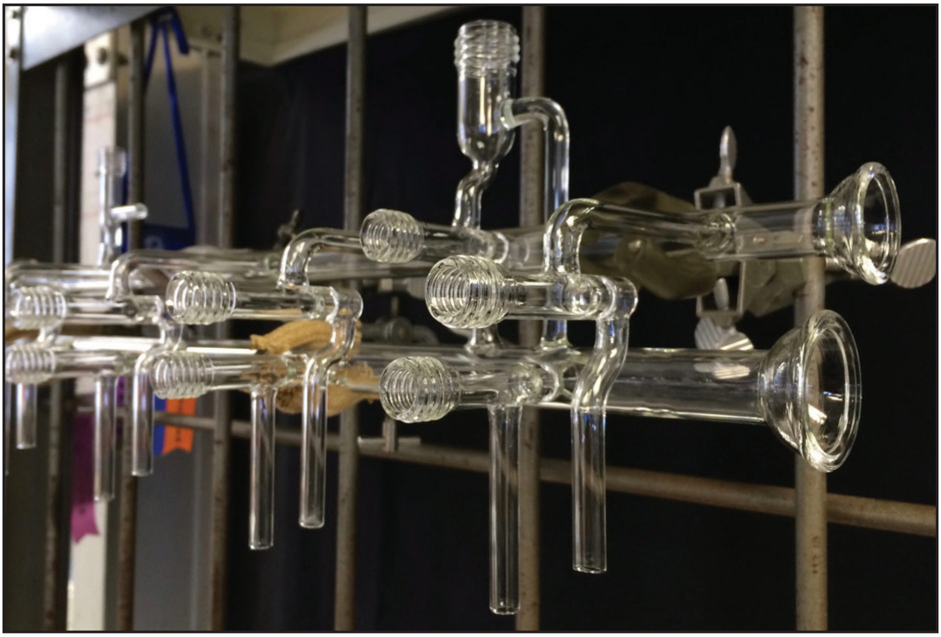

Photo 23

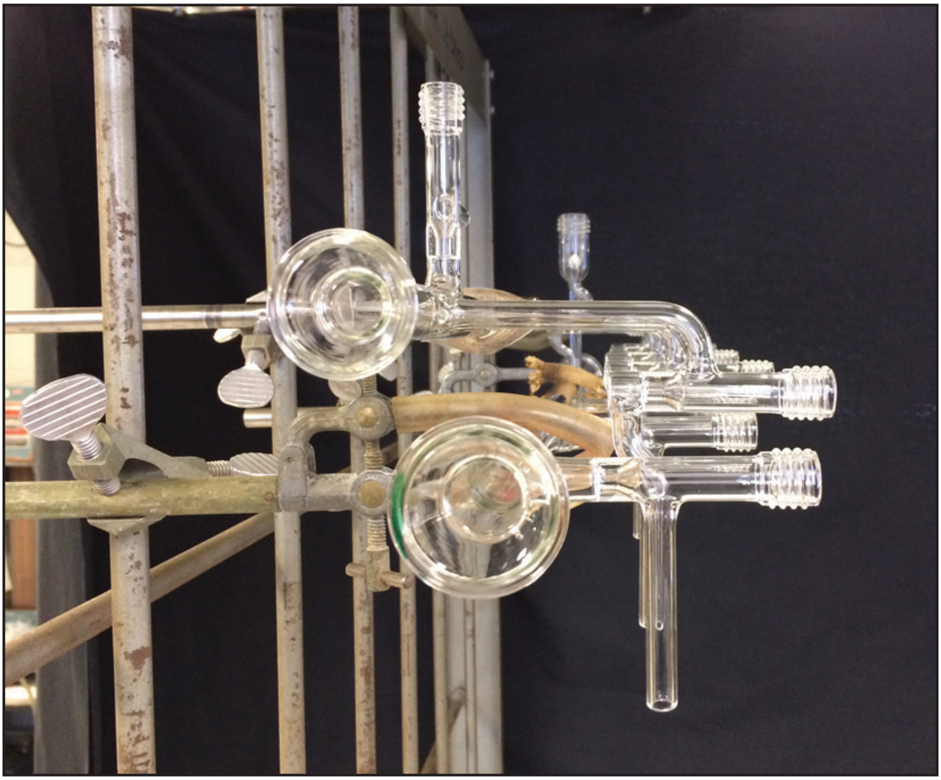

Photo 24

The two manifolds are joined together by using “U” tubes as connectors.

Bend 9 mm medium wall tubing into “U” tubes for the connecting tubes. The distance or width between the arms must match the spacing between the gas and vacuum valve arms. You can choose to use the ‘U’ tube as is or add the desired joint or tubing on the bottom to match the type of connector desired. Keep their height (length) at a reasonable minimum so as to not have too much open volume (Photos 25 & 26).

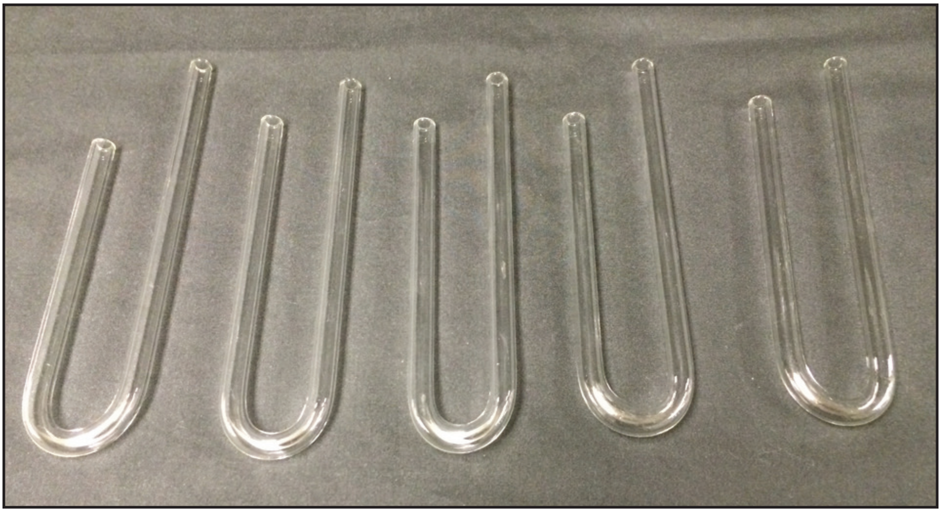

Photo 25

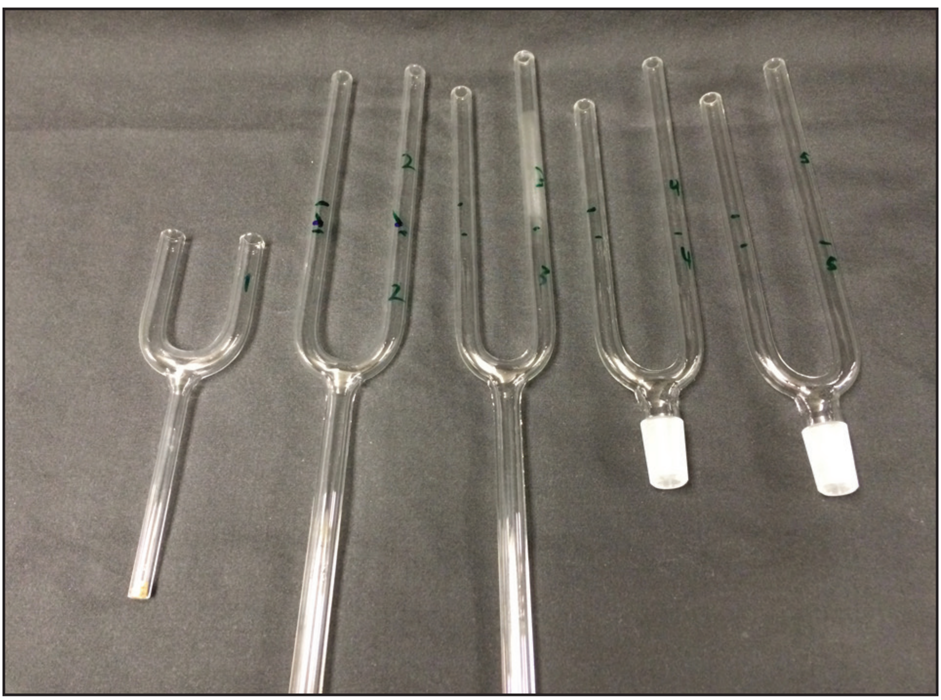

Photo 26

Measure the "U" tubes and cut to fit (Photo 27).

PERFORM A FAKE SEALING OPERATION AS A PRACTICE EXERCISE

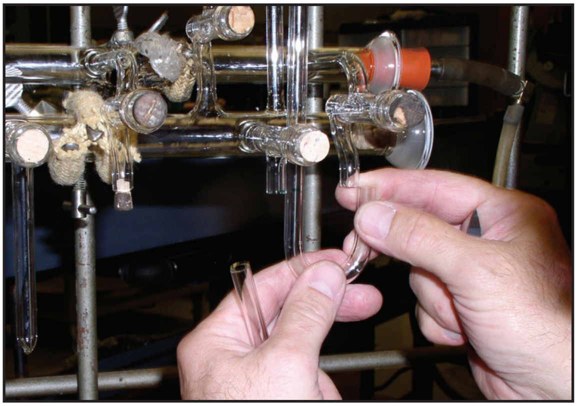

Photo 27

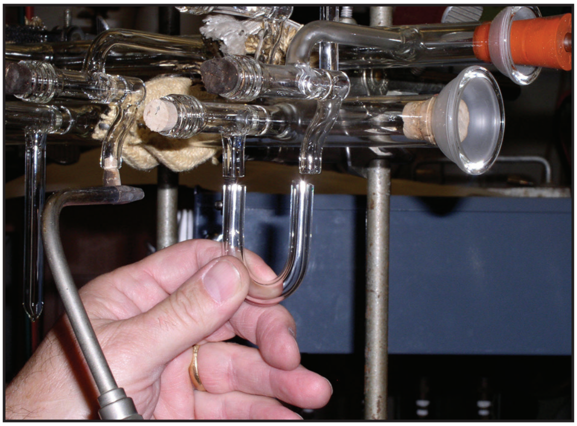

Photo 28

Hold the "U" tube in place and manipulate your hand torch without a flame for practice. By doing this practice "test seal" without the flame, you can determine where you may have tight spots to fit your torch into. Be sure you are able to heat all the way around the tube for a complete seal. You do not want to leave any area partially sealed which could leave you with a pinhole vacuum leak. Also you can see where you might have accidentally hit other parts of the manifold with the flame. These could produce "fire checks," which are small stress cracks. Upon knowing and understanding where the trouble spots are, you can pre-warm them and pay extra attention to them during the flame annealing process (Photo 28).

Seal the "U" tubes onto both the inert gas and vacuum arms of the respective valves (Photo 29).

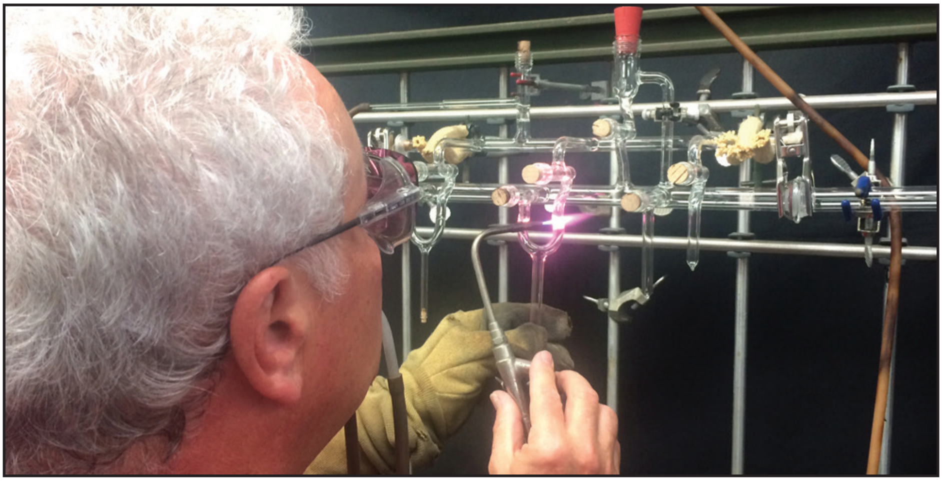

Photo 29

Here are photos of some completed Schlenk vacuum manifolds (Photos 30, 31 & 32).

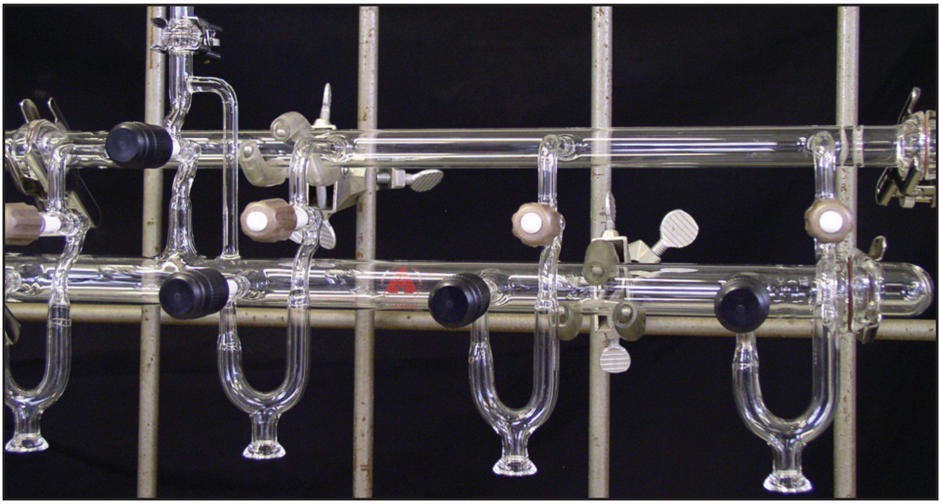

Photo 30

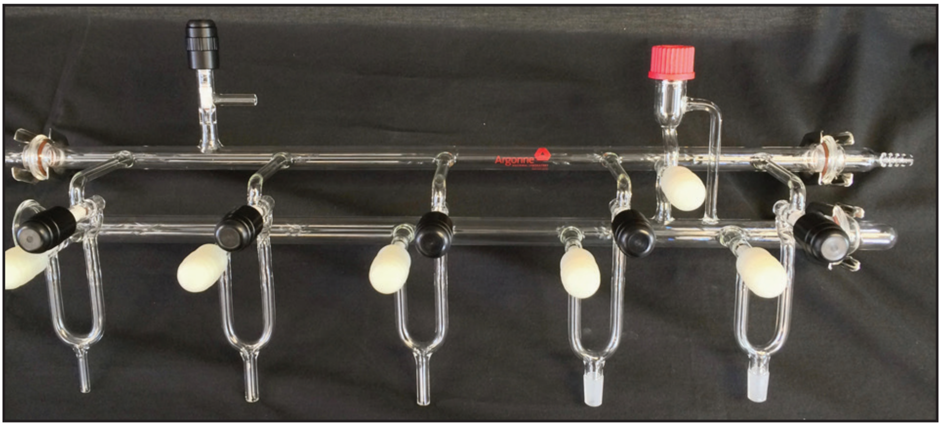

Photo 31

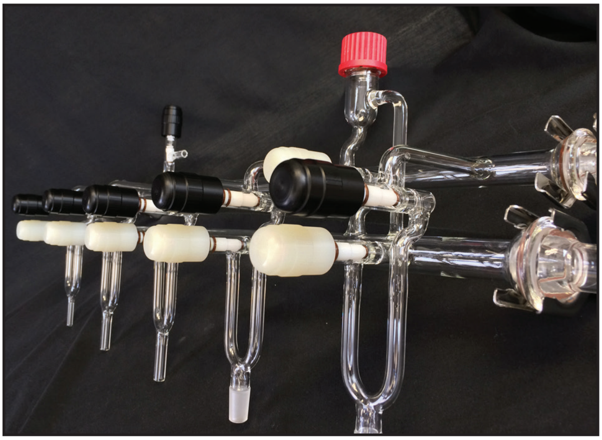

Photo 32

ANCILLARY GLASSWARE

There are a couple of ancillary items you will want to include in a good Schlenk vacuum manifold system. One is a liquid nitrogen cold trap to condense gases so they do not reach your vacuum pump. The second is a mineral oil bubbler to visually gauge the flow of the inert gas through the system. Note that this bubbler has a ceramic check valve to keep any mineral oil from flowing up into your system should you lose your gas flow (Photo 33).

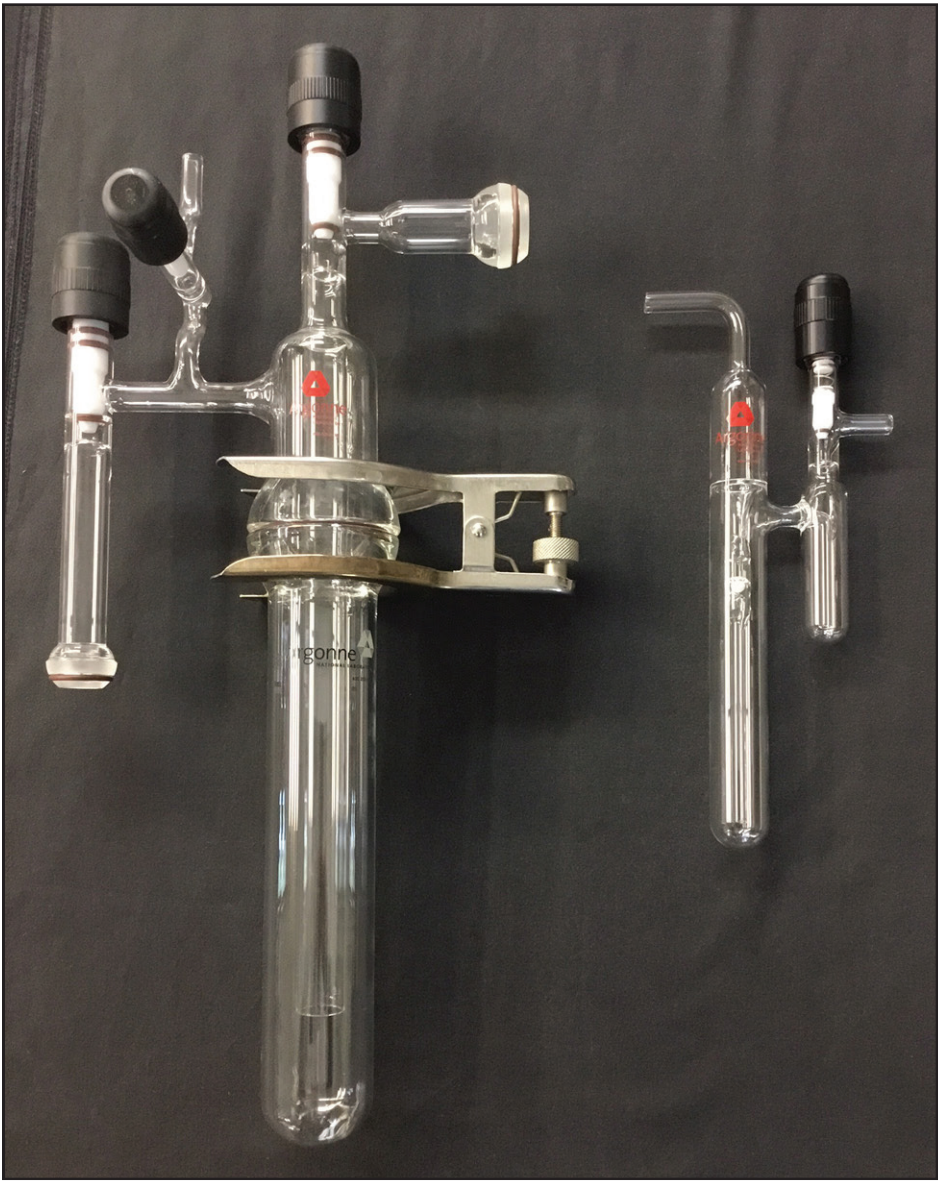

Photo 33

CONCLUSION

It was my intent to show and instruct how to easily make a complex Schlenk vacuum system. We all have had to make them and they can be somewhat troublesome with the issue of making so many attachments and fighting the strain that is put into the manifolds from making all the seal attachments. This style manifold allows you to assemble all the parts and only have to worry about sealing the small tubes that are attached to the valves or stopcocks. This will totally eliminate the introduction of strain into the manifolds.

I do hope that you will try these techniques that I have shared with you and make your next Schlenk manifold this way. You will find that it removes a lot of stress and that it is a fun exercise.

ACKNOWLEDGEMENTS

I would like to thank Kevin Moeller for the photography work associated with this paper and James Hodgson for recommending the addition of the check valve in the bubbler to avoid the possibility of oil backing up into the system.

Discharge Tube Construction, 1969

by
Tracy Drier*

ABSTRACT

Video of Joe Wheeler with his assistant Mike Wheeler building an 18” diameter discharge tube for plasma research by Professor Claude Woods.

In 1969 at the University of Wisconsin – Madison, a glass plasma discharge tube was built by the chemistry department glassblower, Joe Wheeler, and his assistant, Mike Wheeler. This construction process was presented at the 16th ASGS Symposium (1971) which was held in Milwaukee, Wisconsin. It can be found on pages 97-106 in the 1971 *Proceedings*.

At the time, a home movie film was taken of this glassblowing process as well as the installation of the chamber in the laboratory. This film was recently uncovered by Professor Claude Woods. It was converted into a digital format and is presented here.

< .. Digital Quick Time movie embedded ..>

If digital QT movie cannot be shared in this format, then contact Tracy Drier directly.

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Flow-focusing Nozzles for Lymphocyte Processing

by

Adam Kennedy and Alex Doty*

ABSTRACT

This paper outlines the fabrication of a robust borosilicate glass nozzle with a 140 micron opening.

We were approached by researchers in the Biomedical Engineering Department with a request to design glass nozzles for a flow-focusing apparatus built to streamline the study of immune responses and antibodies. Immune responses generate tremendous complexity down to the single-cell level. Prior to this research, methods to understand the genetic complexity of those responses have been limited to ~10,000 cells. The newly developed process increases sample size one hundred fold using our custom glass nozzles to create tiny droplets that will each run an individual reaction.[1] The nozzle's pinhole opening allows for precise control over the flow of a mixture containing all the components necessary for genetically sequencing individual cells, which offers researchers the opportunity to process millions of cells at a time with minimal labor. As such, this technology dramatically advances the study of immune responses and provides access to countless useful antibodies.

The initial process for glass nozzle fabrication was intended to produce a robust nozzle with an aperture measuring between 120-160 microns. After some initial efforts, the researchers narrowed their preference to 140 microns which we applied to our production methods within a 5 micron margin. Rather than pulling a tube to a thin capillary which would present challenges in both precision and overall durability, we elected to create a pinhole which could then be gradually opened to exactly the desired diameter. The final process uses a 3/8" heavy wall borosilicate tube which is heated to collapse and ground using a diamond belt sander to create a precise capillary opening. We encountered many challenges in various stages of the project, but after much trial and error, we have honed a process that allows us to produce batches of fifty at a time with consistent results. The following process details the steps necessary to create a finished product that meets both durability and precision requirements.

The first step is to cut 6" sections of 3/8" heavy wall borosilicate tubing on the saw, ensuring that each piece has at least one cleanly cut end. Uneven ends will not constrict uniformly, and will therefore not produce a proper opening once ground. Cut tubes are rinsed and dried, then moved to the lathe for forming.

The lathe speed is set a bit faster than for normal sealing and small tube work as the increased speed more easily ensures even heating and encourages the tube to close uniformly on center. The ideal flame is soft, somewhere between a bushy annealing flame and neutral working flame, allowing for control and even heat (Photo 1). From the edge of the tube, approximately one centimeter of glass is heated. As soon as it starts to collapse, the flame is immediately moved to just the tip of the glass until the end fully closes

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[1] Jonathan R. McDaniel, Brandon J. Dekosky, Hidetaka Tanno, Andrew D. Ellington, and George Georgiou, "Ultra-high-throughput Sequencing of the Immune Receptor Repertoire from Millions of Lymphocytes," *Nature Protocols* 11.3 (2016): 429-442.

in on itself. This heating method allows for a more rounded nozzle that will produce a correctly shaped opening while maintaining material strength. Once the end closes and the glass loses color, it undergoes a quick flame anneal and is set in the oven for a full annealing cycle. The collapse and flame anneal steps must not be rushed, as being too hasty at this stage can warp the nozzle and ruin the product.

Photo 1. *Relatively soft flame for even heat while maintaining precision.*

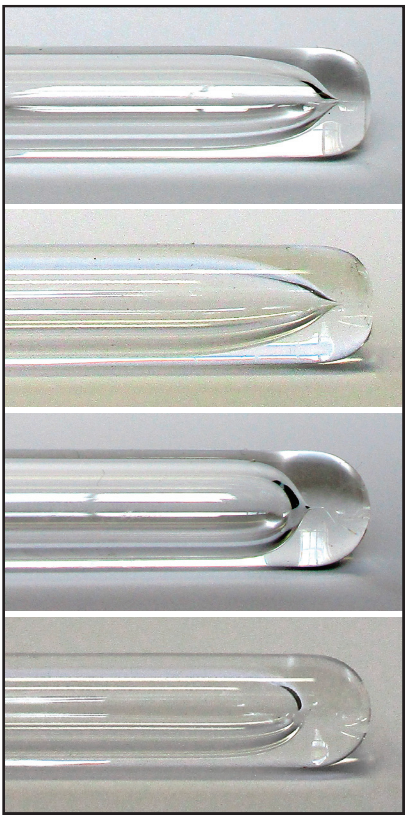

Photo 2. *(From top to bottom) Nozzle with correct inner shape, an inner shape that is too tapered, an inner shape that is too square, and an inner shape with no capillary.*

Nozzles are subjected to the first round of quality control following the oven anneal. Using a set of straight rollers, each tube is rolled to visually inspect for radial symmetry. Any sign of wobble on the closed end indicates a nozzle that will not spray straight and does not meet production standards. Following the first inspection, the inner shape of the nozzle is checked for proper curvature. The ideal inner shape is a smooth curve, which allows for precision as the opening is ground to the specified measurements and produces a straight, even spray when used. Liquid in the final application will not form the proper stream if the inner shape is too square, flat, or tapered (Photo 2). After the asymmetric and improperly shaped nozzles are discarded, the rest move into the grinding and measurement phase.

The rounded tubes are cut to a length of 55 millimeters to best accommodate the size of our microscope stage and to streamline the calibration phase of measurement. The first 1-2 millimeters of the closed end are then removed using the saw (Photo 3). This step eliminates as much of the straight section of the pinhole as possible thereby decreasing clog issues in finished nozzles, and is much more time efficient than moving directly into grinding. It is vital that nozzles at this stage be kept in water between steps, as the glass dust that can accumulate in them is extremely difficult to clean if it has been allowed to dry.

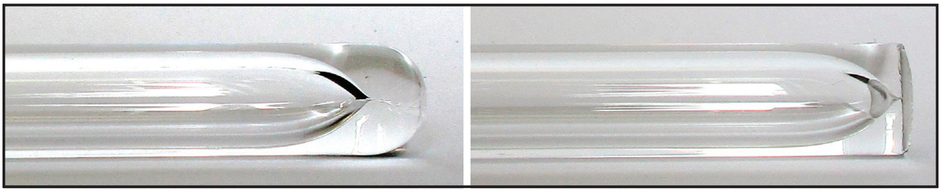

Photo 3. *(Left to right) A nozzle with a long section of straight capillary, same nozzle after the initial saw trim*

Before moving into the grinding stage, a guide fixture is attached to the belt sander to ensure the nozzles are ground at 90 degrees (Photo 4). Our fixture is comprised of an aluminum v block from McMaster Carr and hardware fabricated by an on-campus machinist, Shallaco McDonald, secured to an articulating platform from Thorlabs.

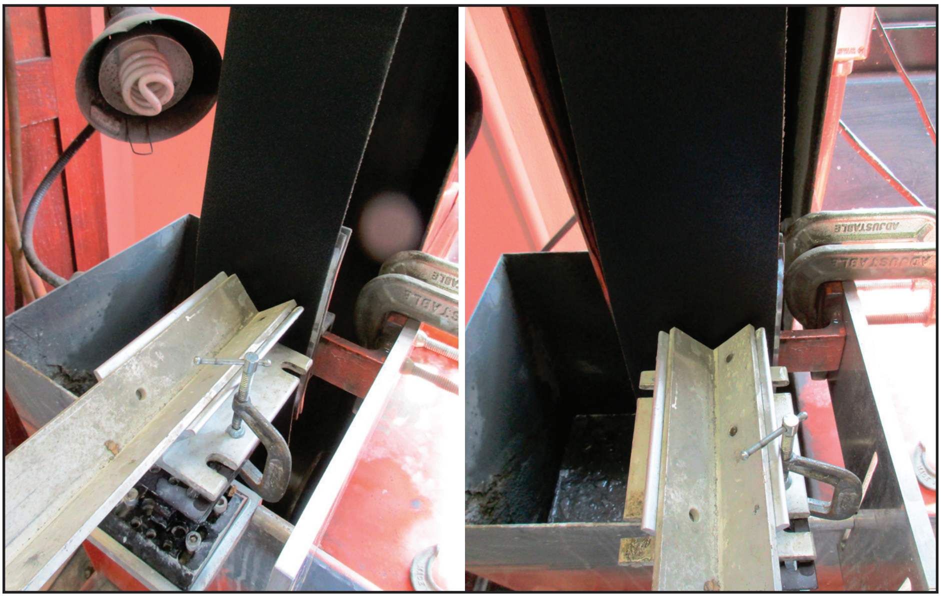

Photo 4. *Diamond belt sander and guide fixture*

After the initial headway made in trimming the first 1-2 millimeters from the rounded end, the remaining grinding can be done with a 220 grit belt. The nozzle is ground until the rest of the straight section is removed and the taper begins to open, taking care not to overgrind. The nozzle is then prepped for initial measurement by rinsing the tube of all debris, cleaning with distilled water, and rinsing with acetone. Compressed air can then be directed through the tube to evaporate the acetone and check that an opening has indeed been established. Spraying liquid through the nozzle at this stage can also confirm an opening and can indicate an over-ground piece as the liquid stream will be visibly oversized.

Following the initial grinding and testing stages, the nozzle is then placed ground face down in a custom drying apparatus on a warm hot plate to evaporate any remaining liquid (Photo 5). Our drying apparatus is made using the methods outlined in Tracy Drier's *Fusion* article detailing construction of a fused well plate, published in November 2011.[2]

[2] Tracy O Drier, "Construction of a Fused, Borosilicate, 81-Well Plate for Sample Stirring," *Fusion* 59.4 (2011): 21-25.

Photo 5. *Drying apparatus*

To create the drying apparatus, the bottom centimeter of a spare beaker was cut to create a faux petri dish. This dish was then filled with a honeycomb-like assembly of 12 mm standard wall rings also cut to 1 cm tall. The dish and rings were placed in a cold oven, brought up to 1,508 degrees Fahrenheit for 1 minute, and then crashed down to a normal borosilicate annealing cycle. This holder keeps the nozzles vertical as they dry, ensuring all moisture evaporates from the end to be measured. Nozzles must be completely dry and clean prior to measurement as any moisture or debris causes visual distortions under magnification.

Once clean and dry, nozzles move into the measurement phase. One of our initial measurement methods relied on 140 micron gauge pins; however, this technique proved less than successful. The pins were extremely difficult to thread into the nozzles' microscopic capillary openings, and many were lost in the shop due to their small size. We eventually settled on a process using an Omax stereo microscope equipped with a two megapixel USB camera and ToupView image processing software. As a time saving measure, we built a nozzle holder by drilling a 3/8" hole in a small scrap of wood and affixing it to the microscope stage with double-sided tape (Photo 6).

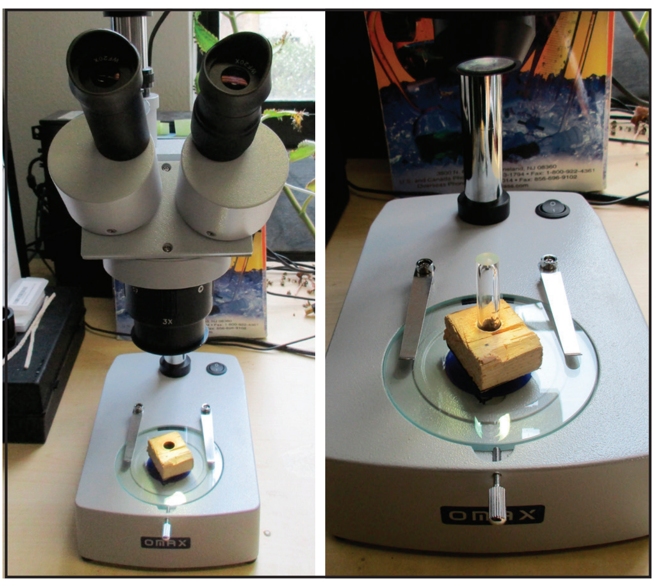

Photo 6. *Microscope arrangement with nozzle holder*

Making adjustments to a magnified sample viewed through a monitor can be cumbersome and time consuming, hence the creation of holders and stabilizers to simplify the process. A calibration slide is necessary for proper measurement of images within the ToupView software, so a holder was similarly created for our 100 scale slide (wherein 1 division is equal to 0.01 mm). A 1 cm tall ring of 12 mm tubing was attached to the slide using ultraviolet glue, which allows the slide to easily sit atop the nozzle and align roughly with the capillary opening to be measured (Photo 7). The calibration slide sits face down on the nozzle surface so that both are on the same plane as even a small difference in focus can significantly affect measurements this small and precise. This calibration is done for each slide individually to ensure accurate measurement between pieces.

Photo 7. *Calibration slide and holder*

The diameter of the capillary opening is then written with a permanent marker on the side of the nozzle, and the process of grinding, rinsing and measuring is repeated two to three times until the nozzles reach the required size of 135-145 microns. Nozzles with irregular openings undergo a short soak in a dilute solution of hydrofluoric acid which can smooth rough edges and create a more circular shape (Photo 8).

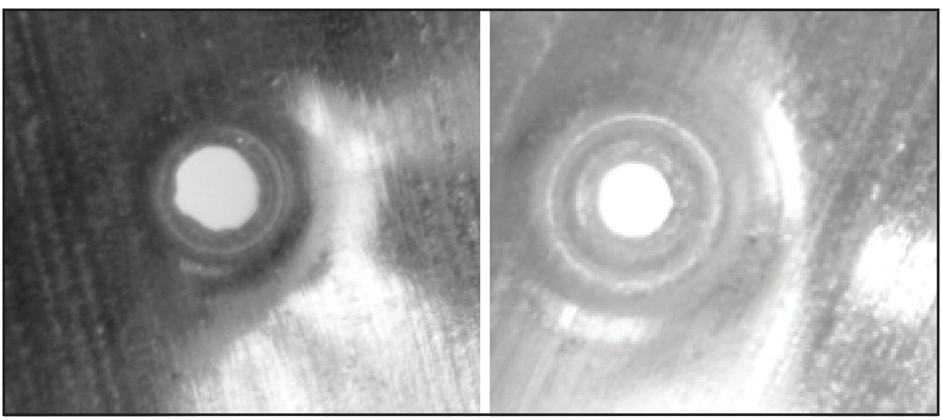

Photo 8. *Microscope image of an irregular opening (left) and the same opening after an acid soak (right)*

Once the opening reaches the target size, nozzles are trimmed to a length of 47 mm and given a slight bevel on the diamond belt to ease entry into the fitting. They undergo a final cleaning to rinse out any debris, and then the final inspection step is to shoot a stream of water out of them to visually verify that the flow is even and straight. Streams that shoot off center are further inspected to determine if they are internally crooked or clogged and are disposed of or cleaned. In extreme cases, nozzles are hooked up to an aspirator in the cleaning sink to suck the debris from either end or put in an ultrasonic bath to shake loose any remaining particles. Nozzles that have passed all inspection phases are dried and packed until shipment.

Overall, the grinding phase has roughly a 75% success rate. Some of the loss can be attributed to nozzles that are formed with an inner structure that is slightly too steep, resulting in unavoidable overgrind. This is a feature that evades detection in the initial quality control, as it is typically invisible to the naked eye. With the proper inner shape, nozzles can be ground in a predictable manner. A finer belt can also be used to achieve a more gradual grind; however the 220 grit belt strikes a good balance between speed and final shape.

This method, while not definitive or exclusive, allows precision without requiring excessive niche equipment. The only necessary tool not widely available in most glass shops is an inexpensive microscope with measuring software.

We would like to thank Mike Ronalter, Jonathan McDaniel, Dr. George Georgiou, Shallaco McDonald, and the University of Texas Chemistry Department for their support.

Fully Jacketed Addition Funnel

by

Richard J Ponton*

ABSTRACT

This paper describes the design engineering and fabrication of a fully jacketed addition funnel, with the jacket encompassing the top and bottom joints as well as the pressure equalizing arm and the stopcock.

A research group came to me with a request; they had a special type of addition funnel attached to a wiping film distillation head and were having trouble with the liquid in the funnel. As a wax based oil, the liquid added to the distillation head began solidifying at very low temperatures and was totally solid by 30°C, just slightly above room temperature. As a result, the material was not draining into the distillation head properly, and was particularly difficult in the metered Teflon® valve. After attempts at using heated tapes and blown hot air, a jacketed solution was determined to be the best.

The customers and I determined the best route was to fully encapsulate the funnel in a jacket: the body, pressure relief arm, valve and bottom joint were all to be jacketed. In this way, the surrounding temperature could be precisely controlled and a high enough temperature could be properly maintained in the entire system (Image 1).

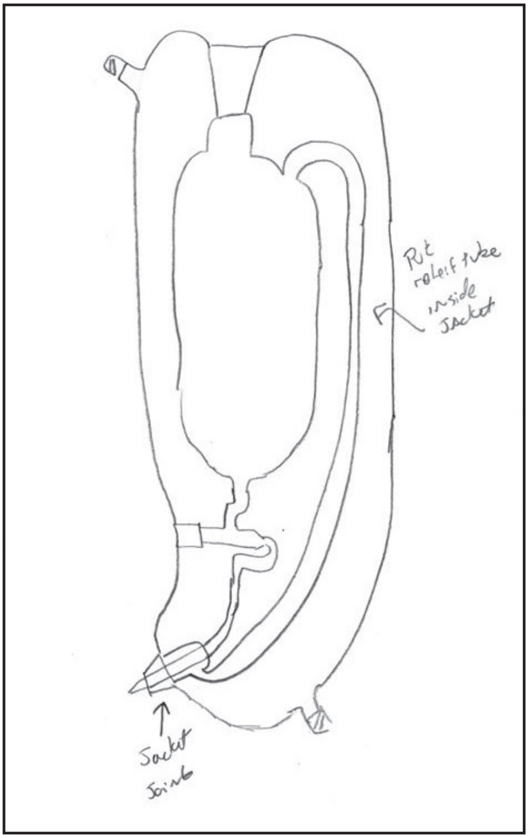

Image 1

In my glass shop, I employ a 3D computer graphics software to do all my design engineering prior to beginning any physical glass work. This allows me the ability to work with my customers, show them images and verify any problems before I begin to do the actual glassblowing. Working from our very rough sketch, I refined the design into a 3D rendered virtual addition funnel (Images 2, 3, & 4).

Using my software (Autodesk® Inventor) I then generated 2D schematic drawings for use while manufacturing the unit (Images 5, 6, & 7).

With the design phase completed and approved by my customer, it was then time to actu-

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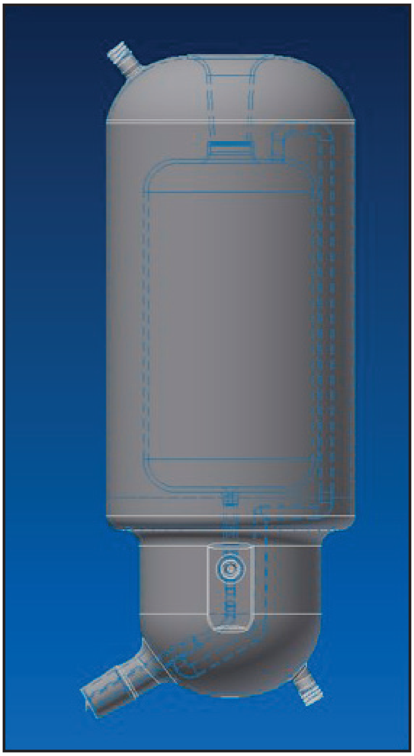


Image 2

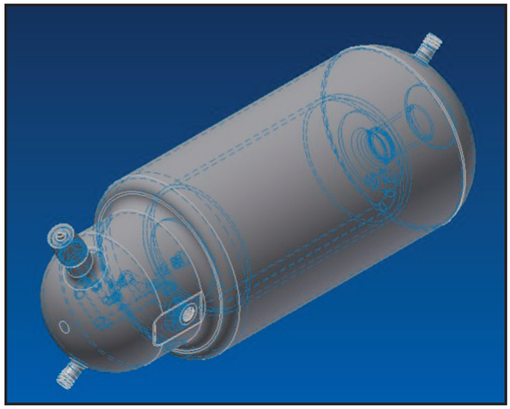


Image 3

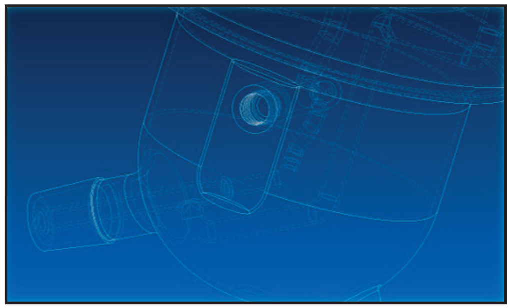


Image 4

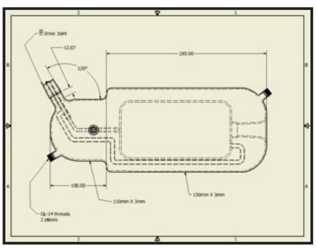


Image 5

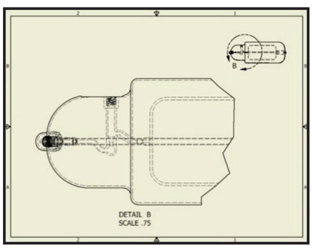


Image 6

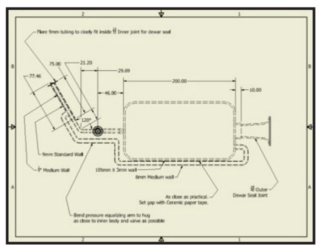


Image 7

ally build the device. A piece of 105 mm x 3 mm tubing was pulled to a near flat bottom, and a 0-4 mm Ace metering valve was sealed onto the center. By using flatter bottoms and tops, I was able to maximize the volume and make the unit as short as possible (Image 8).

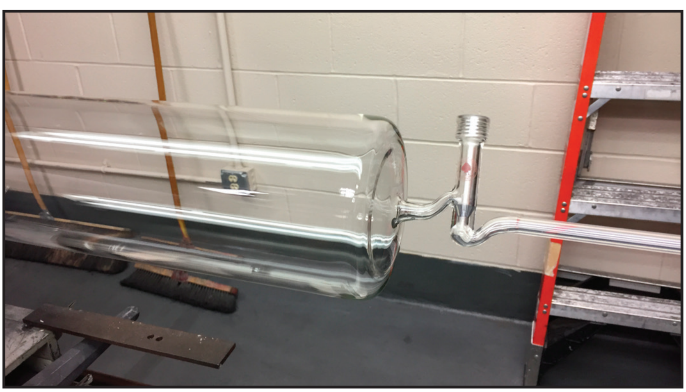


Image 8

Once cool, the unit was flipped in the lathe, a similar flat bottom was pulled for the top of the funnel, and a 24/40 dewar seal joint was attached on center (Images 9 & 10).

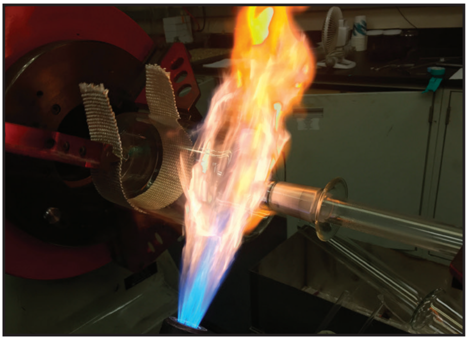


Image 9

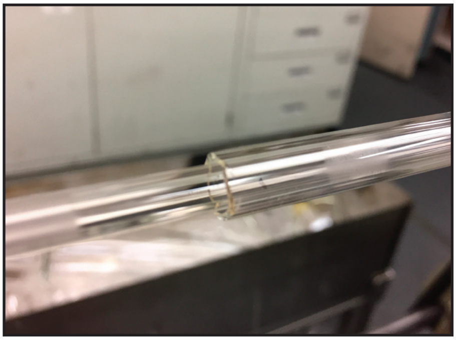


Image 10

The drain below the metering valve proved challenging not only because there was a design requirement that the inside of the inner joint be jacketed, but it needed to sit at a 120° angle, bent from center. Since the joint was required to be a 29/32, it also left me with a very small amount of space to work with, so the tubings were all very small on the drain end. The innermost tubing (the actual drain connected to the Ace valve) was made with ¼" MW tubing, and it was decided that the pressure relief tubing around that was to be 9 mm SW. All of this, along with the pressure relief arm had to fit inside of a 29/32 inner joint, so the closer everything was the better. After being sealed up, the end of the 9 mm tubing was flared to just shy of the diameter at the end of the grind on the 29/32 joint. This flare will eventually be "dewar sealed" to the bottom of the joint, allowing the jacket to extend to the very bottom of the joint (Image 11).

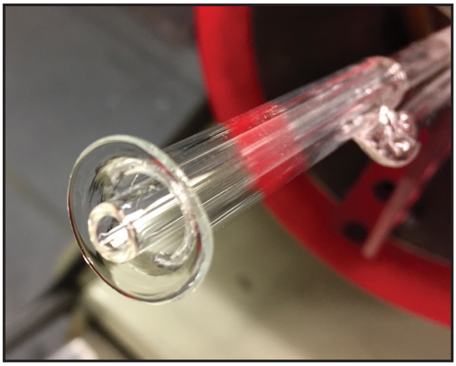


Image 11

After attaching the assembled line to the metering valve, it was bent to the required 120° angle (Image 12).

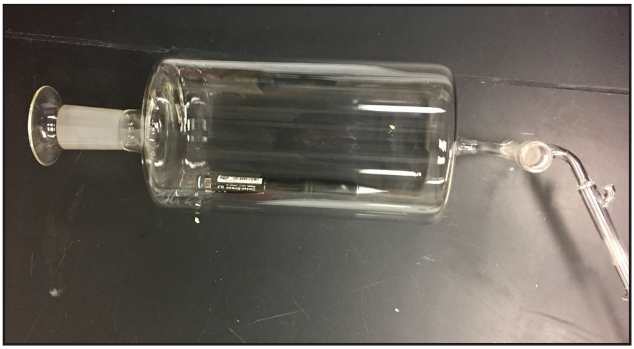


Image 12

The unit still needed its pressure equalizing arm made and attached. Traditional pressure equalizing arms have a sweeping U-bend at the top and generally sit 8-10 mm away from the funnels body before tapering gently down to the joint. In this case, however, a much tighter unit arm was required. The further away the arm sat from the body, the larger the jacket tubing would need to be, which would, once finished and filled, add unnecessary weight (Image 13).

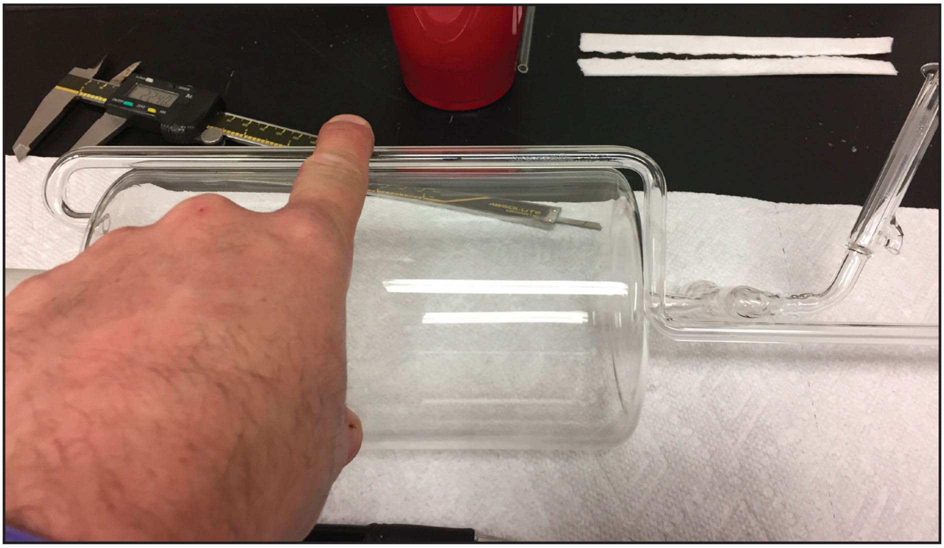


Image 13

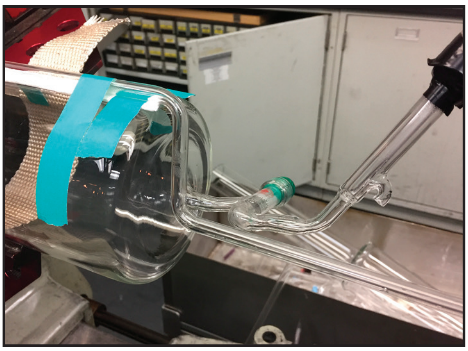


Image 14

Image 15

A strip of 1 mm thick fiber tape was laid between the arm and the body, and the arm was then taped to the unit. Then the arm was slightly bent around the bottom and tucked in as close to the valve as possible before being sealed into the drain tube (Images 14, 15 & 16).

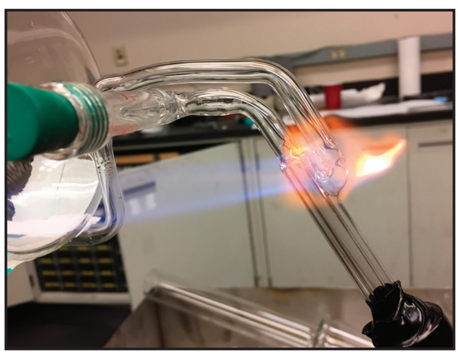


Image 16

Sealing the top of the pressure equalizing arm presented a challenge, as the angled drain tube prevented me from being able to properly hold the body in my lathe. My only recourse to hold the unit was to use a 24/40 inner joint placed in the top dewar seal joint. We have all watched the horror movie of seeing something you are working on "walk" out of the holder joint and shatter on the floor. To mitigate this, I found a scrap piece of tubing in the shop and cut out notches that fit around the drain tube and the metering valve. I was able then to chuck that into my tailstock and wind it in until it touched the bottom of the funnel. This would prevent the unit from falling off the holder joint (Images 17, 18 & 19).

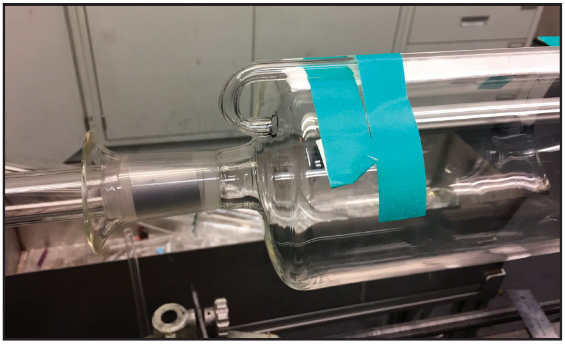


Image 17

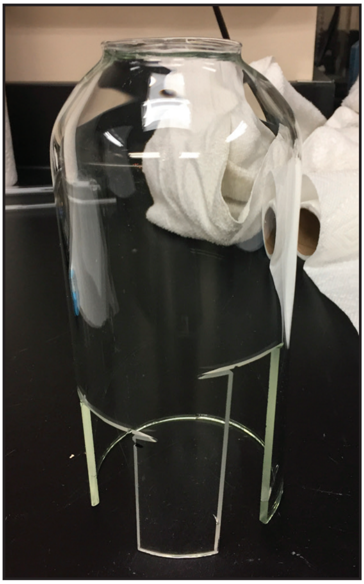


Image 18

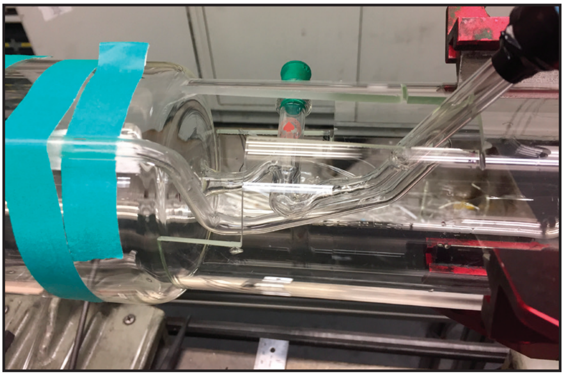


Image 19

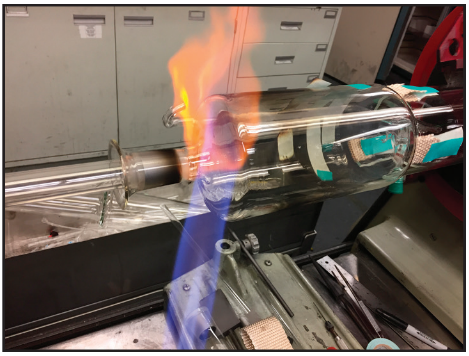


Image 20


Image 21

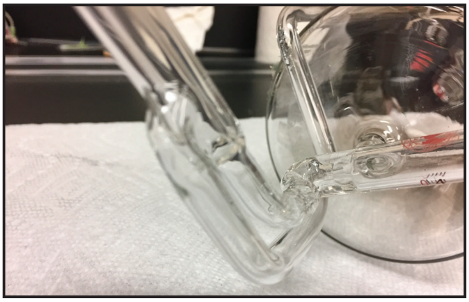


Image 22

With the item secured, the top was heated up and the final seal on the pressure relief arm could be completed (Images 20, 21 & 22).

With the actual funnel now completed, I could begin the next task: building the jacket. A short segment of 110 mm x 3 mm tubing was sealed to a length of 150 mm x 3 mm tubing, which was then pulled into a very shallow round bottom. Shrinking the tubing at the bottom was done so the outer wall would be closer to the top of the inserted Ace valve when the unit is fully assembled and to eliminate some unnecessary jacket volume, again to save on the final weight of the unit (Images 23 & 24).

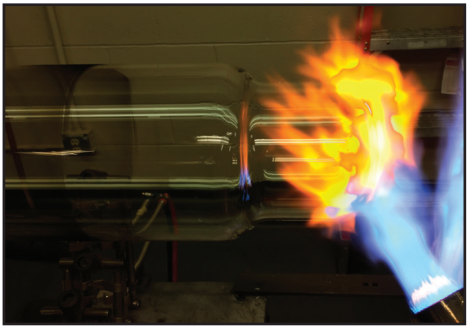


Image 23

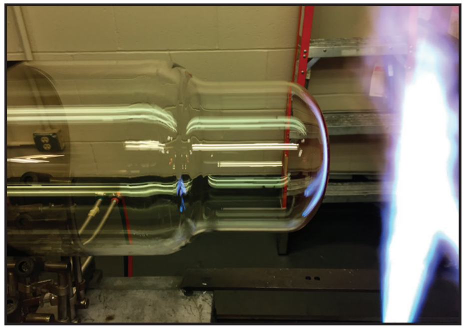


Image 24

To ensure precise placement of the 29/32 inner joint on the jacket, I decided to drill out the hole for the joint rather than simply blow and pick out the hole. By tilting the head of my milling machine, I was also able to precisely set the angle of entry to 120° so that the joint could be later sealed on more accurately (Images 25 & 26).

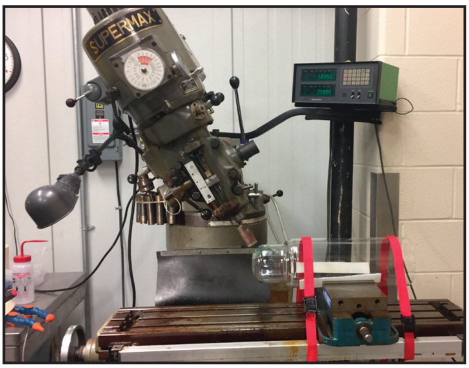


Image 25

Image 26

With the jacket drilled out and cleaned up, the inner unit could now be finally placed inside the jacket. Notice the drain tube sticking out of the drilled hole. The inner unit was secured with corrugated cardboard. It was critical that the drain tube be exactly on center in the drilled hole and that the tube maintain its angle of 120°. For that reason, it became necessary to intentionally place the funnel off center inside the jacket (Images 27 & 28).

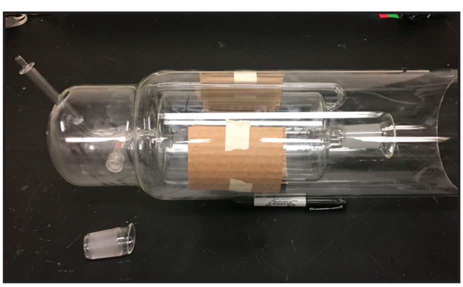


Image 27

Image 28

The inner joint was then prepared. The bottom of the grind was tapered slightly to more closely match the flare I put on the drain tube much earlier, and the joint was test fit (Images 29 & 30).

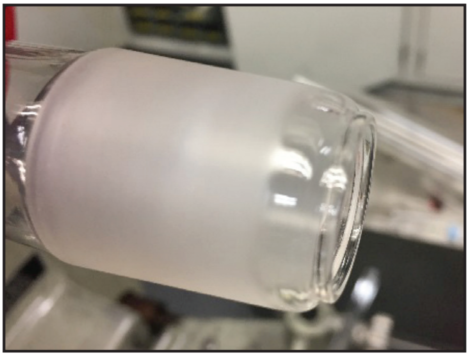


Image 29

Image 30

The unit was then able to be heated up and the joint sealed on. Special care was needed when sealing the joint on; the 110 mm jacket body had to be hot enough to not crack, but the unit had to be cool enough to not let the ¼" MW tubing sag at all. The joint was tacked on and then stitched closed with 2 mm rod top and bottom, and the GL-14 thread that is used for the hose connector was placed on as well. This was then flame annealed (Images 31, 32, 33 & 34).

Image 31

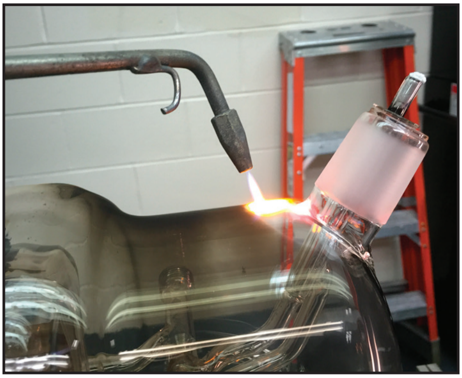


Image 32

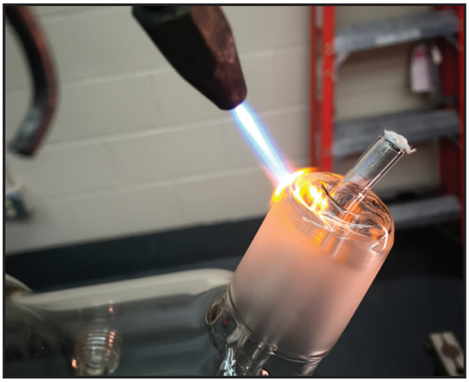


Image 33

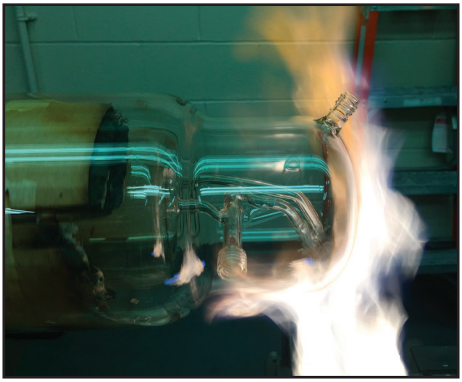


Image 34

The top had to be assembled and sealed in without running the unit through the oven, because at this point, the only thing holding the funnel to the body is the dewar type seal connecting the bottom of the 29/32 joint to the 9 mm SW tubing that is part of the pressure relief arm; so the cardboard had to stay in place. The 150 mm tubing was drawn down into a round bottom directly onto the 24/40 dewar seal joint, the seal was made and worked in, trapping the cardboard inside the jacket. The second GL-14 thread was then connected, and the unit flame annealed (Images 35, 36 & 37).

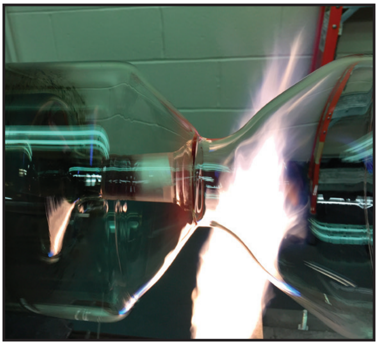


Image 35

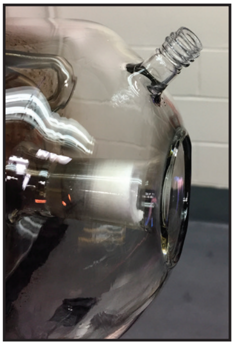


Image 36

Image 37

The unit was annealed in the oven with a very small, slow feed of air directly into the jacket to help combust and burn away the cardboard (Image 38).

Image 38

At this point, the Ace valve had not yet been sealed in to finish the glassblowing portion. The unit was heated up and the seal made. The 110 mm tubing is side ring sealed directly to the top of the Ace valve and the opening blown out. Special care must be taken that you do not over blow the seal or get it too hot since that can distort the thread and ruin the valve. If the valve is ruined at this phase, you will likely have to rebuild the entire unit from scratch (Images 38, 39 & 40).

Image 38

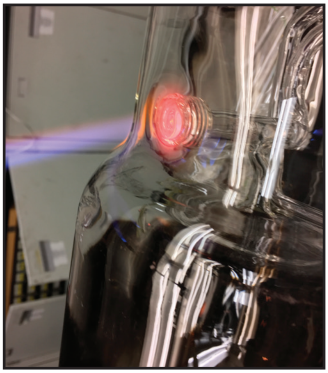


Image 39

Image 40

The final step was simply to regrind and lap in the 29/32 inner joint.

This project posed many difficult challenges and forced me in a few places to reassess my conventional methodology that I usually use while glassblowing. In the end, the results were elegant and simple; the customer received a new addition funnel that, while heavier than their original, functioned the same and looked very like what they were using (Images 41, 42 & 43).

Image 41

Image 42

Image 43

Glass Liner for 5L Glass and Stainless Steel Reactor

by
Doni Hatz*

ABSTRACT

A glass liner is made for a 5 liter glass and stainless steel reactor. The researchers fill the dish with a salt water solution to reduce corrosion of the stainless steel during the experiment. The challenge was to shape a 175 mm outside diameter glass tube to the radius of the metal dish and to avoid two 10 mm probes and an impeller that are not submerged in the fluid.

A researcher requested that a glass liner for a 5 liter reactor be made for upstream research and development. The stainless steel reactor needed a glass dish to hold a salt water solution so as to reduce corrosion of the stainless steel during the experiment. The challenge was to shape a 175 mm outside diameter glass tube to the radius of the metal dish and to avoid two 10 mm probes and an impeller that are not submerged in the fluid.

The glass liner had to fit inside a reactor that has a stainless steel lid with several probes and a stirring mechanism. The middle section is an 18 cm inner diameter glass tubing with a flat flange on top and bottom. The stainless steel base has a dish shaped bottom inside. The contour of the dish is important along with avoiding two 10 mm probes used in this experiment as well as the impeller. The glass dish is 175 mm outside diameter (o.d.) at 12 cm height with two indents for the probes to reach to the bottom of the reactor and another tube sealed inside the dish to shield the impeller (Photo 1).

Photo 1

* The Procter & Gamble Company, 8700 Mason Montgomery Road, Mason, Ohio 45040. Email: hatz.dj@pg.com.

All dimensions are captured in a simple blueprint. The top view of the blueprint shows the areas where the indents are needed for the probes and the size of the inside tube (Photo 2).

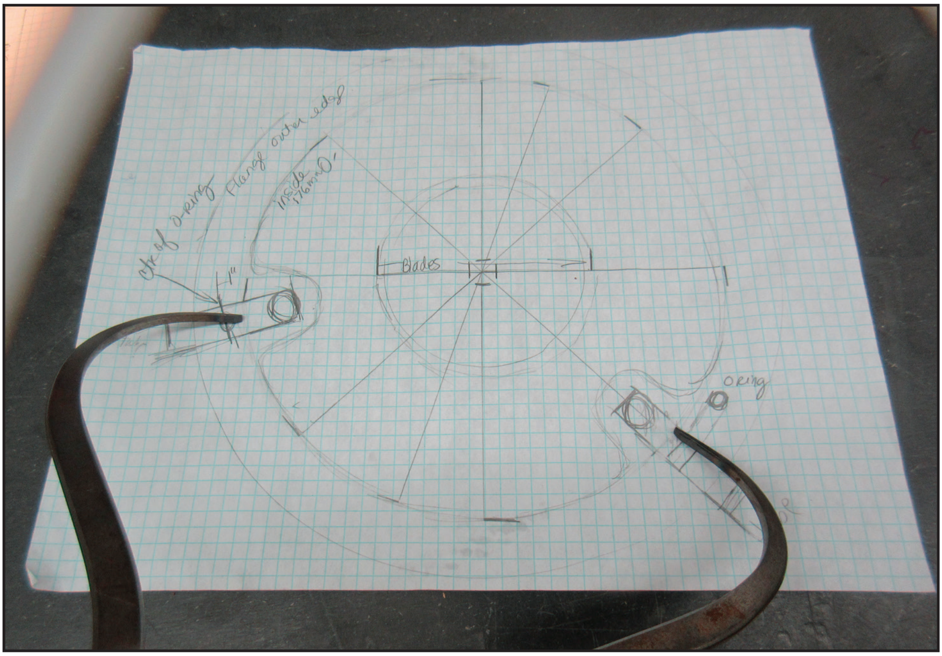


Photo 2

THE MATERIALS FOR THIS PROJECT

150 mm o.d. x 4 mm wall thickness (standard wall) tubing

80 mm o.d. x 3 mm wall thickness (standard wall) tubing

25 mm o.d. x 3.2 mm wall thickness (medium wall) tubing

SHAPING THE DISH

A 150 mm o.d. tube is chucked into the lathe (Photo 3). A short piece of 100 mm tubing (already sealed to 50 mm tubing as a handle) is heated and flared up to the 150 mm (Photo 4). The tubes are sealed and pulled down in the lathe (Photo 5). The round bottom is tooled down with a one-inch graphite rod (Photo 6). The tube is gathered slightly, blown to the 175 mm o.d. (Photo 7) and checked with the calipers (Photo 8); then more of the

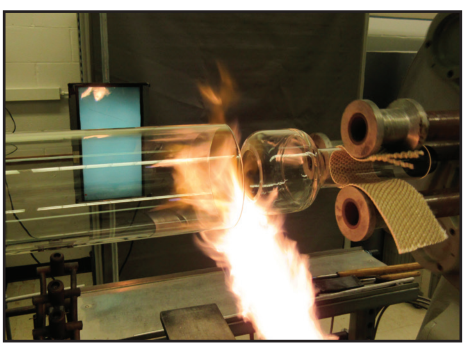


Photo 3

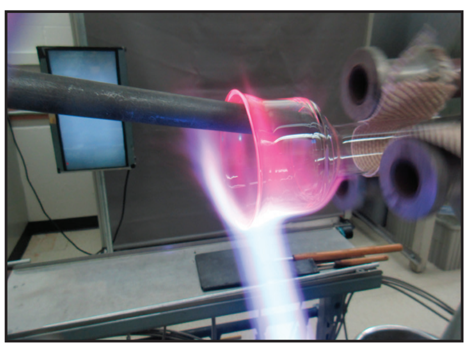


Photo 4

tubing was blown up, gathering the glass enough to keep a consistent wall thickness for another six inches of tubing (Photo 9). The round bottom end is shaped to specifications and the holder is pulled off on the right side (Photo 10).

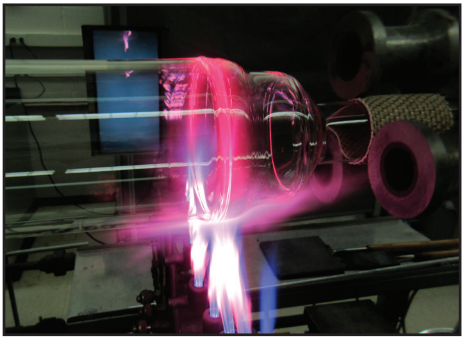
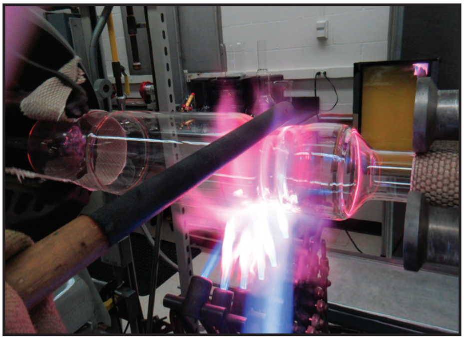


Photo 5



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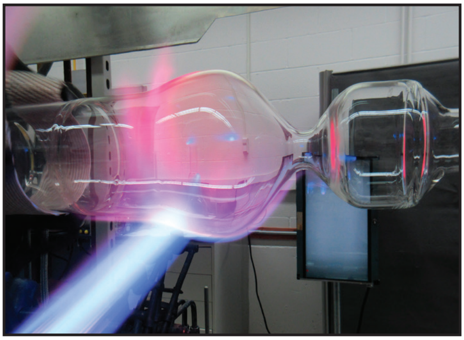

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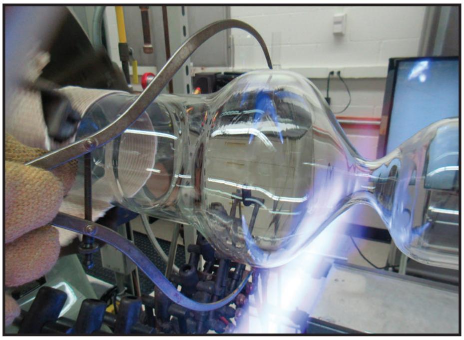

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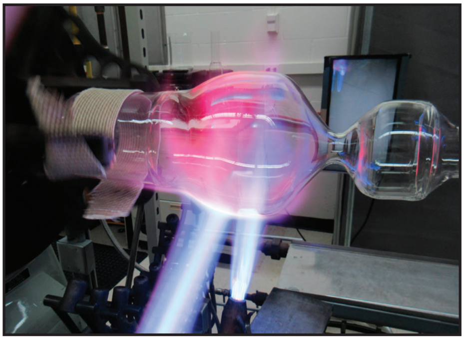


Photo 9

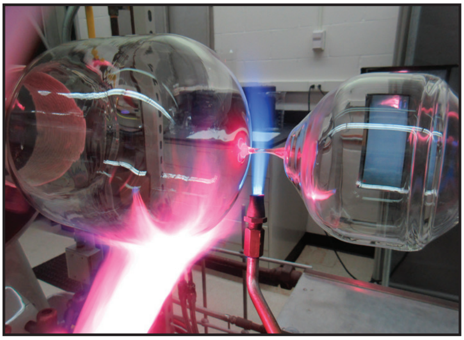
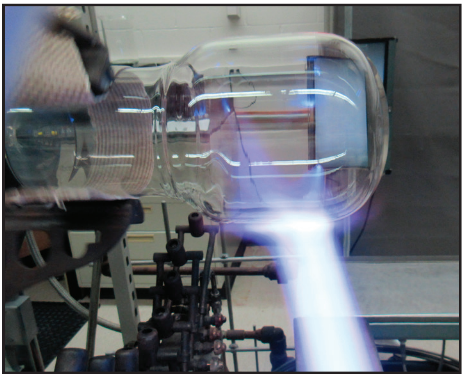


Photo 10

SHAPING THE INDENTS (FOR THE PROBES)

The entire 175 mm o.d. tubing section is heated to prepare for melting the glass inward creating the indents (Photo 11). The tube is marked with a line where the indents must be with a scrap piece of glass since a Sharpie® burns off at temperature. Using a Carlisle CC hand torch, the tube is heated on the top side, straight down onto the tube (Photo 12) sweeping it towards the middle (Photo 13) and on the end, near the round bottom (Photo 14). In this case I did not have to make the indent with tight sharp edges that can thin out the glass; this wide indent allowed for good wall weight in case the researchers bump it with the 10 mm stainless probes.



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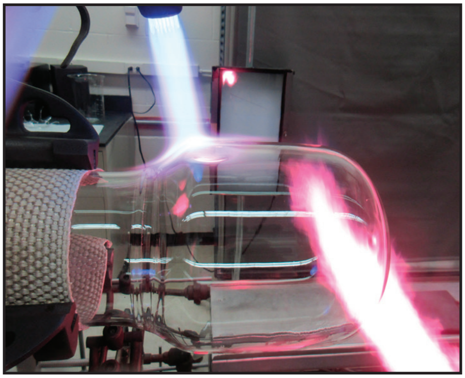

Photo 12

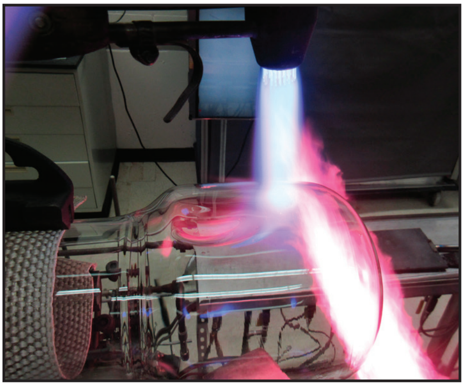

Photo 13

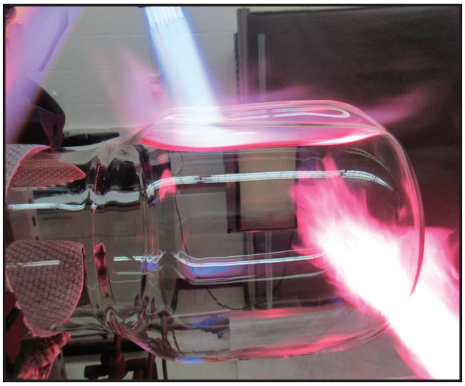

Photo 14

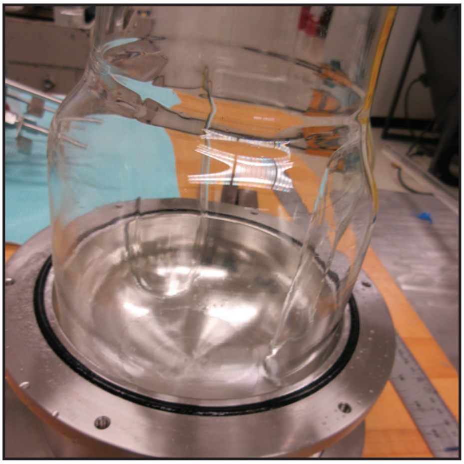

Photo 15

The tube has been annealed and tested for fit inside the stainless steel dish base (Photo 15). The indents are checked for proper alignment on the blueprint (Photo 16).

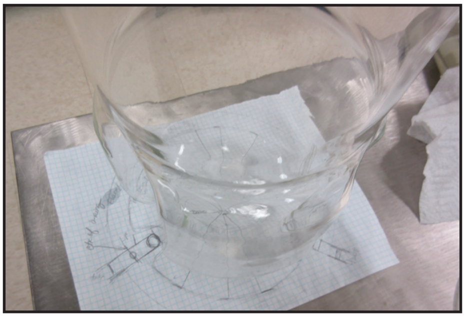

Photo 16

The tube is trimmed down at the diamond saw with an extra half inch of tubing in case of breakage. The tube is carefully cut from both sides since the indent makes it awkward for cutting on the diamond saw (Photo 17). The tube is now fitted inside the reactor with the lid and the probes clear the glass tube (Photo 18). The final height is determined (Photo 19) and trimmed down using the diamond saw.

Photo 17

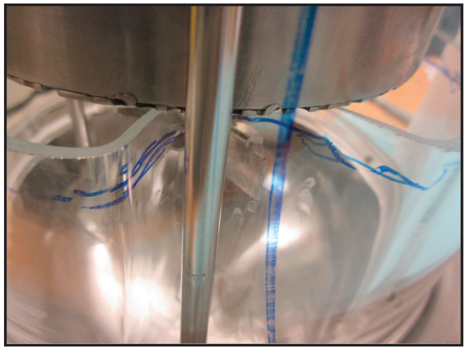

Photo 18

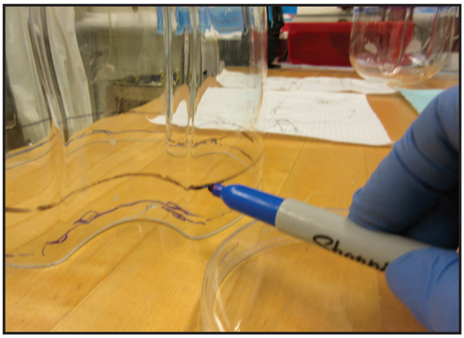

Photo 19

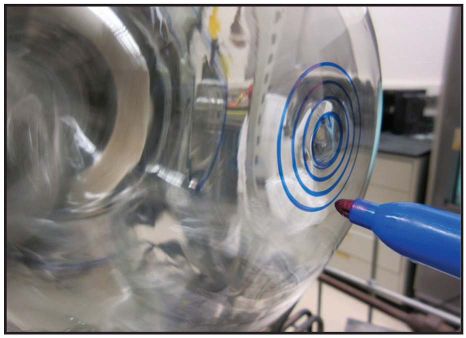

Photo 20

PREPARATION OF THE RING SEAL (THE INSIDE TUBE)

The 175 mm o.d. dish is prepared for sealing the inside piece that protects the stirring mechanism, the impeller. The tube is supported in the lathe. Using a Sharpie®, several grid lines are drawn on the round bottom to be aware of the center for the ~30 mm hole for the ring seal (Photo 20). Using a Foredom flex shaft tool with a little bit of water dripping only

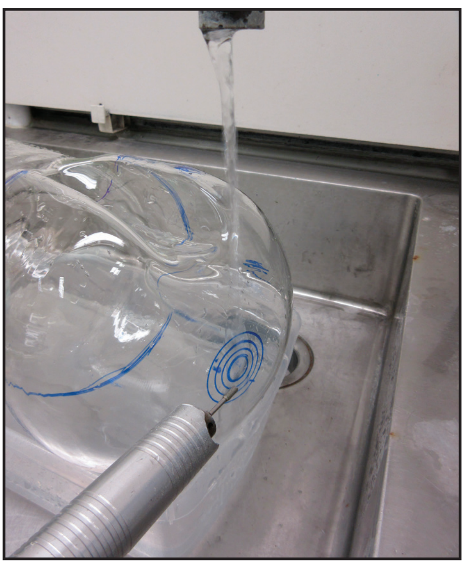

Photo 21

Photo 22

on the glass area (Photo 21), the ~30 mm hole is cut into the tube (Photo 22). The dish is fitted inside the reactor again to check the height (Photo 23).

THE INSIDE STIR BLADE GUARD

A piece of 80 mm o.d. tubing is chucked into the lathe and pulled down with a flat end. A hole is blown out of the center of the flat end and sealed to 25 mm MW tubing. The tube is annealed in the oven. The next day the 25 mm tube is trimmed down and double checked with the blueprint to about 80 mm length (Photo 24). The tube is chucked into the lathe (Photo 25) and a bead is shaped on the end of the lip of the tube (Photo 26) and flame annealed. Once it is cool, the height is calculated to trim the 80 mm o.d. tube to 30 mm length for a total length of 120 mm for the entire piece.

Photo 23

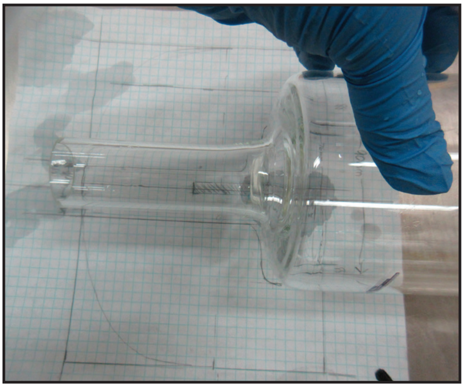

Photo 24

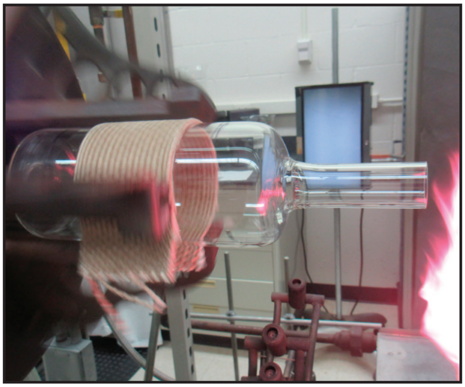

Photo 25

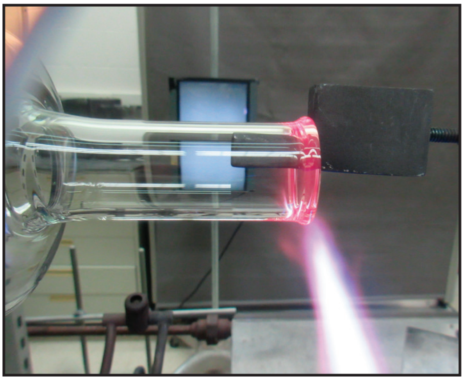

Photo 26

Photo 27

Photo 28

The 80 mm - 25 mm glass piece is flipped upside down with the 25 mm tube upward to fit flush through the hole inside the 175 mm dish. The dish and the stir blade guard are placed in the oven on a steel plate 3/8” thickness x 12” x 12” with graphite paper strips placed in between the metal and the glass (Photo 27). The oven is heated up to 600°C and held at temperature for 10-15 minutes before fusing the ring seal. The Wilt Oven Model 120 is a front-loading oven where the door swings left. I am able to slip the National hand torch fitted with #3 tip, a 12” long neck and metal knobs on the gas and oxygen to handle the heat briefly. Personal protective equipment is a must! I always wear 18” Kevlar sleeves, thick double layer Kevlar gloves, a lab coat and a clear view face shield over my didymium lenses. Small sections of the 25 mm tube are heated in the middle with the graphite rod pressing the glass together a little bit at a time (Photo 28). You must work quickly and fast at 600°C in a small oven. I will preheat the tweezers to spin and rotate the dish for the best angle to fuse and flow the ring seal with the hand torch (Photo 29).

The last step is to fire polish the top lip of the dish and middle piece in the small oven. The same method of heating it up to temperature in the small oven is used to gently fire polish the edges (Photo 30).

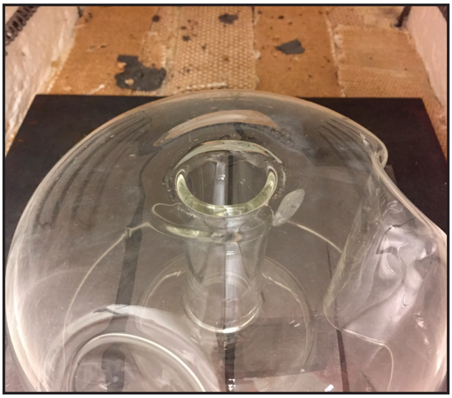

Photo 29

Photo 30

Photo 31

Photo 32

The dish is completed (Photo 31) and placed inside the reactor ready for the researcher to use (Photo 32).

Challenges are routine in our profession but they make our work interesting.

ACKNOWLEDGEMENTS

Many thanks to Procter & Gamble managers Tim Fiedeldey and Matt Paumier for their continued support.

Cold Working in the Scientific Glass Shop

by
Patrick Bennett*

ABSTRACT

Achieving clean seals through cold work.

When I first started my adventure into blowing glass on a professional level, I began to be annoyed with the traditional approach to prep work by scoring, snapping, peeling out the score mark, and then re-working the wall loss in the seal to make it look clean and uniform without any distortion in the seal.

At the time, I was working for the University of Notre Dame, and discovered that if I wet-saw cut my tubing before making seals, and applied a quick swab of 49% hydrofluoric acid followed by neutralizing the acid and a distilled deionized water rinse, as long as I fire-polished the tube end slowly enough, I would obtain perfectly clean seals with no inclusions or score lines.

When I took my job at the University of Minnesota several years later, it had never even occurred to me that I would not have the necessary fume hood to work with HF. So, I started looking for cheap fume hoods that would meet my University's very strict safety standards. After searching to no avail for several more months, I decided it was time to implement a new approach where I eliminated the need for caustic materials entirely.

Several months had gone by, and in truth I had given up on my search for a solution almost entirely, when I came across something I found rather peculiar on YouTube. I unfortunately was too wrapped up in the method I was watching to take note of who was posting it, and in truth my first thought was "why, what a waste of time?" but what I had seen would change the way I approach seals from here on out.

In a very grainy, cell-phone-recorded video, sat an artist ponied up to his faceting machine's horizontal lap; he was meticulously grinding and polishing the ends of tubing preparing them for assembly. It actually took me a few days before it occurred to me why he was doing this, and it was genius. By taking the surface of his tubing down to a near optical flat, he had in fact found a method to create perfectly clean seals with perfect wall weight, without the need for fluorine ions to strip the surface and therefore all impurities left by the wet saw from the glass.

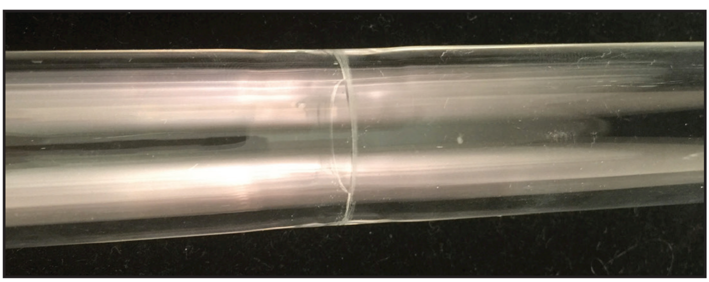

Photo 1

In Photo 1, you see the traditional wet saw cut, then "scrub the tube end before sealing approach." Notice the heavy white line through the seal.

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In Photo 2, the end of a piece of 1” heavy wall tubing is pictured right after the wet saw cut and cleaning with soap and a scrub brush. Note the striations left behind from the saw blade.

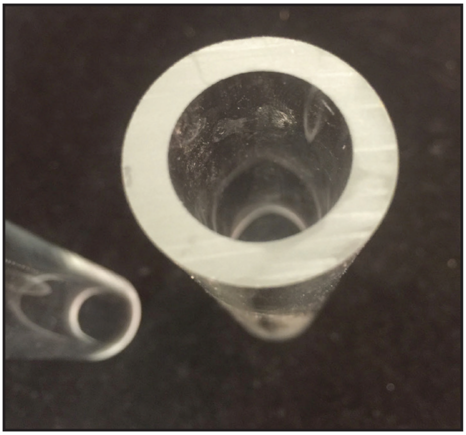

Photo 2

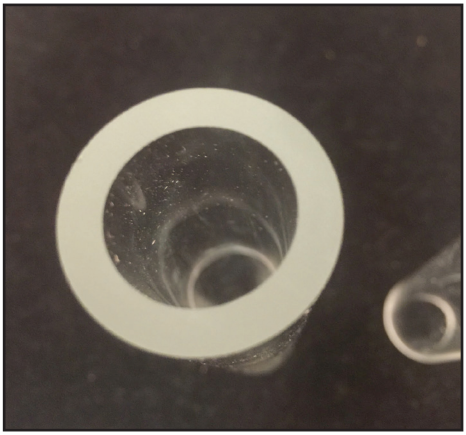

Photo 3

In Photo 3, after the wet saw cut is performed, the tube end is briefly worked on a 600 grit horizontal lap. Notice how the striations from the blade are now non-existent.

In Photo 4, after cleaning the 600 grit prepped tube ends in the ultrasonic cleaner, a seal is performed. Notice the lack of impurities and even witness lines in the seal.

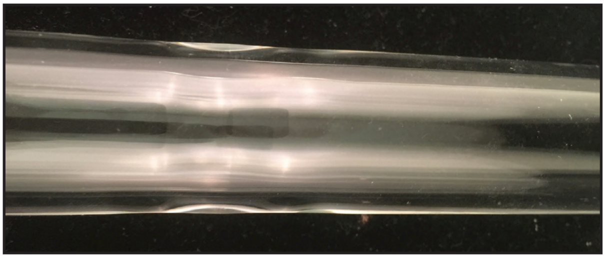

Photo 4

In Photo 5, a comparison of (from top to bottom) the 600 grit prepped and ultrasonic cleaned seal, a 600 grit seal without being cleaned first, and lastly, the simply wet saw cut and scrubbed with soap and water by hand seal.

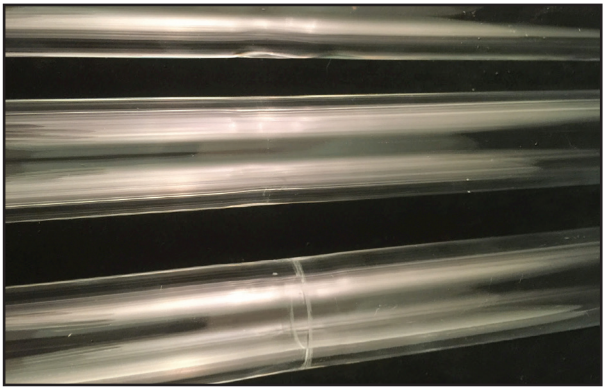

Photo 5

As you can see from the pictures on the right, by just adding in a few extra seconds to each seal with cold working, we achieve an incredibly clean seal that I happily send out to customers on a daily basis now.

Japan 2016 – Travelogue

by

Erich Moraine[*] and Tracy Drier[**]

ABSTRACT

In November 2016, we took a two and a half week trip to Japan to tour scientific glassblowing facilities. A total of 15 factory/production facilities and university research glassblowing shops in Tokyo, Nagoya, Osaka, and Kyoto were visited. Japanese glassblowers such as Hideaki Hashimoto and Tsuyoshi Nakamura have been attending and presenting at ASGS symposia for years. They welcomed our visit and were helpful in setting up the itinerary.

In November 2016, we took a two and a half week trip to Japan to tour scientific glassblowing facilities. Glassblowing traditions in Japan and the West developed independently, so there was much to be learned from observing their processes. We visited a total of 15 factory/production facilities and university research glassblowing shops in Tokyo, Nagoya, Osaka, and Kyoto. In addition to touring glass factories, we attended a regional glassblowing symposium in Tokyo during our visit where we provided demonstrations for the local Japanese glassblowers. The complete trip itinerary is shown in the Appendix.

The university glassblowing shops that we visited were similar to those here in the States in that they were spacious, clean, and well equipped. Their range of capabilities was wider than that of the usual university shop here: where we might typically buy a component, they have the means to produce that component in-house, which was true for most of the production glass shops we visited as well.

The production glassblowing facilities ranged from single-person shops using rudimentary methods, to factories that take up an entire city block and are highly automated. It was common that most were shops that had been passed down through generations. Often their 70 and 80 year old fathers still enjoy working five days a week. It was noted that many shops tended to specialize in one area – such as micro glassware, glass boxes, calibrated glassware, or custom grinding.

We had the chance to familiarize ourselves with their glassblowing burners, which were different from the ones to which we are accustomed. We were each presented with a gift — a metal tool called "hashi" (chopsticks) that was a new concept in glass tools for us. They are manipulated similar to chopsticks for eating, and for glass are used for flaring and constricting softened tubing. Since returning home, we have been busy trying to become proficient with them.

Glassblowing is an oral tradition; not much is written down. We tend to learn from the people we see and under whom we practice. Producing glassware for research is never routine; each project presents unique challenges and requires creative collaboration between the chemist and glassblower. In-person visits to glass shops far away has dramatically expanded our technical abilities, as well as improved our ability to problem solve. Seeing scientific glassblowing from a Japanese point of view has brought a new perspective to our knowledge base and skill sets.

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[**]University of Wisconsin – Madison, Chemistry Department, Madison, WI 53706. Email: todrier@wisc.edu.

A special note of thanks to Hideaki Hashimoto and Tsuyoshi Nakamura, our primary guides and contacts. Tatsushi Iguchi was instrumental in arranging our Osaka program. They went above and beyond the call of duty to help plan and organize a truly amazing visit.

APPENDIX

Glass Shop Survey: Japan 2016 Itinerary

Wed., Nov. 2	To Japan (Narita Airport)
Thurs., Nov. 3	Arrive Tokyo in PM; Skylier train (Keisei train) to Tokyo
Fri., Nov. 4	Tokyo Tokyo Seisakushiyo Co., Ltd. (contact: Hideaki Hashimoto)
Sat., Nov. 5	Tokyo Glass Workshop (www.TRGK.jp) at Maihara Mfg., Koskigaya-city Saitama.
Sun. Nov. 6	Tokyo 1. Nakamura Rika, Inc. (contact: Tsuyoshi Nakamura) 2. Furukawa (Friko) Science and Engineering (contact: Yuji Furukawa)
Mon., Nov. 7	Tokyo Cultural tourist day (travel to Nagoya)
Tues., Nov. 8	Nagoya Nagoya University Glass Shop (contact: Hideko Natsume) Travel to Osaka
Wed., Nov. 9	Osaka 1. Calibrated glassware Factory 2. Nakamura Rika Co., Ltd. (contact: Tatsushi Iguchi) 3. Miyamota Scientific Glass Co. 4. Grinding / polishing factory 5. Toshinaka Glass Factory
Thurs., Nov. 10	Osaka University (contact: Kazunori Watanabe); travel to Kyoto
Fri., Nov. 11	Kyoto; Yamaguchi Glass Corp. (contact: Shinnosuke Yamaguchi)
Sat., Nov. 12	Kyoto; cultural tourist day
Sun., Nov. 13	Kyoto; cultural tourist day
Mon., Nov. 14	Travel to Nagoya; travel to Tokyo
Tues., Nov. 15	Tokyo; Kiriyama (contact: Shimipei Takahashi)
Wed., Nov. 16	Tokyo; Ikeda glass (contact: Kouki Ikeda)
Thurs., Nov. 17	Tokyo; Hario Co., Ltd. (contact: Yusuke Kashiwabara)
Fri., Nov. 18	Tokyo; to USA (Narita Airport)

Fabrication of Liquid-filled Quartz Capillaries for Wavelength Shifting Applications

by
Kiva Ford*

ABSTRACT

This paper discusses the construction techniques involved in fabricating liquid-filled fused silica capillaries for wavelength shifting applications. The purpose of these capillaries, along with where they are used, will also be examined in this paper.

INTRODUCTION

The use of quartz capillary tubes containing wavelength shifting liquid is an effective way to measure light output in particle detectors. These detectors will be exposed to significant levels of potentially damaging radiation. The physics department at Notre Dame (QuarkNet) is working on calorimetry techniques for application in such high radiation environments by employing radiation tolerant shashlik-type electromagnetic calorimeters. The shashlik design uses a configuration of LYSO scintillation crystals and tungsten plates which are both dense and radiation hard materials. These materials allow the unit to be a compact, efficient, and robust detector. The liquid filled quartz capillaries run through the center of the LYSO and tungsten plates. The scintillation light emitted from the LYSO crystals is captured by the wavelength shifting liquid-filled quartz capillaries where it is coupled to photo sensors for readout. (The Shashlik calorimeter with four quartz capillaries running through the LYSO and tungsten can be observed in Photo 1 and Photo 2.)[1]

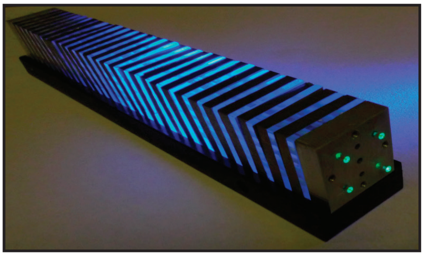

Photo 1

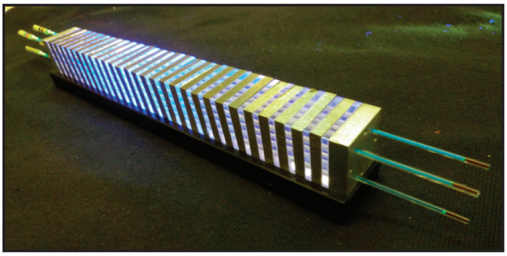

Photo 2

This shashlik calorimeter was designed with the intention of being used at the CMS (Compact Muon Solenoid).[2] The CMS experiment is a large particle detector on the Large Hadron Collider at CERN in Geneva, Switzerland.[3] The Large Hadron Col-

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[1] Mark Egan, *Thick-Wall Liquid Filled Quartz Capillaries for Wavelength Shifting Applications,* Poster presented at Graduate Student and Postdoctoral Fellow College of Science - Joint Annual Meeting (Notre Dame, Indiana: May 4, 2017).

[2] Wikipedia contributors, "Compact Muon Solenoid," *Wikipedia, The Free Encyclopedia*, https://en.wikipedia.org/w/index.php?title=Compact_Muon_Solenoid&oldid=802281089 (accessed November 13, 2017).

[3] Wikipedia contributors, "CERN," *Wikipedia, The Free Encyclopedia*, https://en.wikipedia.org/w/index.php?title=CERN&oldid=806752833 (accessed November 13, 2017).

lider accelerates 2500 bunches of protons around its loop every 25 ns with about 40 interactions per crossing occurring. As each of these high energy protons collides with another, particles are expelled from the collision. The energy from these particles would be converted into an electromagnetic shower by the shashlik and captured by the capillaries inside to be read out (Photo 3).

Photo 3. *CMS (Compact Muon Solenoid)*

CONSTRUCTION

The capillaries are made out of high purity Suprasil® chosen for its radiation hardness. The capillary blanks were purchased from Polymicro Technologies in Phoenix, AZ with an outer diameter of 1 mm and an inner diameter of 400 microns. Finished examples of the capillaries can be seen in Photo 4 and Photo 5.

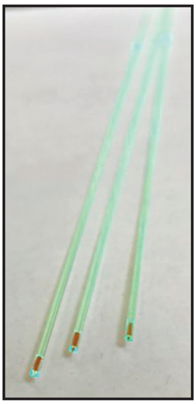

Photo 4

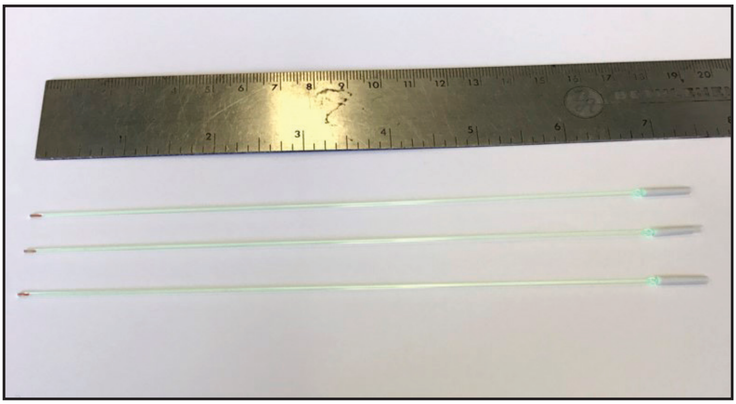

Photo 5

Photo 6

The ruby quartz core that is sealed into the end of the capillaries begins as a piece of 16 mm x 1 mm ruby quartz tubing. This tubing is broken up into pieces and hand pulled down to a solid rod with a diameter of 340 to 380 microns and then cut to the length of about 6 mm. This can be seen in Photos 6, 7, and 8. The ruby quartz core blocking absorbs light traveling through the wavelength shifting liquid, which can be absorbed by the liquid and re-fluoresced leading to longitudinal non-uniformity.

A hole is punctured into a rubber septa and inserted into a piece of rubber latex tubing which I use as a blow hose. The capillary is inserted into the septa. This

Photo 7

Photo 8

makes for a convenient way to get air pressure into the capillary. Then the ruby quartz core is inserted into the open end of the capillary. This can be seen in Photos 9 and 10.

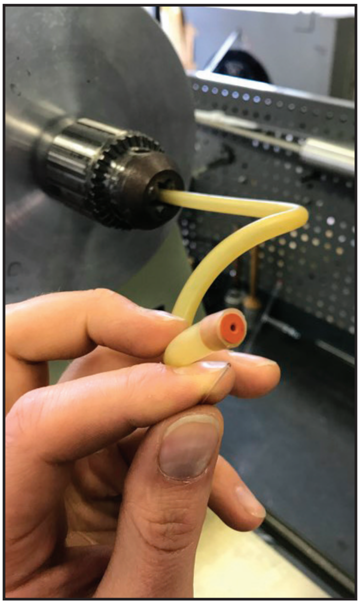

Photo 9

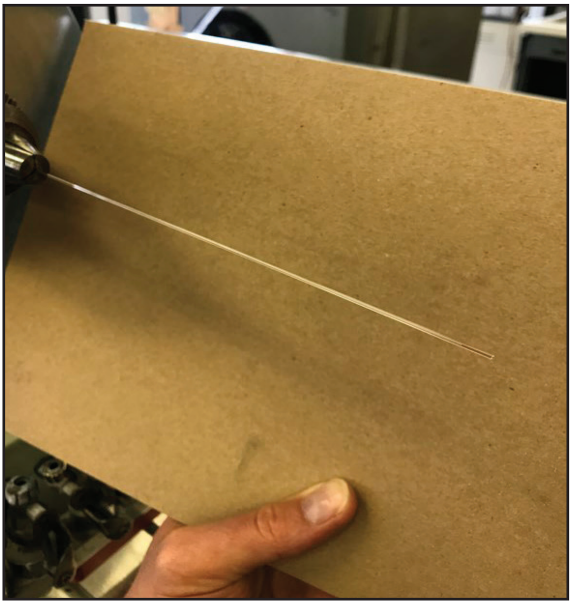

Photo 10

A quartz rod is temporarily sealed to the ruby quartz end which stabilizes the capillary as I constrict the capillary over the ruby quartz and then blow a 2 mm reservoir bubble. This can be seen in Photos 11 and 12. The reservoir bubble allows room for expansion and contraction of the wavelength shifting liquid.

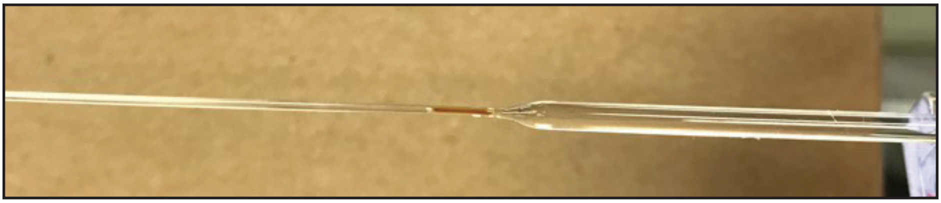

Photo 11

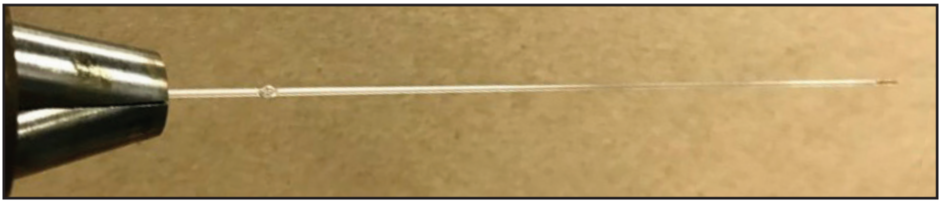

Photo 12

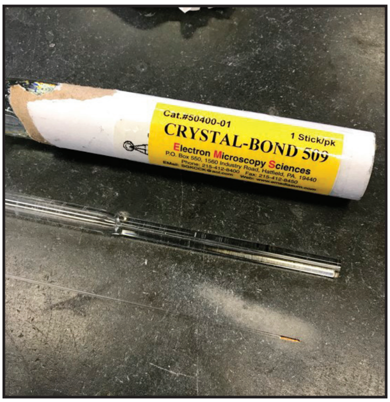

Photo 13

The capillary then needs to be measured, cut, and optically polished. In order to handle the tube during the cutting and polishing steps, I constructed a holder out of 8 mm x 2 mm borosilicate capillary sealed to 9 mm tubing. The quartz capillary is inserted into the borosilicate capillary and temporarily secured using Crystalbond™ 509. I measure the length on the ruby core to 3 mm using a microscope and then cut through the holder using a diamond wet saw. I then use a lapping wheel to polish the ruby quartz end. This process can be viewed in Photos 13, 14, 15, and 16. After these steps are complete, the capillary is removed from the holder.

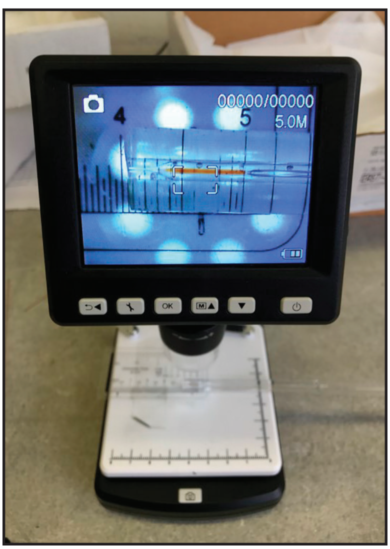

Photo 14

Photo 15

Photo 16

Once the capillaries are cut and polished, they need to be filled with the wavelength shifting liquid. Vacuum is used to suck the fluid into the capillaries. The capillaries are connected to a manifold and a turbomolecular pump is used to pump them down to 10 to the -6 millitorr. The capillaries are then sealed off under vacuum. The sealed off capillaries are then inserted into a borosilicate reservoir and temporarily held in the reservoir using paraffin wax. The reservoir is filled with the wavelength shifting fluid and then a pair of tweezers is used to break the end of the capillary. Once the capillary is broken, the wavelength shifting fluid is sucked into the capillary. This can be viewed in Photos 17 and 18.

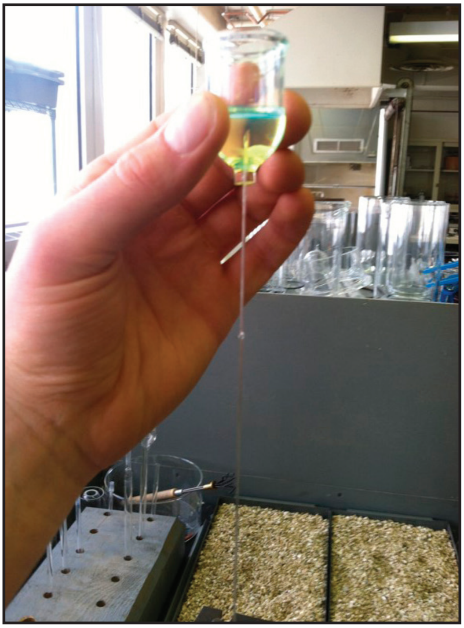

Photo 17

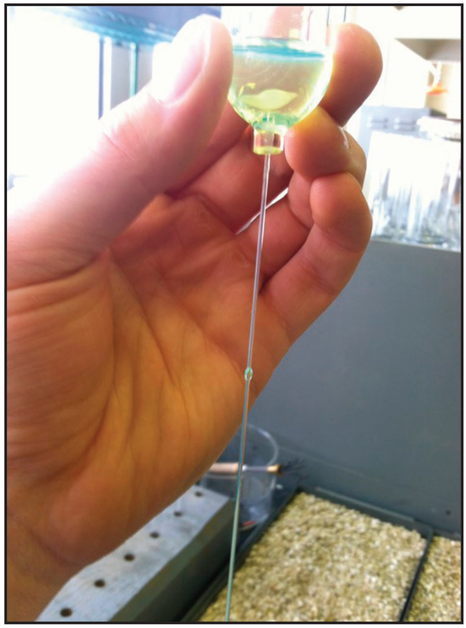

Photo 18

Finally, the excess fluid is removed by pushing a 380 micron thick rod into the capillary down to the reservoir bulb. This will give extra space for the fluid to expand without breaking the capillary. The open end of the capillary is then capped by filling a piece of plastic tubing with Torr Seal® and pushing it over the capillary. This can be viewed in Photos 19 and 20.

Photo 19

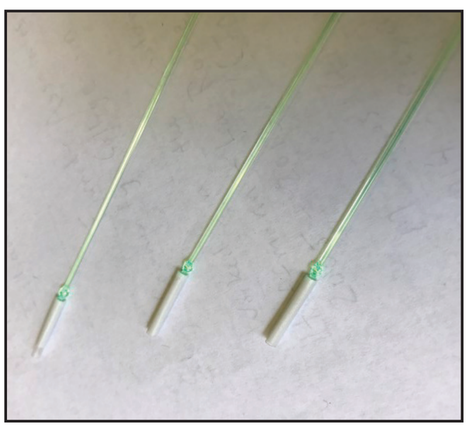

Photo 20

This process was used with great success a few dozen times. The researchers were very happy with the quality of these detector tubes. The researchers then informed me that they would like to place an order for a thousand more tubes, and ultimately they would need about a quarter of a million tubes for their finished detector! I realized that I needed to innovate a way to make these tubes faster, so I ended up altering my process. I will now explain how I altered this process in a way that allowed me to fabricate many tubes at the same time.

I decided to bundle the tubes for the cutting and polishing process. A piece of half inch borosilicate tubing was used to hold the capillaries in place and the capillaries were secured with Crystalbond™ once again. In order to get all of the ruby quartz ends to line up for the cutting process, I measured the distance from the end of the ruby core to the end of the open capillary and cut each capillary to the same length. I could then hold the tube vertically and all of the capillaries would line up evenly. This can be seen in Photos 21 and 22. I then measured, cut, and polished in the same manner as I did originally. Each bundle holds about thirty capillaries. This can be seen in Photos 23, 24, and 25. The capillaries are then removed from the borosilicate holder and moved to the lathe in order to blow the reservoir bubbles.

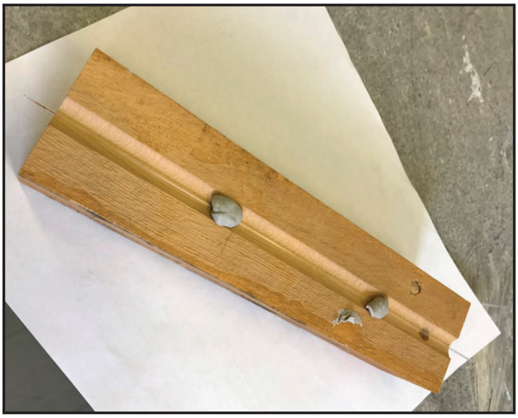
Photo 21


Photo 22


Photo 23

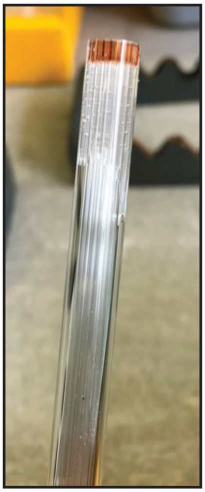
Photo 24


Photo 25

Finally, the capillaries need to be filled with the wavelength shifting fluid. I constructed an apparatus which held about 100 capillaries and the wavelength shifting fluid. The capillaries are placed in the fluid with the open end down. The apparatus is then connected to the turbomolecular pump and pumped down. Once sufficient vacuum is reached, the apparatus is isolated from the turbo pump and the side valve is opened which exposes it to atmospheric pressure. The pressure then forces the liquid up into the capillaries. This is an old process which has been used by thermometer manufacturers for years. This can be viewed in Photos 26 and 27. I should also mention that the filled capillaries always have a small air bubble (about 1 mm in length) at the base of the red quartz after they are filled with the wavelength shifting fluid. The researchers in the physics department place the capillaries in a custom built centrifuge which moves the bubble from one end of the capillary to the other. This process removes the bubble and creates a uniform line of wavelength shifting fluid from the reservoir bubble to the red quartz.

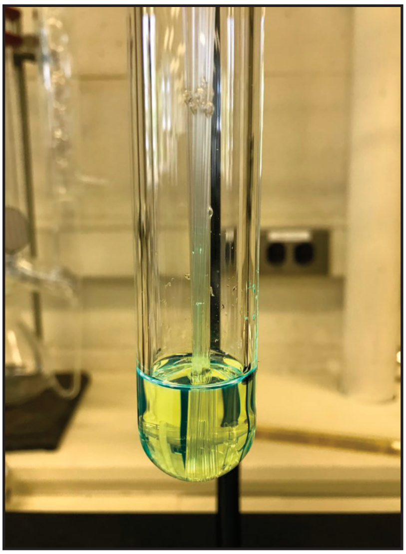

Photo 26

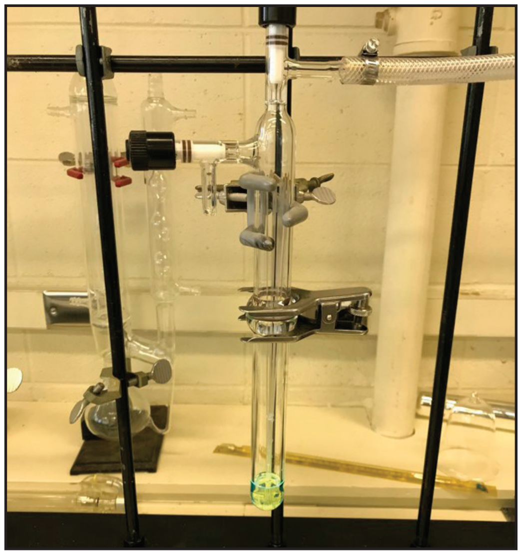

Photo 27

Changing these steps allowed me to fabricate the detector tubes at a much faster pace. It is interesting to see the evolution of a glass blowing project and how making a few dozen of a certain piece can greatly differ from making a few thousand of a certain piece.

ACKNOWLEDGEMENTS

It was an honor to work on this project. I would like to thank everyone at QuarkNet including Randal Ruchti, Barry Baumbaugh, Mark Vigneault, and Mark Egan for answering all of my questions about electromagnetic calorimeters. I would also like to thank them for the experience of collaborating on this project with them. Also thank you to the University of Notre Dame, the Radiation Research Laboratory, and the American Scientific Glassblowers Society.

Calculating the Weight of a Cylinder

by

Richard J Ponton*

ABSTRACT

A simple request for an open topped cylinder became incredibly complex when the customer requested multiple cylinders that had to weigh the same. This paper describes how I calculated their mass before making them.

As with many of our most complicated jobs, they often start out easy. In this case, a customer came to me looking for a simple flat bottom, open top cylinder. Length and diameter were not important so I took a piece of scrap 70 mm tubing I had, pulled a flat bottom, gave it to them and put this job out of my mind (Photo 1).

Photo 1

Weeks later, that same customer returned, asking for some more of "those cylinders." However, instead of some basic cylinders, they wanted a set of three cylinders, one each of three different preset diameters. Each unit's height was variable, and the customer did not care what that diameter was. The hard part: all three cylinders were required to have the same mass with a tolerance of +/-0.0005 grams. What had started out as a super easy, throwaway type of job instantly became one of the more mathematically challenging jobs I have worked on.

The initial idea was to make the glass cylinders and simply start cutting and grinding them, weighing them along the way until they matched. I called this the "brute force" technique. This seemed like it would require too much grinding, washing, drying and weighing over and over. What I needed was a way to predict the unit's mass before I built them so they would already be as close to the same mass as practical when they were built. To arrive at my technique, I started by asking myself two questions: what do I know about glass as a physical material, and what do I know about geometry in general. I knew I could calculate the volume of the actual glass material in such a simple shape; it was, after all, nothing more than a hollow cylinder and a solid disc, but volume by itself did not give me the information I was seeking. Thanks to *The Glass Engineering Handbook*, by E.B. Shand,[1] I also had knowledge of the density of borosilicate glass. Combining the density and volume, I reasoned, I could figure out a predictive mass of the material.

Density (or to be specific the volumetric mass density) of a material is its mass per unit of volume. If the density of borosilicate and the volume present are known, finding out the mass becomes a very simple algebraic function. The density of a material is found by using

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[1] E.B. Shand, *Glass Engineering Handbook*, 3rd Edition (McGraw Hill, Inc., 1984): 2-2 & 2-3.

the following formula: $d = {}^{m}/_{v}$. Since I knew the density and the volume, I simply rearranged the formula to isolate the unknown variable thus: $m = d * v$. Borosilicate glass has a density of 2.23 g/cm³. What was the volume of material needed? To obtain this, I began by calculating the volume of the initial sample I had made for the customer.

The initial unit was constructed of 70 mm x 2.3 mm tubing, had an overall length of 154 mm, and had an internal height of 150 mm. This hollow cylinder is essentially two geometric components: a hollow cylinder and connected at the bottom is a disc of solid material which is geometrically nothing more than a very short, solid cylinder. To calculate the volume of the glass in the cylinder, I used the following formula: $v = \pi R^2 h - \pi r^2 h$, where h is the internal height, R is the o.d. radius, and r is the i.d. radius. For the bottom, I calculated $v = \pi R^2 t$, where R is again the o.d. radius, and t is the thickness of the bottom. Add these two values, and you get the total volume of glass material: $v = \pi R^2 h - \pi r^2 h + \pi R^2 t$. To simplify conversions, I decided to calculate everything in cm, as opposed to our industry standard mm. Since the density is mass per centimeter cubed, this simple step saved me messy conversions later.

$$v = \pi R^2 h - \pi r^2 h + \pi R^2 t$$
$$v = \pi 3.5^2 15 - \pi 3.25^2 15 + \pi 3.5^2 0.4$$
$$v = 94.9154\ cm^3$$

Once I had the volume calculated, I went back to my algebraically reworked density formula and simply plugged in the numbers. (Normally one does not include units when doing math, but I added them in this case so you could see how the units cancel out leaving only the mass unit.)

$$m = d * v$$
$$m = 2.23\ \frac{g}{cm^3} * 94.92 cm^3$$
$$m = 211.661 g$$

My answer was completely wrong! The unit as calculated should have weighed in 35 grams more than the as measured value was. There clearly was something wrong with my methodology. Initially, I checked my math, which was right. The formula was right, so I turned my attention to the glass itself. Taking a serious look at the unit I made, it became clear that what I calculated was a fundamentally different piece of material than what I had made. My calculation was based off a straight hollow cylinder with a solid disc connected to the bottom. What I had made was something with a firecut and beaded top, and a pulled bottom. A pulled bottom, no matter our skill level, can never be as consistent in its uniform thickness as I calculated. As it turns out, when I measured the inner height (150 mm, remember?) my caliper had hit a high ridge of material coming off the radius. The bottom was only around 1.5 mm thick, not 4 mm. Rather than try to account for all of these variables, which would be possible but would have made the calculation insanely complicated, I redesigned the glassware to reflect my math (Photo 2).

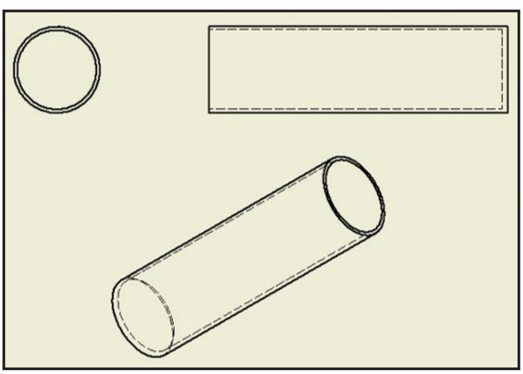

Photo 2

Since the math was based off a hollow cylinder with a disc, I decided to make my new set using tubing cut on my saw with a glass disc sealed on the bottom using a vacuum chuck. Diameters my customer and I agreed upon were 56 mm, 65 mm and 70 mm tubings, with glass discs on the bottom of 2.25” 2.5” and 2.75” respectively. Since the diameters and mass were set numbers, and the height was the variable, I reasoned the smallest diameter unit would need to be the tallest, the largest the shortest. To make my initial test piece and verify that this concept would work, I began with the smallest diameter unit. Since the client did not care much about the height, I could arbitrarily set the height of the first unit (the other two sizes would be shorter in relation to this first one). I selected 19 cm because it was the tallest length I could stand up inside of the enclosed 5 place balance I was using for the project. Recalculating got me a new mass number.

$$v = \pi R^2 h - \pi r^2 h + \pi {r_{disc}}^2 t$$

$$v = \pi 2.8^2(19) - \pi 2.55^2(19) + \pi 2.8575^2(.3175)$$

$$v = 87.98$$

And density

$$M = v * d$$

$$M = 87.98 * 2.23$$

$$M = 196.196\ g$$

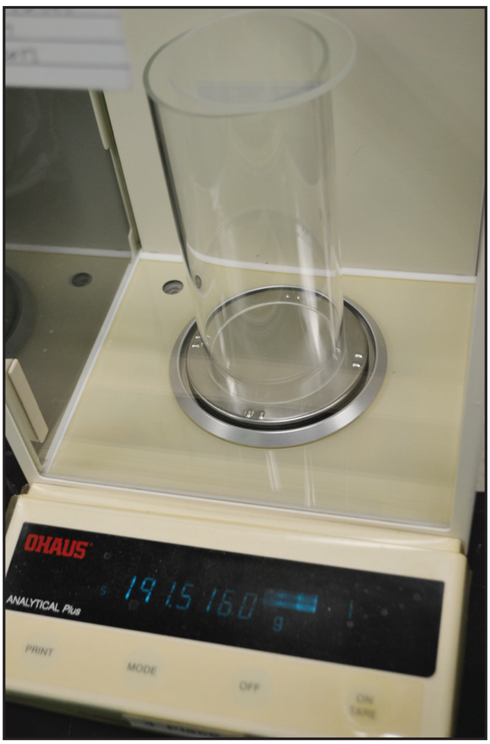

Photo 3

With this number calculated, I went to the saw, cut a length of 56 mm tubing at 190 mm, and once it was washed and dry, set it in my balance enclosure with a 2.25” glass disc sitting on it to see if this methodology would work (Photo 3).

This time the calculation was correct within 5 grams. Considering that I measured the tube with a ruler and marked it with a Sharpie®, I considered this an acceptable tolerance and that my method did, in fact, work. I resolved to be more precise in my cut to try and get closer going forward.

As I began to think about the calculations, it very quickly became obvious I was dealing with a lot of numbers: I had three different outer radii, three inner radii, three disc radii, a known volume, a known density, two known heights, and two unknown heights. I listed all 15 numbers and assigned them algebraic letter designations to keep them straight:

h_1 = Height of large cylinder in cm: __?__
h_2 = Height of medium tube cylinder in cm: ___?___
h_3 = Height of small tube cylinder in cm: 19 cm
h_4 = Thickness of disc in cm: 0.3175 cm
r_1 = Outer diameter radius of large tubing in cm: 3.5 cm
r_2 = Inner diameter radius of large tubing in cm: 3.277 cm
r_3 = Outer diameter radius of medium tubing in cm: 3.25 cm
r_4 = Inner diameter radius of medium tubing in cm: 3.015 cm
r_5 = Outer diameter radius of small tubing in cm: 2.8 cm
r_6 = Inner diameter radius of small tubing in cm: 2.55 cm
r_7 = Radius of large disc in cm: 3.4925 cm
r_8 = Radius of medium disc in cm: 3.175 cm
r_9 = Radius of small disc in cm: 2.8575 cm
v = Volume of glass in cm^3 (ml)
d = Density of glass 2.23 g/cm^3

You will remember the full volume calculation for the flat bottom cylinder looked like this: $v = \pi R^2 h - \pi r^2 h + \pi r_{disc}{}^2 t$. To ease the calculation, I again algebraically reworked the formula [illegible]late the unknown variable (h).

$v = \pi R^2 h - \pi r^2 h + \pi r_{disc}{}^2 t$ becomes $h = \dfrac{v - \pi r_{disc}{}^2 t}{\pi R^2 h - \pi r^2 h}$

I had three cylinders, but since I was allowed to select the height of one of them (the 5.6 cm tube), I only had two unknown heights to calculate

For the 6.5 cm tube, the calculation worked out to be:

$$h_2 = \frac{v - \pi h_4 r_8{}^2}{\pi r_3{}^2 - \pi r_4{}^2}$$

$$h_2 = \frac{87.98 - \pi.3175 * 3.175^2}{\pi 3.25^2 - \pi 3.015^2}$$

And the 7.0 cm tube calculated to be:

$$h_1 = \frac{v - \pi h_4 r_7{}^2}{\pi r_1{}^2 - \pi r_2{}^2}$$

$$h_1 = \frac{87.98 - \pi.3175 * 3.4925^2}{\pi 3.5^2 - \pi 3.277^2}$$

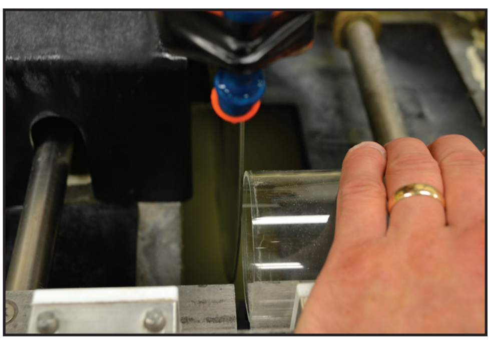
Photo 4

After all that math work, I was finally able to begin construction. Being as precise as my tools allowed, I cut the 65 mm and 70 mm tubing to the calculated lengths (Photo 4).

After cutting, washing and drying them, I weighed all three in a similar fashion to my test case: the clean, dry glass cylinder with the disc just sitting on top. I did not want to spend the time to assemble the units unless the weights were right.

After being cut, my three units came out with the following masses:

5.6 cm tube: 189.4271 grams
6.5 cm tube: 188.0161 grams
7.0 cm tube: 191.5151 grams

Since no target weight was given, as long as the three units matched, I selected the unit with the lowest mass to be my target mass to get the other two down to. The 56 mm tube needed 1.411 grams of material removed, and the 70 mm tube needed 3.499 grams of material removed. I knew that in the end I would need to do some fine-tuning grinding using basic "brute force" work, and I reasoned that the 56 mm tube was close enough to leave alone, but the 70 mm tube was heavy enough that I needed to remove a large amount of material. Rather than just start cutting and risk trimming too much, I calculated how much height 3.499 grams of 70 mm tubing actually was.

$$v = \frac{m}{d}$$

$$v = \frac{3.499}{2.23} = 1.5213cm^3$$

$$h_1 = \frac{1.5213}{\pi 3.5^2 - \pi 3.277^2} = .36cm$$

.36 cm = 3.6 mm. To ensure I did not accidentally remove too much, I only removed 2 mm of the 3.6 mm that I needed to take out. Removal of material was done with a lapping wheel as opposed to a saw because 2 mm is less than the thickness of the blade, or a belt sander which is often too aggressive (Photo 5).

Photo 5

From here, the assembly finally began, and was really simple and very basic glassblowing. The disc was held in a graphite vacuum chuck and was then sealed to the tubing before being placed in a preheated oven for annealing (Photo 6).

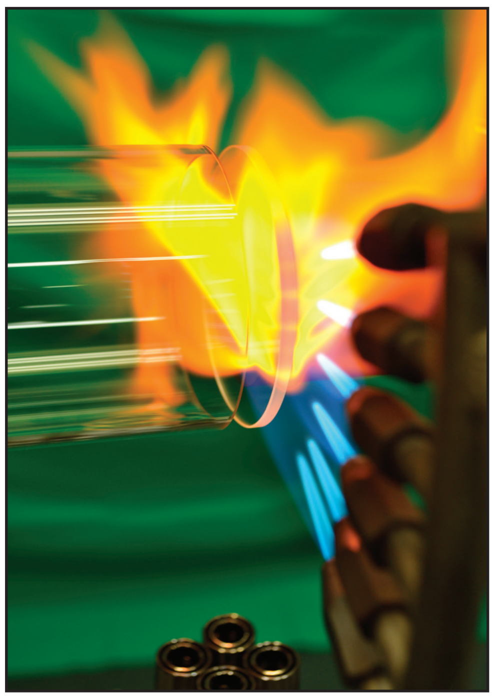

Photo 6

Once annealed, the units were inspected, the tops were given a slight grind to remove a few edge imperfections, cleaned and reweighed. This reweighing gave me a final baseline for the final fine tuning of the units:

5.6 cm tube 189.4203 grams
6.5 cm tube 188.0253 grams
7.0 cm tube 189.8055 grams

The 6.5 cm tube was selected as complete, and 188.0253 grams became the target weight for the three units (Photo 7).

Photo 7

Fine tuning of the remining two units required simply grinding the units for a bit, first on a belt sander, then a lapping wheel, and finally using fine diamond pads. After a small amount of material was removed, I washed the units in soapy water, rinsed them in deionized water, dried them with a lint free cloth and then blew them out with nitrogen. Only after all of these steps was I able to reweigh the unit (Photos 8, 9 & 10).

Photo 8

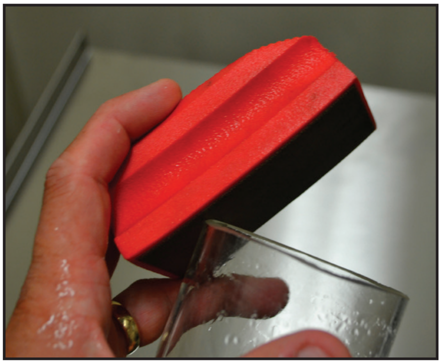

Photo 9

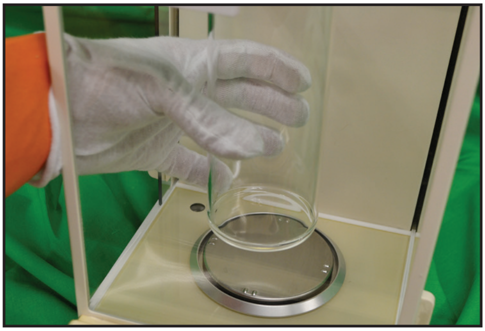

Photo 10

I very quickly discovered that once I got to the drying step, I needed to wear inspection gloves, because my numbers kept jumping around at the bottom end of the register, in that 5th digit of my balance. After a little asking around, and a bit of time on the Googles, I discovered that a human fingerprint has an average mass of 50 micrograms,[2] a full 10% of my tolerance. By simply adding inspection gloves, I was able to eliminate these

[2] Cedric Neumann, et al., "Quantifying the Weight of Fingerprint Evidence Through the Spatial Relationship, Directions and Types of Minutiae Observed on Finger Marks," *Forensic Science International,* Volume 248 (March 2015): 154-171.

variables. This process was incredibly time consuming: it can take up to two minutes for a five-place balance to "settle down" to a consistent reading, the washing drying steps take time, and even the grinding. This part of the process alone took almost a full eight-hour day. I was able to finally not only meet but exceed the tolerances requested, with the final variance only being 0.0003 grams:

5.6 cm tube 188.0250 grams
6.5 cm tube 188.0253 grams
7.0 cm tube 188.0253 grams

As with all our work, the more we repeat a process, the better we get at them. After making four different sets (which thankfully do not have to match across sets, only within a designated set), I now routinely get the mass of the three cylinders to match to within 0.5 grams after the initial saw cut. This increased accuracy was made possible largely to a new tool my facility machine shop manufactured for me: my shop built me a new saw stop for my diamond saw that had an optical positioning stage mounted to the face. I can now set the stop close, and then, using the optical positioning stage, dial it so the distance from the stop to the blade is extremely precise (Photos 11, 12 & 13),

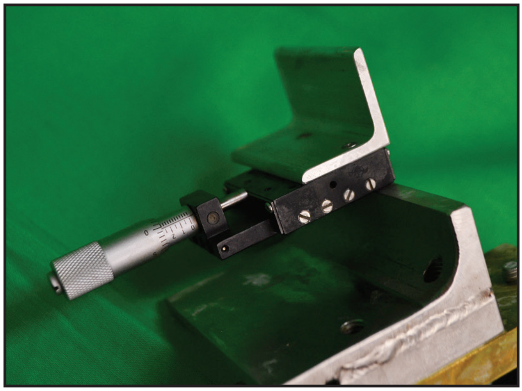

Photo 11

Photo 12

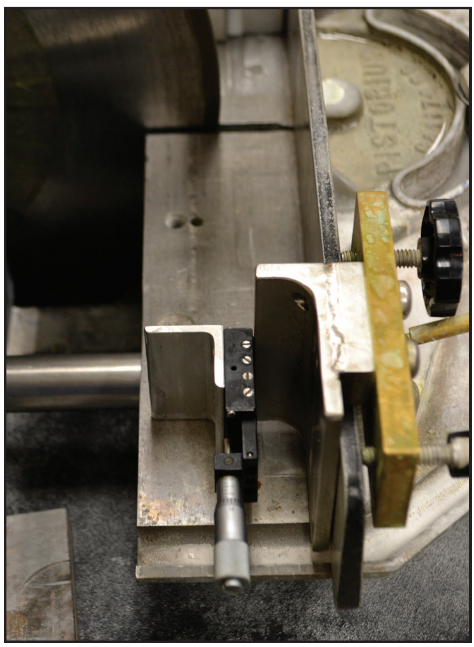

Photo 13

All in all, this project, while incredibly simple in terms of actual glassblowing execution, proved very tricky in its planning and setup.

Twisting on the Lathe: A Simple Technique for Consistent Twists on Any Glass Lathe

by
Elijah J. Aller*

ABSTRACT

A simple method for making twists in tubing or rod is presented. This method leads to consistent and repeatable results.

INTRODUCTION

This paper will present a simple method, suitable for any glass lathe, for making twists in tubing or rod, with readily available materials. Consistent and repeatable results can be achieved with a little practice. Once the twist punty has been created, it will last forever. Two tubes (one that fits inside the other with 1 mm of space in between) (Image 1), some Teflon® thread sealing tape, and a silicone stopper that fits into the outer tube are required.

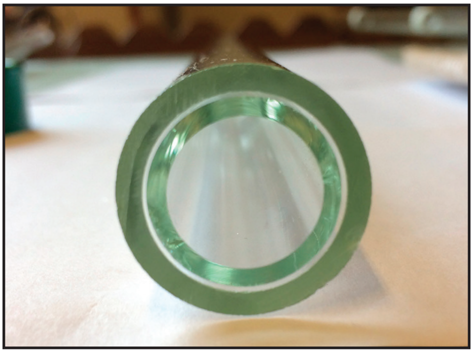

Image 1

METHOD

To begin, cut both tubes to the same length; we suggest using a saw to obtain nice clean ends. Flare one end of the outer tube just a little where the silicone stopper will fit (Image 2). Try to match the angle of the flare to that of the stopper. Cork can also be used if silicone is not available.

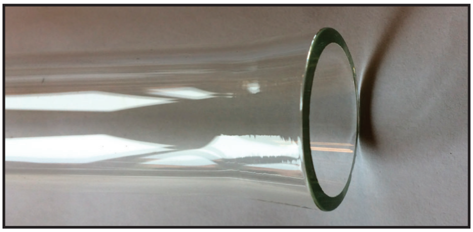

Image 2

On the inside tube, two sets of indentations will need to be made (Image 3). First, the inside tube will need to be straightened (Image 4). To achieve this, set one end of the

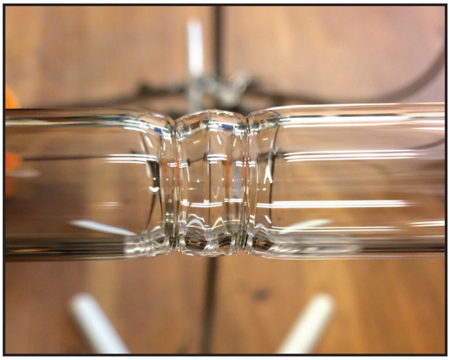

Image 3

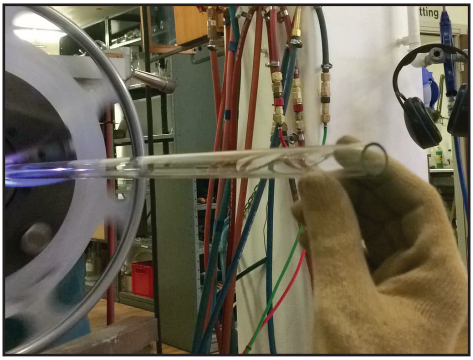

Image 4

*Department of Chemistry, University of Oslo, Oslo, Norway. Email: elijahjaller@gmail.com.

tube in the headstock leaving the other end free. Apply heat with a bushy flame near the chuck jaws, and hold the other end with your fingers. A Kevlar glove will reduce friction between your fingers and the glass. Heat until the glass softens a little bit, and center the free end. Keep holding it centered until the glass cools. Attach a punty rod to the end of the centered tube. The two sets of indentations should be spaced approximately one foot apart. The space between indentations should match the width of the Teflon® tape being used, with the edges of the tape reaching into the indentations; this prevents catching while the tube is being moved in and out (Image 5). Use the corner of your graphite paddle to create small grooves in the tubing. If the bubble between the groves is smaller in diameter than the tubing o.d., blow it back out until it is the same size. Wrap the tape around the bubble between the indentations until it just barely fits into the outer tube. Do not make it too tight (Image 6). Repeat for the other indentation.

Image 5

For the stopper, use a cork boring tool that is the same size as the inner tube. Cut the center of the stopper out leaving a narrow taper on the outside and a straight wall on the inside (Images 7 & 8). This will be used to fix the tubes together for

Image 6

Image 7

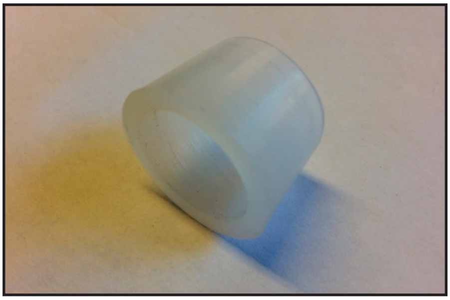

Image 8

pushing and pulling using the tailstock (Images 9 & 10). If you do not have a cork boring tool and silicone stopper, a piece of packing tape can be used to fix the tubes together after the twisting is finished.

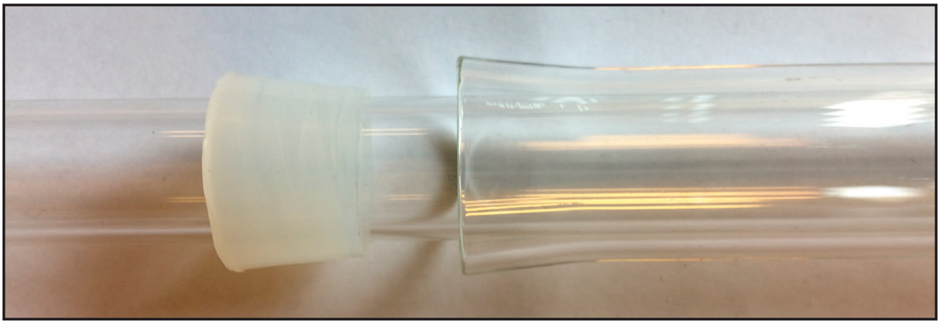

Image 9

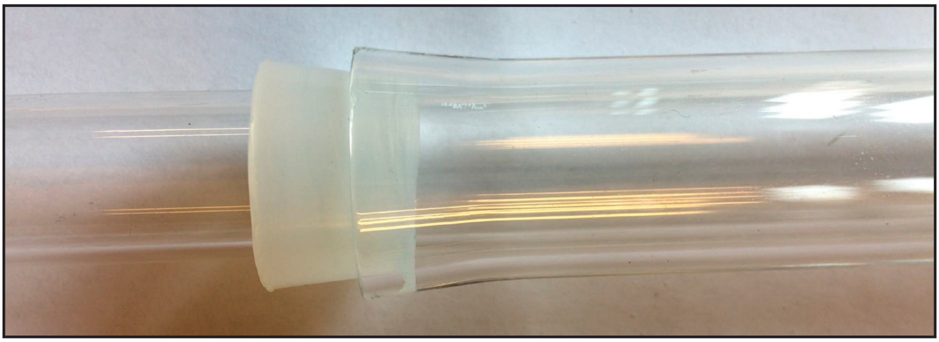

Image 10

Now, close one end of the inside tube and add a short (6" to 8") section of rod (Image 11). The author prefers to use 12 to 15 mm rod. Larger rod may crack due to strain build up (Image 12). Now you are ready to twist!

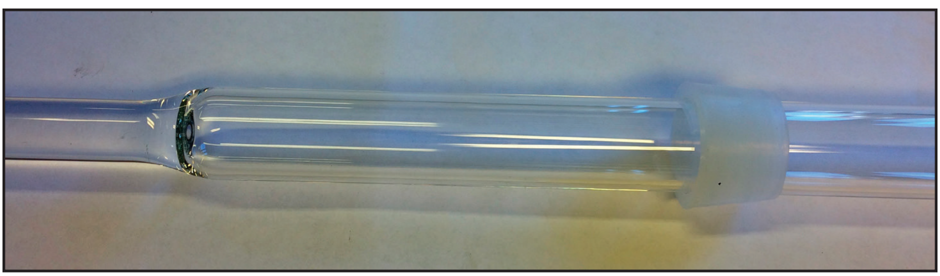

Image 11

Image 12

Image 13

Use the twist punty as you would any punty; it is not used for blowing. Place the twist punty into the tailstock in the locked position. Attach it to the tube you will be twisting. Allow the connection to cool. Move the tailstock away from the tube a little to put the punty into the open position. Be careful that the Teflon® tape does not come into contact with any flame source as it could burn and create fluorine gas which is toxic!

CONCLUSION

This simple method for making twists in tubing or rod is suitable for any glass lathe with readily available materials. Consistent and repeatable results can be achieved with a little practice. Once the twist punty has been created, it will last forever (Image 13).

BIO

Elijah Aller has been flame working for 25 years. He started with soft glass when he was 18 years old, then began working with borosilicate at age 20. He has been working as the scientific glassblower for the University of Oslo for the last four years.

2017 Technical Posters

Steven M. Anderson

"Scleral Lenses for Treatment of Dry Eye Syndrome, Etc."

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Division of Engineering and Cardiology Research
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"An Alternative Way of Assembling Brass Fittings"

Precision Glassblowing of Colorado
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Tracy O. Drier and Phillip J. Calabretta

"Scientific Glassblowing: A Look Through the Glassblower's Didymium Glasses"

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Bryson Dejong

"Straight Talk on Bent Liebig Condensers"

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Tracy O. Drier

"Anatomy of Kaliapparat"

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Tracy O. Drier and Dongki Lee

"Photoelectrochemical Water-splitting Cell"

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Tracy O. Drier and Erich Moraine
"Glassworking Tools of Japan"
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W260 N7751 Jay Lane, Sussex, WI 53089
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Doni Hatz
"Sealing Small Ampoules"
The Procter & Gamble Company
8700 Mason Montgomery Road
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James R. Hodgson
"Petri Dish Pitfalls – Fabrication Challenges of a Two-choice Pitfall Bioassay"
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Richard J. Ponton
"The Easiest Glass Shop Job Ever…With a Twist"
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Anthony Remeika
"Construction of a Borosilicate Water Bottle"
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Benjamin Revis
"Making a Recess for Powdered X-ray Diffraction Insert"
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2017 Technical Demonstrations

Patrick Bennett – *University of Minnesota*
Accurately Cutting Angled Filters and Lenses

Joseph Gregar – *Argonne National Laboratories*
Construction Techniques for an Interesting Refluxing Condenser

Hideaki Hashimoto – *Tokyo Seisakushiyo Co., Ltd.*
Crack-off Machine

Jack Korfhage – *Bayou Glassblowing*
Flanged Buchner Funnel

Tatsushi Iguchi – *Nakamura Rika Co., Ltd.*
Joints Made Japanese West Side Style

Kyle Meyer – *University of Georgia*
Sleeving Uranium Tubing

Tsuyoshi Nakamura – *Nakamura Rika, Inc.*
Borosilicate Glass Box

Lori Neu – *University of Pittsburgh*
Dead Glass: How to Identify it, How to Get Rid of it, How to Prevent it

Bob Singer – *Technical Glass Products*
Fabricating Small Frits

Carrie Sohayda & Deni Kornbluh – *High Volume Oxygen, LLC.*
Oxygen Generators: Best Use Practices; Torch Demonstrations

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Blake Bortolin
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Katrina Daenzer
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