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Custom Stir Shaft for 50 L Reactor

By
Kiva Ford*

ABSTRACT

The fabrication of a large custom borosilicate glass stir shaft for a 50 L Reactor.

This paper is about the fabrication of a large custom borosilicate glass stir shaft for a 50 L Chemglass Reaction system. There are many challenges when creating a large stir shaft, and this paper will address and solve those challenges.

I was asked to create an all-glass stir shaft for this pictured Chemglass reaction system (Photo 1). The chemists were having problems with particles being caught in the Teflon blades on the stir shaft that was originally provided with this reaction system (Photo 2). Every time the chemists would run a reaction, they would have to take the

Photo 1. *Chemglass Reaction System*

Photo 2. *Original stir shaft with Teflon blades*

entire reaction system apart to clean out the Teflon blades on the stir shaft (Photo 3). This turned out to be a time-wasting and tedious process. These reactors are difficult to take apart and are easy to break while doing so. Their solution was to have an all-glass stir shaft as it does not have the nooks and crannies of the Teflon stir shaft. The smooth edges of the glass make it difficult for particles to accumulate on the shaft. Using an all-glass stir shaft would eliminate the need to take the reac-

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tion system apart to clean the Teflon blades. Instead a cleaning solution would be run through the system in between reactions. This would save time and the possibility of breakage occurring while taking the reaction system apart.

Photo 3. *Close-up of the Teflon Blade*

The most difficult part of the fabrication process was cutting and assembling the glass fins onto the main body of the stir shaft (Photo 4). The smaller fins on the stir shaft were cut from ¼" plate glass and the larger fins were cut from ½" plate glass (Photo 5).

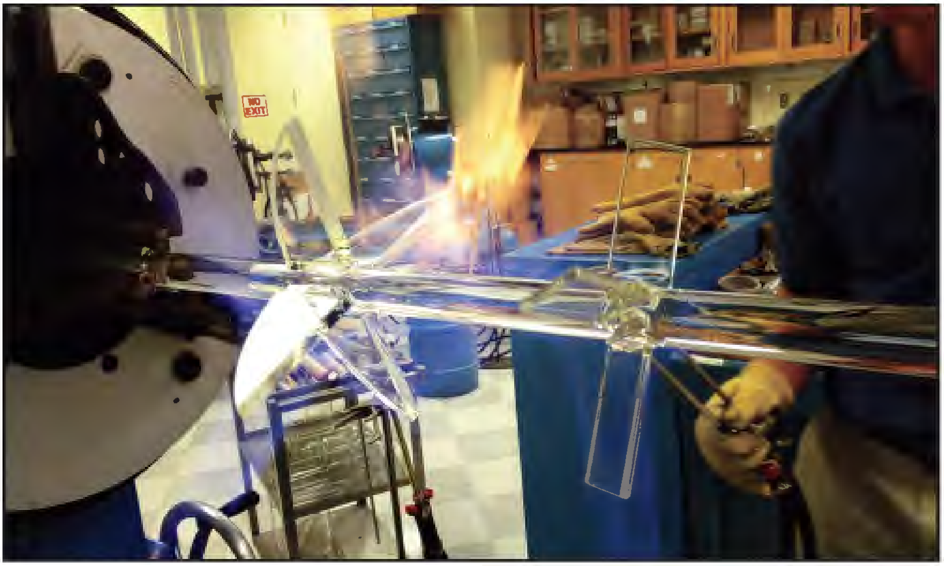

Photo 4. *Glass stir shaft mid-fabrication*

Photo 5. *Glass fins to be attached to stir shaft*

The dimensions of the completed shaft were 47" in length and 10" at the widest point (Photo 6). The top of the stir shaft was made using 1" precision ground and polished borosilicate glass rod. The part of the shaft to which the fins were attached was made using standard 1 ¼" rod. In order to seal the fins to the main body of the stir shaft, I created a holder to help with the sealing process (Photo 7.)

Once the stir shaft was finished and turning in the lathe, I had to adjust

Photo 6. *Me holding a completed stir shaft*

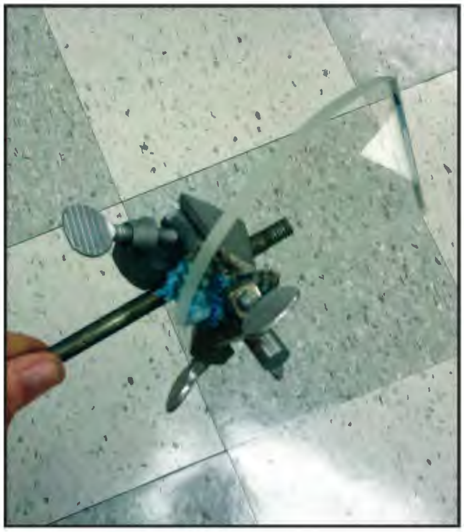

Photo 7. *Holder for holding glass fins*

my oven to the proper annealing cycle. I used the annealing cycle from *Contemporary Lampworking*[1] (Photo 8). It was important to follow the annealing cycle for 1 ¼" glass to ensure that the stir shaft was stress free.

I was worried that the shaft would bend in the oven during the annealing process so I came up with a support system using balls of aluminum foil to support the shaft while it was being annealed (Photo 9, *see page 6*). By loosely balling up the aluminum foil, I was able to firmly press the stir shaft into the foil. This created a cheap and effective method of ensuring that the shaft would stay perfectly straight during the annealing process.

I ended up making eight of these shafts, and they have been running smoothly for about a year. The chemists were very pleased with the final results.

Table 1

Annealing rates and temperatures for borosilicates, including Corning Pyrex™ 7740, Kimax KG33, Schott Duran, Northstar, Glass Alchemy.

COE = 32 x 10⁻⁷, Annealing Temp. = 1050°F

d	R_{fh}	Annealing T	t_{soak}	R_{sc}	Endpoint	R_{fc}
(inches)	*F°/min.*	°F	*min.*	*F°/min.*	°F	*F°/min.*
0.1	2687.50	1090	15	468.75	910	1812.50
0.2	671.87	1070	15	117.19	910	453.12
0.3	298.61	1070	15	52.08	910	201.39
0.4	167.97	1070	15	29.30	910	113.28
0.5	107.50	1050	15	18.75	910	72.50
0.6	74.65	1050	15	13.02	910	50.35
0.7	54.85	1050	15.	9.57	910	36.99
0.8	41.99	1050	15	7.32	910	28.32
0.9	33.18	1050	15	5.79	910	22.38
1	26.88	1050	15	4.69	910	18.12
1.25	17.20	1030	23	3.00	910	11.60
1.5	11.94	1030	34	2.08	910	8.06
1.75	8.78	1030	46	1.53	910	5.92
2	6.72	1030	60	1.17	910	4.53
2.25	5.31	1030	76	0.93	910	3.58
2.5	4.30	1030	94	0.75	910	2.90
2.75	3.55	1030	113	0.62	910	2.40
3	2.99	1030	135	0.52	910	2.01
4	1.68	1030	240	0.29	910	1.13
5	1.08	1030	375	0.19	910	0.7

Table 2

Annealing rates and temperatures for Effetre/Moretti soda-lime glass.

COE = 104 x 10⁻⁷, Annealing Temp. = 968°F

d	R_{fh}	Annealing T	t_{soak}	R_{sc}	Endpoint	R_{fc}
(inches)	*F°/min.*	°F	*min.*	*F°/min.*	°F	*F°/min.*
0.1	826.92	1008	15	144.23	830	557.69
0.2	206.73	988	15	36.06	830	139.42
0.3	91.88	988	15	16.03	830	61.97
0.4	51.68	988	15	9.01	830	34.86
0.5	33.08	968	15	5.77	830	22.31
0.6	22.97	968	15	4.01	830	15.49
0.7	16.88	968	15	2.94	830	11.38
0.8	12.92	968	15	2.25	830	8.71
0.9	10.21	968	15	1.78	830	6.89
1	8.27	968	15	1.44	830	5.58
1.25	5.29	948	23	0.92	830	3.57
1.5	3.68	948	34	0.64	830	2.48
1.75	2.70	948	46	0.47	830	1.82
2	2.07	948	60	0.36	830	1.39
2.25	1.63	948	76	0.28	830	1.10
2.5	1.32	948	94	0.23	830	0.89
2.75	1.09	948	113	0.19	830	0.74
3	0.92	948	135	0.16	830	0.62
4	0.52	948	240	0.09	830	0.35
5	0.33	948	375	0.06	830	0.22

Photo 8. *Chart of annealing cycles from "Contemporary Lampworking Volume 1"*

[1] Bandhu S Dunham. *Contemporary Lampworking*, Volume 1 (Arizona: Salusa Glassworks, Inc., 2002.) 226.

Photo 9. *Stir shaft pressed into aluminum foil*

Impossible Seal: Fused Quartz to Tungsten Seals Without the Use of Grading Glass

by

Jeffrey R. Anderson, Sr.*

ABSTRACT

Creation of hermetic glass-to-metal seals in fused quartz assemblies provides a series of unique challenges inherent to the special properties of fused quartz. The inclusion of a high purity tungsten wire into the cell design has a tendency to magnify these difficulties even more. When the application of graded sealing glass is restricted or eliminated, the creation of a proper hermetic seal between tungsten and quartz becomes one of the most difficult seals that a scientific glassblower can face. This paper will discuss a method used at the National Institute of Standards and Technology for the creation of these seals.

INTRODUCTION

Work in the glass shop at the National Institute of Standards and Technology provides one with ample opportunities to practice and experiment on unique apparatus designs with requirements that would rarely be encountered in the production environment. One such project involved the production of a fused quartz instrument with high purity tungsten wires hermetically sealed through quartz tubing.

The project involved the use of fused quartz tubing to create the cell body. Smaller tubes were used to form the inlet, and two tubulations were used for anode and cathode seal tubes. High purity tungsten wire leads were welded to the cathode then fed through the quartz tubes. Although the tungsten and quartz have a difference in their thermal coefficients of expansion, the use of graded sealing glasses was restricted due to project requirements. The seal could not be anything but tungsten and quartz.

QUARTZ PREPARATION

The first major preparation involved the cleaning of the fused quartz tubing. This was accomplished through the use of a series of six washing stations. At the first station the quartz was submerged in a bath of methanol for one hour. This alcohol bath was then followed by a bath of acetone for one hour. After proceeding through the alcohol and acetone baths, the tubing was quickly run through aqua regia to remove any remaining impurities. A series of three distilled water baths removed any cleaning chemicals that remained and the tubes were dried overnight in a 400°C oven.

TUNGSTEN PREPARATION

The tungsten used was of very high purity (99.999%). Through early testing, it was determined that low purity metals increased the chance of seal failure and the decision was made to pay the extra expense for high purity materials to increase our success rate.

The tungsten used was treated in a hydrogen rich reducing atmosphere. The tungsten was treated (reduction) for thirty minutes at 1000 degrees centigrade. After treating, the tung-

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sten was stored in a glove box that was purged with filtered air. As an extra precaution, a side chamber was used that was purged with high purity argon. Best results were obtained by using the tungsten within one day of annealing to prevent oxidation from causing seal failure. Even when using high purity argon, the metal should be used as soon as possible.

SEAL CONSTRUCTION

The setup process to create the seal is crucial. Any mistakes during the setup will lead directly to a failed seal. It is imperative that the seal be supported on both sides of the quartz tube to help relieve any stresses that would otherwise compromise the seal. When threading the wires into the tubing, a mixed forming gas composed of 5% high purity hydrogen and 95% high purity nitrogen must be used to avoid any contamination of the wires or quartz. Tests with commercially available forming gasses proved to work well. The entire assembly must be purged with the forming gas for one hour after threading the wires. The purge gas is turned off just before the seal is made so the seal can then be created using flame velocity only.

INSPECTION

Once the seal has cooled, an inspection under a microscope will provide an accurate predictor of seal integrity. If cracking is visible under a microscope soon after cooling, then the chances of a failed seal are high. Tungsten and glass colors after sealing were also a good predictor of seal quality: the tungsten should retain its bright coloring after sealing and the quartz should remain clear and allow easy viewing of the metals. If this is not the case, then a contaminant has been introduced to the seal and a failure is imminent.

CONCLUSION

Although they possess a large differential in thermal coefficients of expansion, it is possible to create a hermetic seal between fused quartz tubing and thin (0.0127 mm) tungsten wire without the need for any graded sealing glasses. Through the work on this project, it was demonstrated that with proper setup and following a strict regimen of cleaning and contamination control, these seals can be created with a high degree of success.

In the pictures below, the first (Photo 1) shows a seal with no oxide, good seal. The second (Photo 2) shows oxidation that caused the cracking.

Photo 1. *Good seal*

Photo 2. *Bad seal*

Large-Area Picosecond Photo-Detectors Project Techniques of Construction

by

Joseph S. Gregar[a] & Robert Wagner[b]

ABSTRACT

This paper contains the construction techniques and complex fabrication of a new photo-cathode detector that is many times larger than any currently commercially available. Many scientific glassblowing techniques were utilized, and some non-glassblowing bonding techniques are shared. This is an all-glass photo-detector, almost 9" square by only 0.497" thick, that has the potential to become the standard in many different applications including in the medical field.

Experimental high energy physicists are developing large-area photo-detectors to measure the time-of-arrival of particles with 1 pico-second time resolution and spatial resolution of millimeters. These photo-detectors can also be used for Positron-Emission Tomography (PET), a medical screen. The new technology is a factor of 120 times better than the present state-of-the-art. This involves a number of intellectually challenging areas: demonstration of a working large-area micro channel plate photo detector, three-dimensional modeling of photo-optical devices, the design and construction of ultra-fast (200 GHz) electronics, the 'end-to-end' (i.e. complete) simulation of large systems, real-time image processing and reconstruction, and the optimization of large detector and analysis systems for medical imaging.[1]

This presentation will exhibit and explain the complicated construction techniques that were required to assemble a photo-detector made completely of glass that measures almost 9" square by only 0.497" thick. Many new glass techniques had to be learned and most were achieved by studying the numerous failures encountered.

A suggestion was made early on to construct these new prototype photo-detectors using Schott Borofloat B-33 plate glass. The interesting challenge was to figure out the best sealing or bonding techniques to use. The main issue is size because, at 8-5/8" x 8-5/8" x ½" thick, these detectors are quite large. The bottom plate (referred to as the anode plate) and the top plate (aka the cathode plate) are 3.7 mm thick, prone to cracking, and their flatness cannot be compromised. The best option for repeatable success was to "frit" the plates onto the glass frame. After much trial and error, the proper sealing temperature was determined at 470ºC which is lower than the 565ºC annealing temperature. Furthermore, each frame needed a glass tube attached onto the side of the frame for future vacuum processing. All standard glassblowing operations needed to be completed be-

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[1] "Large-Area Picosecond Photo-Detectors Project," Henry Frisch Ph.D. (October 10, 2009). Accessed November 5, 2009. <http://psec.uchicago.edu/>

fore the fritting operation. I will now present the procedures required to construct a complete photo-detector frame (Photo 1).

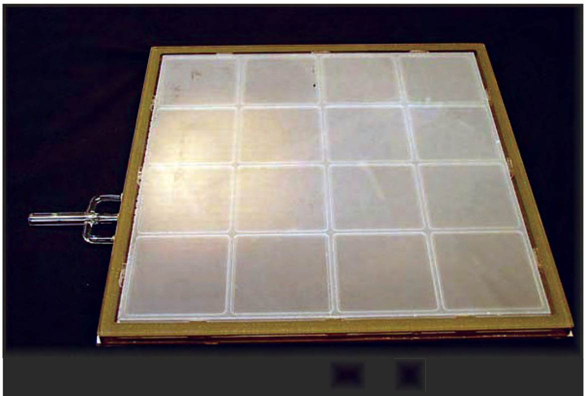

Photo 1

THE FRAMES

The first step is to produce a blank frame of B-33 Borofloat glass. The optimal wall thickness is .200” thick. It was determined that the best and most economical way to achieve this was to have the frame cut out of a large plate using the water-jet cutting process. The water-jet process uses an abrasive compound that is forced through a tiny orifice using extremely high-pressure water and air. These machines are generally computer controlled and can hold reasonable tolerances. It was imperative to find companies that had experience cutting glass plate using this technique. They had to use a very fine grain garnet for their abrasive and be willing to work with tolerances of +/- 0.002”. It took us several tries to determine which companies would produce a quality product to our specifications (Photos 2 & 3).

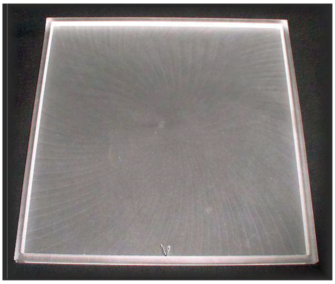

Photo 2. *Cut frame: “Water jet” cut from large piece of 6.4 mm thick Schott Borofloat plate. The abraded surface finish is determined by the abrasive grain size.*

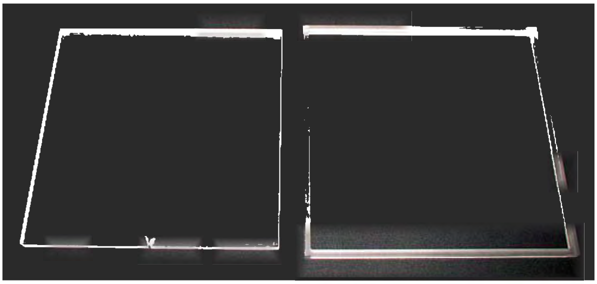

Photo 3. *Cut frame: Notice where the starting point is on the dropout piece on left.*

EVACUATION PORT

The frame is prepared by drilling a 3 mm diameter hole in the sidewall of the frame. The first frames were only 6.4 mm thick so it was necessary to seal a short section of 5 mm o.d. standard wall tubing onto ¼” medium wall tubing. The 5 mm o.d. tube is then spliced onto the hole in the frame’s sidewall. This will be the vacuum pump-out site for future evacuation. Great care is necessary to perform the glassblowing operations on the frame without producing cracks or causing distortion. The frame must be warmed slowly and kept hot during the sealing operation. It is imperative for the future fritting operations that the faces of the B-33 frame not have any distortion. Delicate glassblowing techniques were required using a very small, sharp flame. Occasionally I would use a “Tiny Torch” for this but I had better success using a Hoke Jewelers hand torch with a #1 tip. At first I would drill the holes completely through the sidewall. It was always a challenge to blow inside the tube to make a good smooth seal and I would stick the point of a tapered graphite rod inside the bottom of the hole. I later discovered that if I did not drill the hole completely through, I could easily blow inside the tube and make a perfectly smooth seal (Photos 4a & 4b).

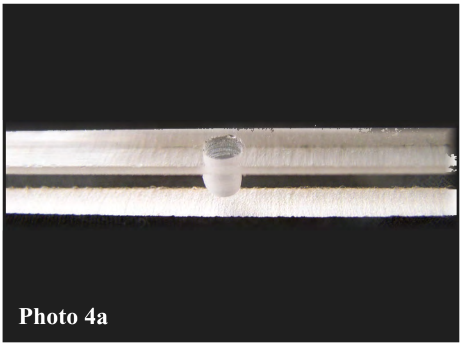

Photo 4a

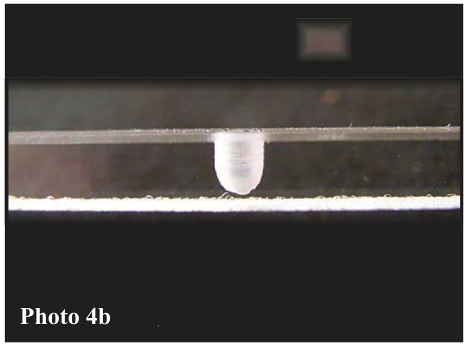

Photo 4b

After the seal was complete, I simply used the tiny flame and heated from the bottom and carefully blew out the hole. At this point it is necessary to flame anneal the entire area that has been worked taking care to not overheat and have the frame sag or distort. After a few of these tubes broke off, I added two 4 mm support rods to the assembly. This helped considerably in the side-to-side forces, although the tubes were still slightly vulnerable in the up and down directions (Photo 5).

Photo 5. *Tabulated frame with bracing.*

The newest generation frames are now made from 9 mm thick Schott B-33 Borofloat glass plate. This allows one to drill a 1/8" diameter hole and seal a 1/4" o.d. medium wall tubing directly onto the frame. This gives us a larger evacuation hole and less chance of distorting the top and bottom surfaces of the frame during the flame sealing operation.

GETTER SPACERS/HOLDERS

The "internals" (as we call them) consist of three 8" square glass grid spacers and two multi-channel plates, (MCP's). The inside dimension of the frame is nominally 8.250" square. To support and center the "internals" I add twelve spacers (three on each side) to the inside of the frame walls. These spacers will also support a flat strip of getter material to absorb any moisture that may develop inside the completed photo-detector during operation. When completed, these large photo-detectors will be hermetically sealed and heated in an oven to out-gas using a high vacuum pumping system. The getter will be a long continuous strip around the inside perimeter of the frame and needs to be supported. I decided to use custom made rectangular tubing made to the specifications for height and thickness, which worked well. The dilemma was how to securely attach them to the inside walls of the frame. My initial technique was to use a very small flame and melt the inside edge of the tubing and stick it to the frame walls using a sharp graphite pointer. This technique worked fine; it was, however, very time consuming. The frame and spacer locations needed careful pre-heating and post flame annealing. I later discovered that using thinned out silver ink fired at 400ºC worked perfectly and silver would not contaminate the internal atmosphere (Photo 6).

Photo 6. *Getter spacers*

Photo 7. *Multiple frames getter spacers*

This process requires four separate oven firings but I prepare multiple frames and fire them together (Photo 7).

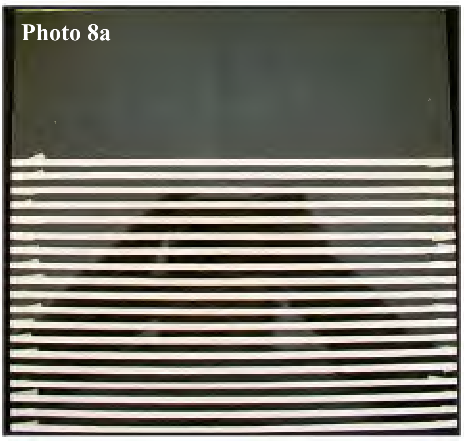

Photo 8a

ANODE PLATES

The next hurdle was the production of a conductive silver grid pattern on the anode plate. I originally masked off strips with 1/8” wide automotive pin striping tape. The tape was positioned so that there were forty clear glass stripes between the tapes. I painted these clear glass areas with conductive silver ink and let them air-dry overnight (Photos 8a, 8b & 8c).

The tapes were removed and the striped plates were fired in the oven at 565ºC. This

Photo 8b

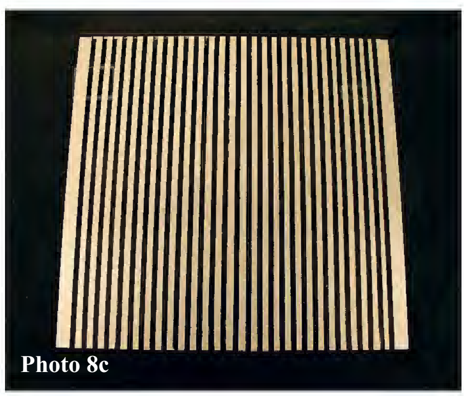

Photo 8c

step proved time consuming so I looked for a vendor that could produce silver ink decals.

These decals were silk-screened, wetted, and slid onto the anode plates. I gently squeegeed them with a flat sheet of neoprene rubber about the size of a business card to remove excess water and trapped air bubbles. They were allowed to fully air-dry overnight and then fired in the oven at 565ºC. The decals worked well: today we have the silver grid pattern silk screened directly onto the anode plate and fired. This has proved to be more economical and a big time saver. The silvered strip lines will carry electrical current into the photocathode cell to power the multi-channel plates and the getter strips (Photo 9).

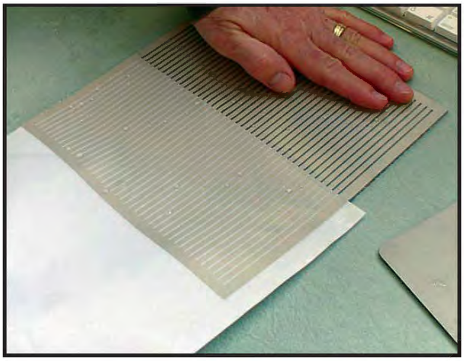

Photo 9. *Decal transfer*

THE FRITTING PROCESS

The striped anode plate then needed to be joined to the tubulated frame. A glass fritting technique was suggested but I had little knowledge of the process. I remembered seeing this technique while on a tour during an American Scientific Glassblower Society's Southeastern Section meeting. This was a very difficult process to master. With some research and several attempts, I slowly succeeded in bonding glass-to-glass parts. I had little or no success bonding glass to plates with silver strips. Some of my early fritting failures are shown here (Photos 10a, 10b, 10c, 10d, & 10e).

Photo 10a

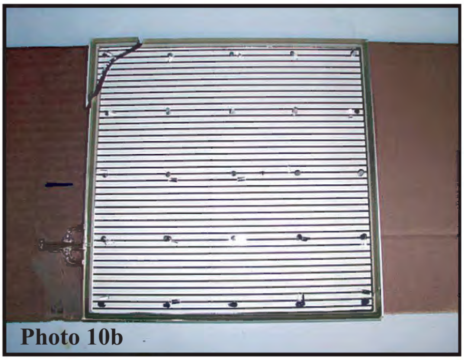

Photo 10b

Photo 10c

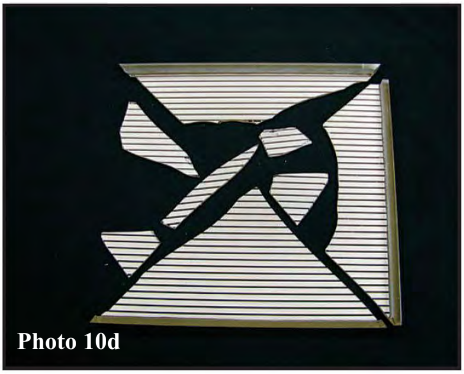

Photo 10d

Since the frame is 8-5/8" square and the anode plate needed to remain extremely flat without distortions, flame-sealing was not successful. Instead, because all the glass components are Schott B-33 Borofloat, I used Schott #G018-223 K3 frit paste. The Schott frit was thinned slightly using Schott Binder #Lab17692. The frit material was drizzled onto one surface of the frame using a small stainless steel lab spatula. The frit was applied carefully so it would not flow down the sides of the frame. The frame was then placed in the oven for three different firing cycles. I found that immediately placing it in the oven after applying the frit produced the best results (Photo 11).

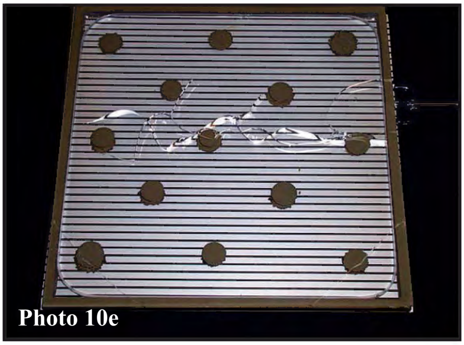

Photo 10e

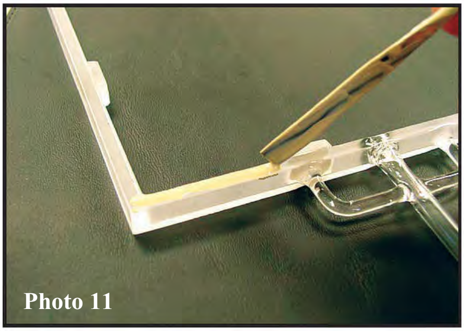

Photo 11

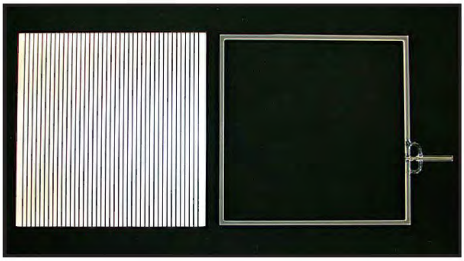

Photo 12. *Anode plate and frame*

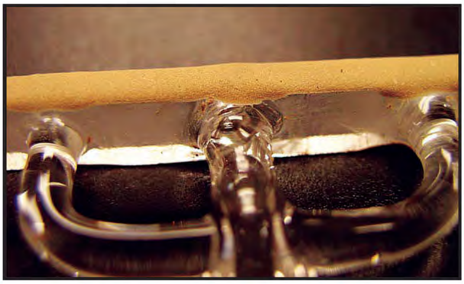

Photo 13. *Out-gassed frit*

It took many attempts to achieve a solid hermetic seal that did not crack. Remember the frit has to fill in the voids or depressions between the silver strips and we are trying to bond to both glass and silver. After many, many trials of adjusting the firing parameters, I attribute the key to the frit bond success to a much longer and slower out-gas cycle than the manufacturer's recommended recipe (Photo 12).

The first heating is called the out-gas cycle. This requires raising the oven temperature very slowly, at a rate of 1 degree per minute until 370ºC is reached. The second step is to hold the temperature at 370ºC for 90 minutes. You will notice after this cycle that the texture or surface of the frit appears to be very dry and porous (Photo 13).

Photo 14. *Glazed ftit*

The third heating step is the glaze cycle. The temperature is raised 3.3°C per minute to 470ºC. The fourth step is to hold at 470ºC temperature for 60 minutes. The surface of the frit is now hard and shiny (Photo 14).

These four heating steps can be programmed into the oven controller and run sequentially. The frame may be taken out of the oven when it has cooled enough to touch.

The next step is to frit or bond the anode plate onto the prepared frame. A glass holding fixture was constructed to aid in the alignment of the frame and anode plate. Small glass plate sections and tubes were cut and bonded onto a ½" thick Schott Borofloat plate using the silver ink bonding technique described earlier. The small plates set the location of the frame, and the tubes were used as stops for locating the anode plate correctly over the frame. (Photo 15).

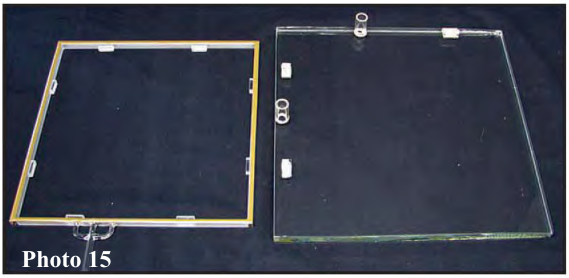

Photo 15

Tubular ½" quartz rings inside the frame are cut .006" taller than the height of the frame wall. These rings act as spacers to regulate the finished height of the fritted anode plate. This also helps the fritting material from squeezing out and becoming too thin (Photo 16).

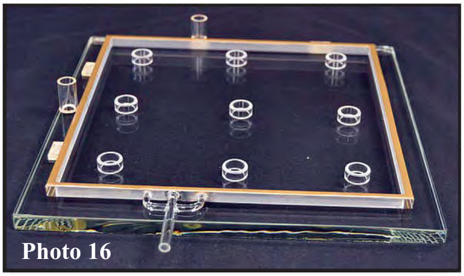

Photo 16

The anode plate is positioned on top of the frame with the silver grid pattern face down (Photo 17).

Photo 17

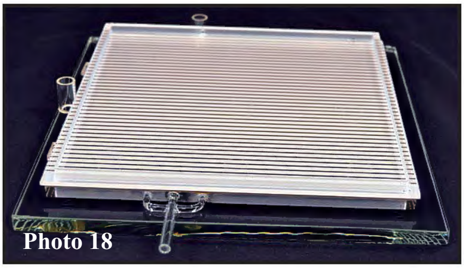

Photo 18

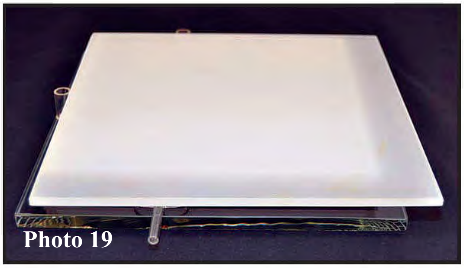

Photo 19

A second "bare" frame is positioned on top of the anode plate to force the pressure downward directly onto the fritted frame walls (Photo 18).

A quartz plate is placed on top of the second frame to support weights that will be added. The quartz plate is 9" x 9" x ¼" thick so it overlaps the frame and will not sag with the weights on it during the heating cycle (Photo 19).

Add 6,350 gram (16 pounds) of stainless steel weight on top of the quartz plate. This is to compress the frit when it reaches its fusing temperature of 470ºC (Photo 20).

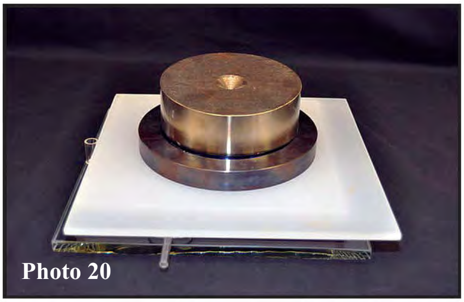

Photo 20

Here is a side view of the frame assembly in the oven. Note all the different layers (Photo 21).

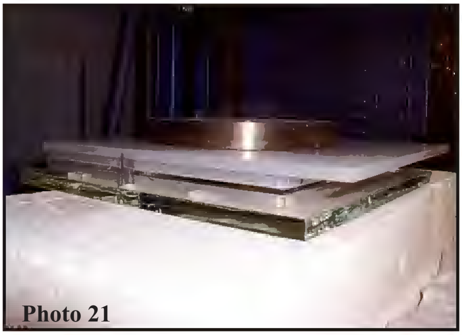
Photo 21

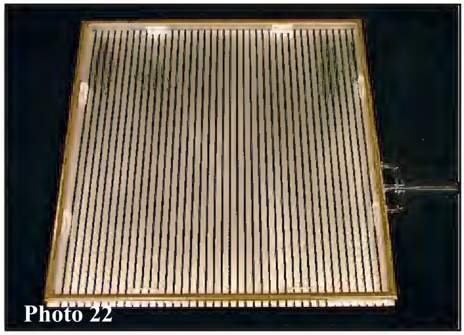
Photo 22

Completed lower anode plate seal (Photo 22). Note: The top surface of this frame has been prepared with frit to seal the cathode plate on later; observe the getter holder spacers bonded to the inner surfaces of the frame.

THE INTERNAL STACK

The internal stack of the photodetector includes three glass grid spacers and two multi-channel plates (MCP's).

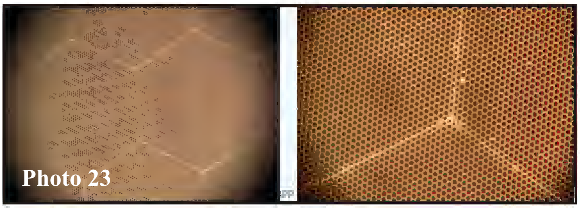
Photo 23

A multi-channel plate is a borosilicate glass plate with "tens of thousands" of porous spaces or channels produced using a technique similar to that of fiber optic manufacturing (Photo 23).

Here is a photo of a full size 8" x 8" multi-channel plate (Photo 24).


Photo 24

The grid spacers have been water jet cut from a solid piece of Schott B-33 Borofloat plate (this process was discussed before). They range in thickness from 1 mm, 1.2 mm, 1.5 mm, 1.8 mm and 2 mm. The thicknesses of the grid spacers are chosen to give the internal stack the correct height inside the frame (Photo 25).

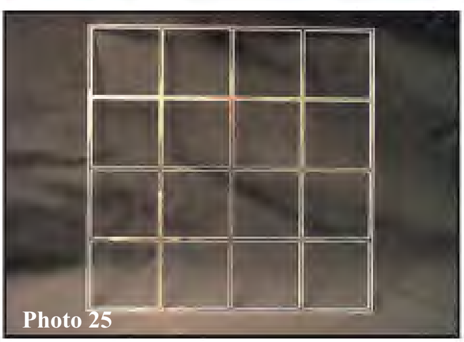
Photo 25


Photo 26

I made two styles of prototype grid spacers before the water jet process was adopted. One was the "grid" form and one was termed the "modified spiral." Although these looked nice, they did not have the flatness that was required (Photos 26 & 27).

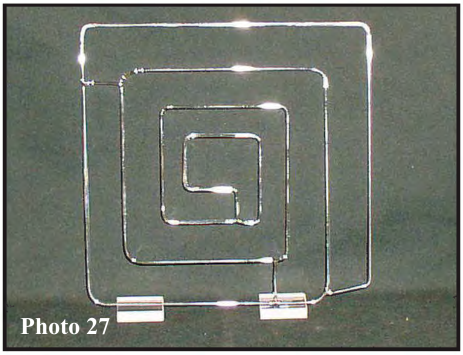
Photo 27

INSTALLING THE INTERNAL STACK

Place grid spacer #1 into the clean empty frame (Photo 28).

Install the first Multi-Channel plate (Photo 29).

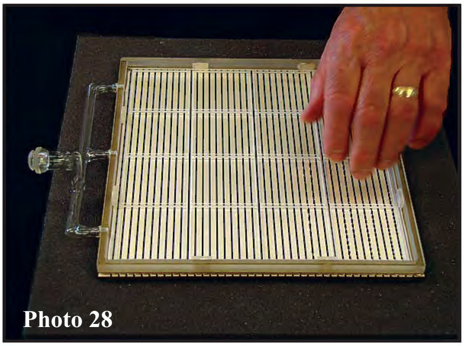
Photo 28

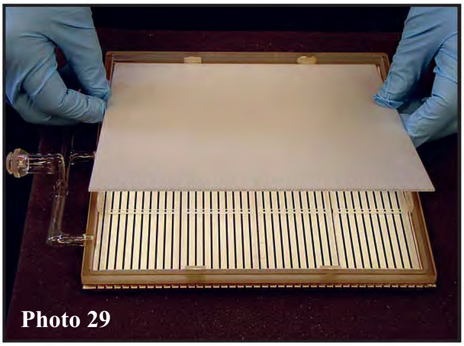
Photo 29

Install the second grid spacer and the second MCP plate (Photos 30a & 30b).

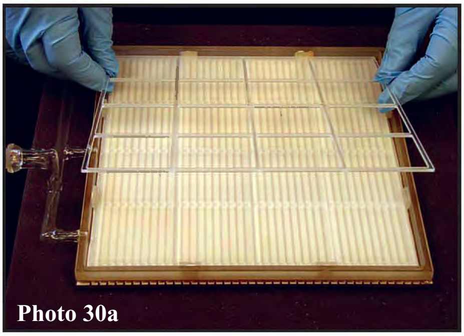
Photo 30a

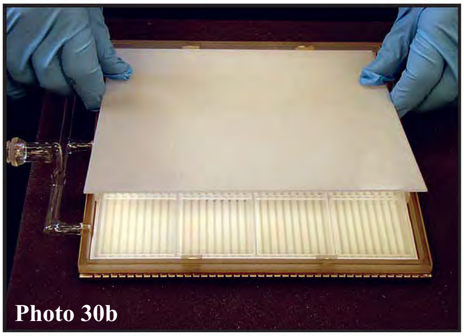
Photo 30b


Photo 31

At this point the space must be precisely measured to determine the thickness of the third and final grid spacer. The top surface of the grid spacer must be .006" above the top surface of the frame. This .006" height extension is important so the top cathode will be properly supported to avoid implosion when the atmosphere is removed from inside the cell by means of a high vacuum pumping system (Photo 31).

Here is a photo of the complete stack of internals positioned inside the frame (Photo 32).

COMPLETED PHOTO CATHODE ASSEMBLY

For prototype testing, a cathode plate with an aluminum-coated inner surface is fritted on top to make a vacuum tight hermetic seal. All the components of the "inner stack" have an Atomic Layer Deposition (ALD) coating for electrical resistivity and insulation properties (Photo 33).

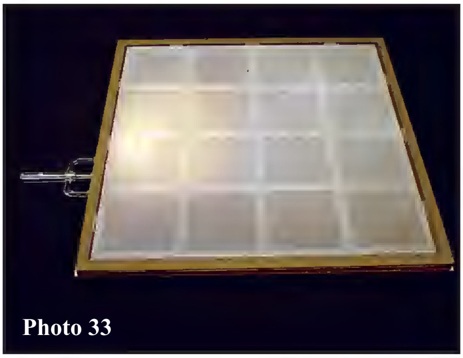

Photo 33

Photo 32

During the preparation of this paper a major frame design change occurred. A triple vacuum port design has been adopted that allows for faster pumping speeds and is less vulnerable to breakage. However, it means more complicated glassblowing is required. With this style, three drilled holes are necessary. Again the holes are not completely drilled through the sidewall. A 10 mm diameter manifold is fabricated and sealed onto the three vacuum port tubes (Photos 34a & 34b).

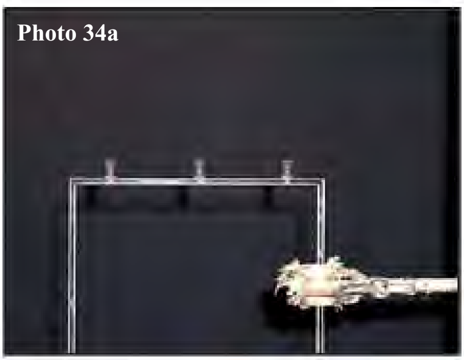

Photo 34a

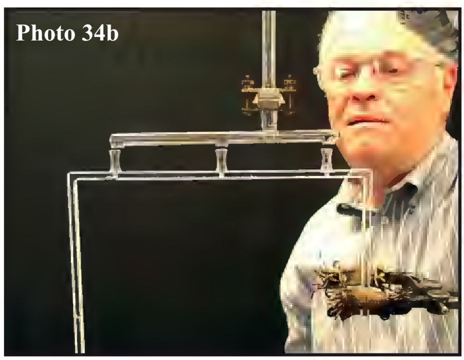

Photo 34b

Photo 35a

Photo 35b

Extra care is needed here to seal the three tube locations without cracking. You must keep all three locations on the frame and the manifold hot at all times during this sealing operation. Careful post flame annealing is required to remove the strain from the sealing operation without warping or distorting the frame and its surfaces (Photo 35a & 35b).

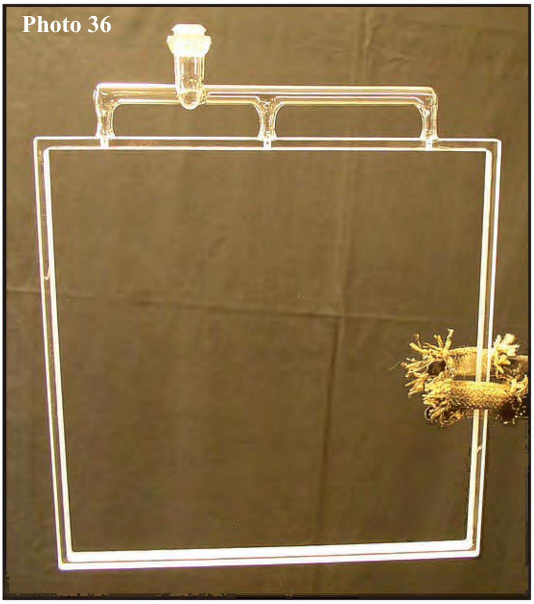
Photo 36

The completed addition of the 3-port vacuum manifold (Photo 36).

In conclusion, this has been a very interesting project to be a part of for the last two years. I learned many new techniques and tricks that I hope are conveyed clearly. Although many of the techniques did not involve flame sealing glassblowing, it was beneficial to the project to have a scientific glassblower involved. Many times we do not realize how much glass information we have accumulated and are able to share.

ACKNOWLEDGMENTS

I would like to acknowledge project leader Henry Frisch, Ph.D., University of Chicago, Robert Wagner, Physicist, Argonne National Laboratory, and Dean Walters, Mechanical Engineer, Argonne National Laboratory.

I would also like to thank Jeff Babbitt, Scientific Glassblower, University of Kentucky and Wib Mateyka for consultation on the glass fritting techniques.

The Safe Use of Propane Fuel in the Glass Shop

by
William DeFlorio[a]

ABSTRACT

Propane can be both a productive and destructive chemical. Although it has its industrial uses, it can be very dangerous when it is not handled with care. Keeping a properly maintained work environment and taking care when burning propane is very important for the safety of workers.

Propane is a commonly used fuel with both consumer and industrial applications. Often it is used to grill up steaks in the back yard; but if it can power your grill, why not your torch? Many glassblowers use methane or hydrogen to heat their material; propane, however, can prove to be a more economical fuel because of the lack of infrastructure like city gas lines. But, since it is a different gas than methane or hydrogen, it comes with a different set of safety considerations. For example, the fuel must be stored differently, as a pressurized liquid in a cylinder rather than as a gas, and different safety detectors must be installed in the shop. Different grades of equipment must also be used for propane instead of the equipment used to run on methane.

In terms of pricing, the decision between propane and methane comes down to infrastructure. If the shop in question has a fuel line from the city which supplies it with natural gas, methane is often the better choice. In an article on the *Glassline* website hotglass.com, Henry Grimmet, an American Scientific Glassblowers Society member from Glass Alchemy, Ltd., puts the price of piped-in natural gas at \$0.07 per 10,000 British Thermal Units.[1] He also states that the heat output of natural gas is around 1050 BTU's per cubic foot of gas.

Bottled natural gas, on the other hand, can be much more expensive. Bottled methane is rarely utilized as a fuel for torches that would be used in glassblowing or even welding. It is therefore a specialty gas. The only real industrial use for bottled methane is in plasma cutting. As a result, methane stored in a cylinder, at \$5.00/10,000 BTU/ft^3, is much more expensive than methane from a pipe.

Propane, on the other hand, cannot be piped in from the city as easily. It is much more commonly bottled. Bottled propane is often used by welders and even more frequently by grilling enthusiasts. Consequently, the price of a propane cylinder is kept relatively low. At \$0.15/10,000 BTU and 436,000 BTU/20# cylinder, bottled propane is 30 times cheaper than bottled natural gas by BTU.

To somebody trying to set up a shop in a location without a natural gas line, bottled fuel is a necessity. And for somebody trying to run a business, the cost of fuels is a critical consideration, especially when the price difference is so great. So when a city line is not provided, propane becomes the only viable option.

But how much heat are you getting out of these fuels? Is the price difference accompanied by a difference in performance? To look deeper into the matter, there are a few different ways to measure the potential of a fuel. Ways to evaluate a fuel's potential are

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[1] Grimmett, Henry. "Propane or Methane! What is your studio?" *Glass Line* 17.6 (2004). Accessed June 17, 2011. <http://www.hotglass.com/access/v17n6/Propane.html>.

the flash point and the fire point.

The flash point is defined as the lowest temperature at which a volatile substance will form an ignitable gaseous mixture with air. For propane, this is at -104°C and methane at -188°C.[2] So both gases will ignite at a much lower temperature than will ever be recorded in a shop under atmospheric pressure. The fire point, however, is a more relevant measure. The fire point of a gas is usually about 10°C higher than the flash point. This is the temperature at which the volatile solution will burn and sustain combustion for five seconds when ignited with an open flame. Looking at these measures of propane and methane, they are both very low temperatures and the difference between them is relatively small; this likely accounts for the small difference between the heat of the two different flames.

The temperatures of propane and natural gas flames are also not very different. With propane burning at 2820°C and natural gas at 2770°C the difference is negligible when assuming that the two flames will travel at the same speed.[3] But the difference here too is negligible: propane, burning at 12.2ft/s, is actually slightly slower than the slow burning methane at 15.2ft/s. As a reference point for these numbers, acetylene burns at 25ft/s and hydrogen at 36ft/s.[4]

So looking at these numbers, one might say that there is not a huge difference between the amount of heat that can be obtained from a propane torch and from a methane torch. This is not the case, however, because it is the chemical potentials of the two gases that must be considered. They are two different molecules and therefore will burn differently on a chemical level. The differing arrangement of atoms in the gases leads to a wildly different array of behaviors by the two substances. The diagrams below illustrate this change in structure. Figure 1 shows a propane molecule whereas Figure 2 shows a methane molecule.

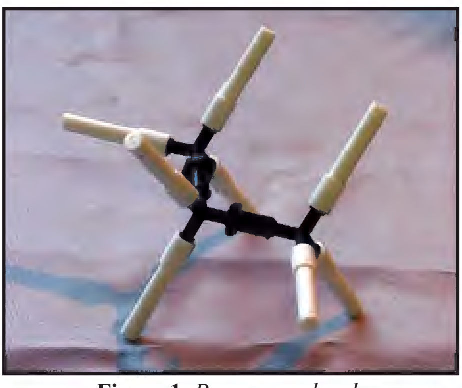

Figure 1. *Propane molecule*

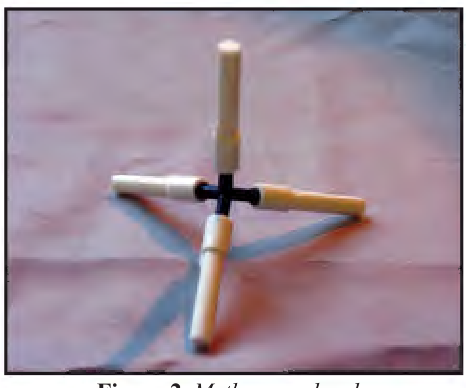

Figure 2. *Methane molecule*

As can be seen in these Figures, propane is a three carbon molecule held together with single bonds and saturated with hydrogen atoms. Methane has only a single carbon and four hydrogen atoms.

[2] "Properties of Fuels (a)." Alternative Fuels & Advanced Vehicle Data Center. US Department of Energy, n.d. Accessed August 3, 2011. <http://www.afdc.energy.gov/afdc/pdfs/fueltable.pdf >.

[3] "Flame Temperatures some Common Gases."Engineering Toolbox. N.p., n.d. Accessed August 3, 2011. <http://www.engineeringtoolbox.com/flame-temperatures-gases-d_422.html>

[4] Andrew D. Althouse, Carl H. Turnquist, and William A. Bowditch, *Modern Welding: Complete Coverage of the Welding Field in One Easy-To-Use Volume* 4th ed. (South Hollan, Illinois: The Goodhear-Willcox Co., Inc., 1980) 715.

Since a carbon to carbon single bond and a carbon to hydrogen single bond have comparable amounts of energy stored within them, it becomes apparent that each propane molecule has a much higher potential for the release of usable energy when broken down in a process such as combustion. Because of the unique nature of these structures, each molecule can be expressed in a more convenient short-hand. Propane can be represented by C_3H_8 and methane as CH_4 . The commonly accepted measurement for this amount of energy, the usable energy released from the breaking of chemical bonds, is called the heat of combustion. This is abbreviated as ΔH_{comb} and is a measure of the heat absorbed into a system as kilojoules per mole of substance, or enthalpy change, specific to the designated combustion reaction. A mole of a substance is a set number of molecules of that substance. It is approximately 6.02×10^{23} molecules. The ΔH_{comb} measure is in essence the extra energy left in the surroundings after the bonds of the reactant molecules have been broken and the bonds of the product molecules have been formed. So ΔH_{comb} is the sum of all the bonds in either propane or methane and oxygen minus the sum of the bonds in the resulting carbon dioxide and water vapor. These bond energies can be looked up in any chemistry textbook or chemical reference manual. Also note that ΔH_{comb} is a measure of heat being absorbed into the system during reaction, so a negative value corresponds to heat being released from the system into the surroundings. This is the useful energy which will do work such as heating glass.

To look at the numbers, ΔH_{comb} CH_4 = -802kJ/mol. Propane has more than double this output per mole at ΔH_{comb} C_3H_8 = -2044kJ/mol.[5] To put it into standard units, propane releases approximately 91,690 BTU's for every US gallon of volume. Looking at these numbers, it should come as no surprise that a propane flame can deliver much more heat than a methane flame. The vast difference in cost between the bottled forms of the two gasses is accompanied by a similarly vast difference in heat output.

Moreover, one other piece of thermo-chemical data must be noted. As a serious safety concern, the flammability limits of each gas must be considered. The flammability limit, or explosive limit, of a gas is the percentage of gas in air by volume at which the mixture will ignite. This number is based upon the concept of stoichiometry which is the idea that for the maximum efficiency of each chemical reaction, the reactants need to be present in a set ratio of moles. The closer the reactants are to this ratio in a combustion reaction, the more heat that is released. So, in order to get the maximum amount of heat out of a fuel, it must be burned with oxygen in a stoichiometric mix. If the fuel is too far out of proportion with oxygen, it will not burn at all. This is where flammability limits come from.

Converting from moles back into percent by volume, the flammability limits can be more useful. Methane will burn at 5-15% by volume in air. Propane, on the other hand, will ignite as low as 2.37% and will continue to burn only up to 9.5%.[6] Thus even though methane will burn in a much larger window of concentrations, propane only needs to be half as concentrated to burn in air. Accordingly, it will ignite easier, and it will take much longer to burn out as the concentration will only drop low enough for the flame to burn itself out at half of the concentration which would be required to do this by natural gas.

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[5] ΔH_{comb} CH_4 and ΔH_{comb} C_3H_8 calculated from standardized data obtained from *CRC Handbook of Chemistry and Physics*, 89th edition, Ed. David R. Lide (Cleveland, OH: CRC Press, 2008 - 2009).

[6] "Properties of Fuels (a)," Alternative Fuels & Advanced Vehicle Data Center. US Department of Energy, n.d. Accessed August 3, 2011. <http://www.afdc.energy.gov/afdc/pdfs/fueltable.pdf >.

The chemical structure of propane does not, however, only affect how much work can be done with the torch. In addition, the differing structure of propane brings on a new set of safety concerns and procedures. As a representative of the local propane distributor Metro Welding in Waltham, MA stated in a phone conversation, “propane is a whole different animal.”

Due to the fact that it is a large, heavy molecule, propane will sink in a mixture with air. Unlike methane or hydrogen, which will vent off on its own and diffuse through the air, propane will pool in the low spots. This is often just the floor, but propane will also collect in cracks, drains, and even top-loading ovens, building in concentration until it reaches that 2.37% flammability limit. Also, propane is odorless, like natural gas, and is treated with an odiferous chemical called ethyl mercaptan. This chemical can be smelled at concentrations as low as one fifth of the lower flammability limits of propane, so the human nose will likely be able to detect a propane leak much sooner than the leak is able to ignite. According to the Propane Education and Research Council, the minimum concentration for human detection of ethyl mercaptan by nose is one part of the additive in 2.8 billion parts of air.[7]

Processed propane, however, can undergo what is called odor loss which makes it impossible for the human nose to detect a leak. This can happen when a leak is underground. By the time that the propane has reached the surface, the smell will be neutralized by the earth. Odor loss can also occur when ethyl mercaptan is exposed to bleach or rust: bleach will oxidize the ethyl mercaptan into ethyl sulfonic acid, and similarly ferric-ferrous oxide, or rust, will oxidize the ethyl mercaptan to form diethyl disulfide.[8] These chemicals cannot be easily smelled; it is important therefore to not allow air or moisture to get into a propane tank. Along with taking away the odor, rust can structurally weaken the bottle as well.

Photo 1. *Properly placed propane detector*

In order to detect potentially dangerous leaks, it is important to have a propane detector installed in the shop and that it be properly installed. As was mentioned earlier, a propane leak will pool down by the ground. This makes the positioning of a propane detector different from that of a methane or hydrogen detector.

A properly placed detector is shown in Photo 1. The unit is located on the wall about one foot above the floor and also above the drain so that if a pool of propane forms, the gas will be detected before the pool can accumulate too high. If the propane detector were to be placed like that of a hydrogen

[7] Propane Education and Research Council. Accessed June 17, 2011. <http://www.propanecouncil.org/>.

[8] “Odourant Fade - Propane and Natural Gas,” Sintra Engineering, 07/03/2010. August 3, 2011. <http://www.resolvematters.ca/pages.php?pid=5&sid=1&ref=31>.

or natural gas detector on the ceiling of the shop, the whole room would have to fill up with propane before the detector would be set off. This would be too late. So in order to be warned of a leak in time, users should place their detectors in low spaces where pools are likely to start forming.

Another piece of equipment which is good for early detection is a good read-out interface for the detector. The meters shown on the interface pictured in Photo 2 display the concentrations of the gases being detected in the shop. It can be helpful to see the needle rising on the meter before the alarm is activated. The alarm itself is loud and irritating by design. If the leak can be detected simply by reading the needle's position on the dial and corrected early on before the alarm goes off, then this is a better situation.

Photo 2. *Leak detector read out*

In the event of a leak, the facility should be ventilated with positive pressure. A fan should be placed so that it is blowing air into the room rather than pulling air out. If the fan is pulling air out of the room, it pulls the propane and air mixture right past its motor; sparks from the motor can then ignite the fuel-air mixture and the leak, which could have been safely dealt with, swiftly turns into an explosion.

To avoid a leak, the use of proper equipment is essential. This equipment should also be kept in tiptop shape. The best place to start when looking at propane equipment is the source, or the tank. The tank, also referred to as a cylinder or bottle, is where the propane is stored. Unlike other higher pressure cylinders, the propane tank is welded. For example, an oxygen tank, which has an internal pressure of approximately 2250 pounds per square inch, is pressed out of one solid piece of metal; the surface is smooth. An LP tank however, does not have a smooth surface: it is generally made in several different pieces and then welded together. This is because it has a lower pressure, about 140psi, and does not require the strength of the single stamped piece of steel. It is much cheaper and easier to weld the tanks, so there is no reason to go through the extra process of stamping it out like an oxygen tank.

	20# Steel	20# Alum	33# Steel	33# Alum	43# Steel	43# Alum
Capacity (gallons)	4.7gal	4.7 gal	7.9 gal	7.9 gal	10.3 gal	10.3 gal
Weight (empty)	26.5 lbs	19.5 lbs	35 lbs	23 lbs	40 lbs	27 lbs
Weight (full)	46 lbs	40 lbs	69 lbs	56 lbs	83.5	70.5 lbs
Overall Height	19.5 inches	20.5 inches	27 inches	28.5 inches	33 inches	34 inches
Diameter	12.5 inches	12.5 inches	12.5 inches	12.5 inches	12.5 inches	12.5 inches

Figure 3. *Common tank sizes*

The chart shown in Figure 3 lists the various sizes of propane tanks you are likely to come across and their capacities.

Photo 3 shows a propane tank labeled with its notable exterior features.

Photo 3. *Tank with notable exterior features*

The propane tank has a rounded bottom to provide structural integrity: the sharp angle created by a flat bottom tank, would not be strong enough. Consequently, a ring is welded onto the bottom of the tank so that it can sit flat on the floor without rolling around. This ring also provides a spacer in between the ground and the surface of the tank, keeping the bottom of the tank away from water or other corrosive materials which may be spilled on the floor. The collar welded to the top of the tank provides users a safe way to lift and secure the tank rather than by the valve. It also, similar to the cap on an oxygen or natural gas bottle, helps to protect the service valve, the main outlet from the tank, in the event of a collision. As the Smith Equipment online safety training warns, this is vitally important, because if the cylinder's valve is sheared off, the tank quickly transforms into a torpedo. On a side note, oxygen tanks should never be lifted by their caps as the cap is only threaded onto the tank and is not meant to hold the weight of the entire cylinder. Also, any tank, no matter the gas, should always be properly secured, usually by tank straps, and oxygen tanks should be capped when not in use. Arc welders must never be struck on the surface of tanks as this may weaken or even rupture the pressurized tank. Tanks with visible structural damage should be inspected before further use. Damage to the outside of a tank could lead to a leak or rupture.[9] According to www.propane101.com tanks must be inspected before continued use after 12 years from the date of manufacture and every five years after that. Also, no type of cylinder should be transported in an enclosed vehicle. If a leak develops inside the vehicle, it will not be able to ventilate and a dangerous buildup of gas will occur.

According to the National Fire Protection Association, propane tanks larger than 1lb should not be stored inside. The Propane Education and Research Council, on the other hand, takes a more conservative standpoint and advises that propane tanks should never be stored inside any enclosed area whatsoever; instead they should be stored outside on some sort of secure footing, like a concrete pad.[10] Often, brick or cinderblock walls are built around such outside tanks. This is to prevent motor vehicles from colliding with the tanks or even piping coming off of the tanks.

One consideration for exterior tanks is temperature control. The tanks should not exceed 120°C but can also not get too cold in the winter.[11] Since propane is stored as a liquid, its

9 Bruce Buhler, "Technical and Product Training," Smith Online University. Smith Equpiment, n.d. Accessed July 7, 2011. <http://www.smithequipment.com/moodle/course/view.php?id=3>.

10 "Propane Safety," National Fire Protection Association, May 2010. Accessed June 17, 2011. <http://www.nfpa.org/categoryList.asp?categoryID=304&URL=Safety%20Information/For%20consumers/Gasoline%20&%20propane/Propane%20safety&cookie_test=1>.

11 "Propane 101: Promoting Propane Safety…Through Better Understanding." Accessed June 17, 2011. <http://propane101.com/>

vapor pressure will drop with the temperature. When it gets too cold outside, not enough of the liquid will vaporize to create adequate line pressure; the tanks then freeze. This does not literally mean that the propane solidifies, but it will stay in a liquid form. When the propane cannot vaporize, the tank is useless. This also happens when too large a volume of propane is being removed from the tank at a rapid rate. Just like a larger scale model of a wart "freeze off" cylinder, a consumer medicine found at many drugstores which is simply a small container filled with propane, the rapidly moving gas causes a significant drop in temperature. If the rate of gas leaving the tank is high enough, this drop in temperature can be enough to freeze the cylinder.

According to the National Fire Protection Association, some changes have recently been made to propane tanks to make them safer for the consumer. Since 2002, tanks between 4 and 40lbs have been required to be fitted with an Overfill Prevention Device, or OPD. Some industrial tanks are exempt. The OPD is a spring-operated pressure release valve. This valve makes it impossible to fill the tank up to more than 80% of its capacity. Tanks with an OPD will either have a triangle shaped handle or will have the letters "OPD" stamped into their collar.[12] For example, the tank below in Photo 4 has been fitted with an OPD.

Photo 4. *Tank with OPD valve – note triangular handle*

Because of this "OPD" pressure release valve, it is important to fill and store propane tanks upright. This is because when it is in the tank, propane is a liquid. The valve is at the top of the tank. When the tank is upright, the vaporized propane can make contact with the valve and flow as required. If liquid is in contact with the valve, the valve becomes useless.[13]

Another recent modification to propane tanks is that the tanks now need to be hooked up to a regulator for gas to flow out of them. Before this change, a tank could simply be left out with the valve open and it would vent. Now, if the tank is not connected to a regulator, the valve will not vent propane, even if the service valve handle is left in the open position.

Following the flow of propane from the tank to the torch, the next stop is the regulator. Regulators should be kept free of dust or oil: the sudden compression of gas within a regulator can create a momentary elevation of temperature which can be enough to ignite a dirty regulator. This creates what is referred to as "regulator burnout" which can damage the regulator, injure equipment

[12] "Propane Safety." National Fire Protection Association, May 2010. Accessed June 17, 2011. <http://www.nfpa.org/categoryList.asp?categoryID=304&URL=Safety%20Information/For%20consumers/Gasoline%20&%20propane/Propane%20safety&cookie_test=1>.

[13] "Propane 101: Promoting Propane Safety… Through Better Understanding." Accessed June 17, 2011. <http://propane101.com/>

operators, or create a gas leak. Similarly, oxygen regulators should only be used with oxygen tanks. The contamination of oxygen in a regulator used on a fuel bottle or fuel in a regulator used on an oxygen bottle can also result in burnout.

Acetylene, another bottled fuel, has a much lower tank pressure than LP. Because of this, an acetylene regulator can safely be used on an LP tank as long as the application is relatively small. The two are not, however, completely interchangeable: an LP regulator may never be used on an acetylene tank. The LP regulator is designed to operate at a higher pressure and will not be able to adequately control the rate of acetylene being released from the tank. Acetylene is unstable at the higher pressure which would be created, causing the situation to quickly become dangerous.

After the regulator, the propane flows into the hosing. This hosing, as recommended by the Metro Welding representative, should be T-grade hosing, not the R-grade typical of use with other fuels. Propane will corrode R-grade hosing as well as O-rings and gaskets made of this material in torches and pilot lights. The T-grade is resistant to the propane corrosion and is therefore used to create a more safe work environment by yet again helping to avoid leaks.

The propane makes its way through the hosing into the torch to be burned with the oxygen. But sometimes, due to a pressure differential, as the Smith Equipment online safety resources state, the propane and oxygen can mix before they are intended to do so in the system creating an ignitable mixture where there is not supposed to be one. This is called a backfire or a flashback. In the case of a backfire, the torch's flame simply goes out with a loud popping noise. With a flashback, the problem is more severe: there is a sustained combustion where there is not supposed to be one in the system. The flashback can damage equipment and injure the operator. In the event of a flashback, turn off the tank if it is safe to do so and try to get the fire under control. Then call emergency services if necessary. It is handy in emergency situations to have the number of the local fire department written down by the telephone ahead of time. A backfire or flashback can happen with a system running on any fuel gas, however it can be especially damaging with propane, due to propane's potential for destruction.

More important than learning to deal with a flashback is working to prevent one from happening in the first place. As with the vast majority of safety concerns, prevention of a problem is better than cleanup. The first thing to do is to install flashback arrestors on the shop's torches. Some torches come from the manufacturer with an arrestor already installed. If this is the case, it is not necessary to put an additional arrestor on the system. The best place for a flashback arrestor is right between the torch and the hoses. This is because the hose is the most common place for a flashback to occur. The mixture of fuel and oxygen happens within the torch and then often this mixture travels back into the hose before it is ignited. The flashback arrestor contains a check valve to help ensure that gas is only flowing in the correct direction and a sinter filter. The sinter filter takes heat away from the flame of the flashback in order to extinguish the fire before it becomes uncontrollable. Purging the oxygen line before lighting a torch significantly reduces the possibility of having a flashback as this forces out any fuel which may have been contaminating the line. In addition, simply keeping torch tips clean helps to ensure that gas is flowing properly though the system.

To avoid the pressure differential which causes flashbacks, never run the contents of oxygen tanks at pressures lower than 40 psi. In other words: do not run the tanks dry. This low pressure zone can force fuel from the torch back into the oxygen line resulting in a problem.[14]

As a final note of safety, it should be stated that in addition to creating minute amounts of nitrous oxide, a lung irritant, incomplete combustion can also occur with a propane flame. This means that the torch can produce carbon monoxide. Carbon monoxide is odorless and poisonous. It has a greater affinity for the hemoglobin in blood than oxygen and binds to it more readily, rendering human blood unable to transport vital oxygen to the muscles and other organs. Even if there is sufficient oxygen in the atmosphere, a person will suffocate, turning red. Ventilation will help to get rid of any carbon monoxide which forms. A carbon monoxide detector will also help to alert users of the presence of the poisonous gas.

Propane can be a dangerous fuel. According to the National Fire Protection Association, United States fire departments in the year 2003 through 2007 responded to propane-related incidents which resulted in 34 civilian deaths, 135 injuries, and $48 million in direct property damage. In addition, annually, there were 1,170 home structure fires because of propane. It is a dangerous chemical with extreme potential for catastrophe, but when used safely and correctly, it is an incredibly powerful and useful fuel.[15]

[14] Bruce Buhler, "Technical and Product Training," Smith Online University. Smith Equpiment, n.d. Accessed July 7, 2011. <http://www.smithequipment.com/moodle/course/view.php?id=3>.

[15] "Propane Safety," National Fire Protection Association, May 2010. Accessed June 17, 2011. <http://www.nfpa.org/categoryList.asp?categoryID=304&URL=Safety%20Information/For%20consumers/Gasoline%20&%20propane/Propane%20safety&cookie_test=1>.

Solvent Purification Stills: Function and Design

by

James R. Hodgson[a]

ABSTRACT

There are probably as many designs for solvent purification stills as there are scientific glassblowers. This paper will address how they are used, some historical designs, and things to keep in mind when fabricating or designing a still for laboratory use.

INTRODUCTION

Chemists, especially those doing synthesis, often require solvents which are water-free or oxygen-free for satisfactory results. One way of obtaining these "pure" solvents is to combine the solvent with an appropriate drying or deoxygenating agent and then distill the solvent under inert gas for collection and use. The literature contains many appropriate solvent/reagent combinations and procedures for obtaining "pure" solvent. This is frequently done in a solvent purification still (Figure 1).[1]

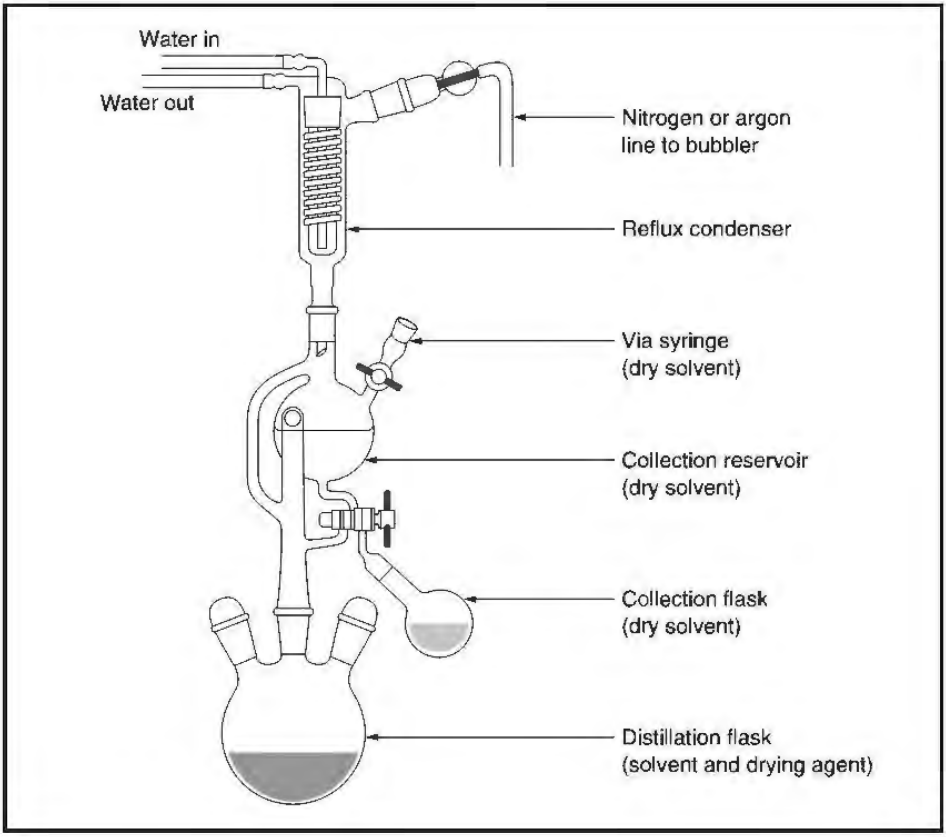

Figure 1. *Solvent distillation setup*

[a] Department of Chemistry, Kansas State University, Manhattan, Kansas 66506. Email: hodgson@ksu.edu.

[1] Modern Organic Synthesis in the Laboratory by Li, Limberakis, & Pflum (2007) Fig.1.1 By permission of Oxford University Press, Inc.

TYPICAL SOLVENT STILL

There are probably as many different designs for solvent purification stills as there are glassblowers. Most of them will contain the following features.

1. A distillation flask to contain the solvent and drying agent
2. A collection reservoir to hold the distilled, dried solvent
3. A condenser to condense the distilled vapor and direct it to the collection reservoir
4. An inert gas inlet to keep the solvent under a blanket of oxygen-free, moisture-free gas
5. A way to take off the dried solvent for use
6. A valve to distribute the solvent to a collection point or return it to the distillation flask.

Here are a few of the many different designs of solvent purification stills. They run the gamut in looks and ease of fabrication (Figures 2-7 and Photos 1-4).

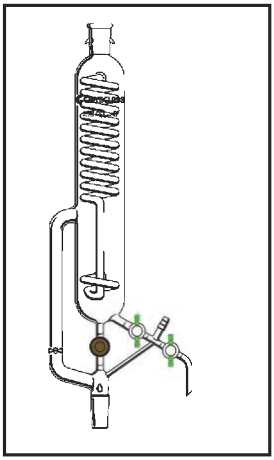

Figure 2. *AF-0740 Solvent Distillation Apparatus, Airfree*[2]

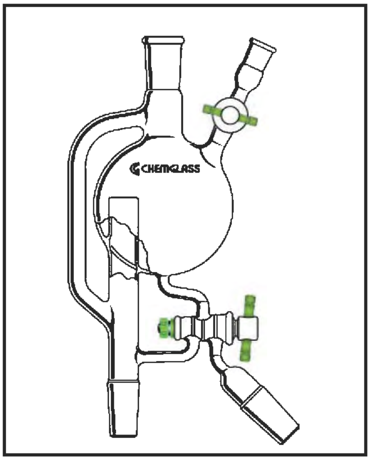

Figure 3. *CG-1233 Distilling head, Solvent, Modified*[2]

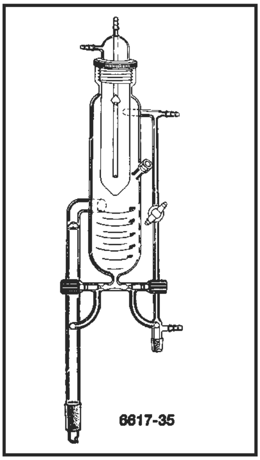

Figure 4. *"6617 Solvent Still*[3]

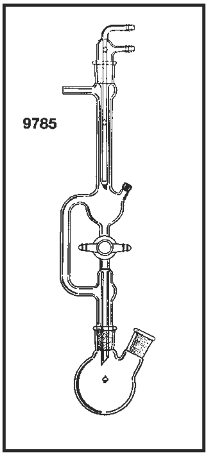

Figure 5. *"9785 Still, Solvent Reflux, Micro*[3]

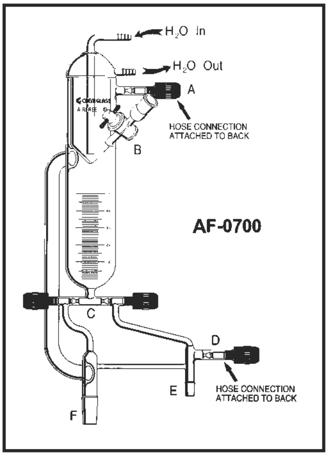

Figure 6. *AF-0700 Solvent Distillation Head, Airfree*[2]

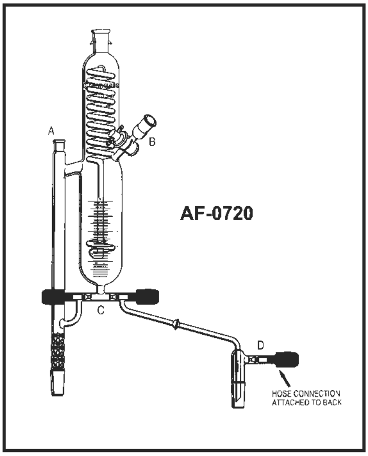

Figure 7. *AF-0720 Solvent Distillation Apparatus, Airfree*[2]

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[2] Used with permission of Chemglass Life Sciences.

[3] Used with permission of Ace Glass, Inc.

Photo 1

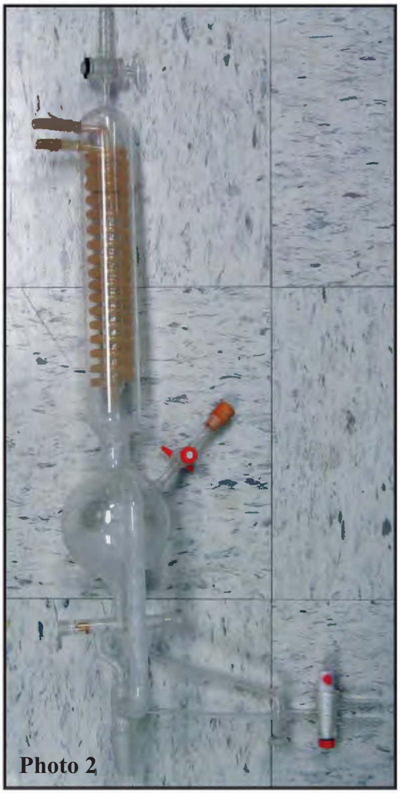

Photo 2

Photo 3

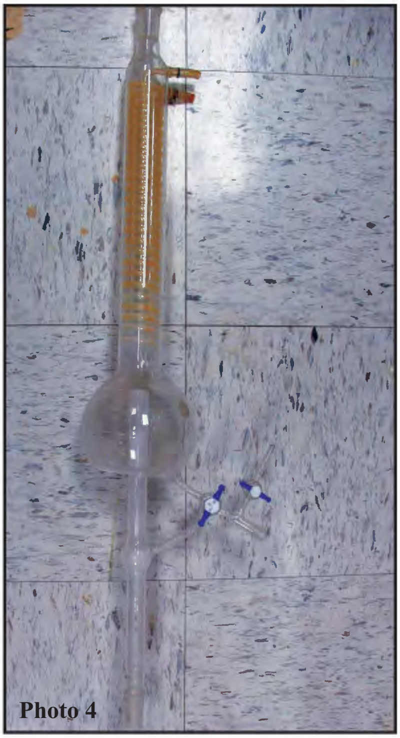

Photo 4

DESIGN CONSIDERATIONS

As scientific glassblowers, we would like our work to function properly, be as easy to use as possible, be fairly robust, be simple to fabricate, and look professional. Here is a design that meets those qualifications (Photo 5). Using the typical features of a solvent purification still, we will examine this design in light of our desires as scientific glassblowers and of the chemists who may be using it.

Photo 5

Photo 6

1. Distillation flask: The distillation flask should be large enough to hold sufficient solvent so that it does not run dry if the reaction is left unattended and it should be appropriately sized to the quantity of solvent normally used. A second neck for the addition of more solvent or to be used when quenching the still is also useful (Photo 6).

2. Collection reservoir: The collection reservoir is usually several times smaller than the distillation flask. If the still is left unattended while solvent is being collected, any excess will return to the distillation flask through the overflow tube, preventing the distillation flask from running dry. In this design, the vapor tube and overflow tube are the same. Ring sealing the tube into the center of the flask reduces the likelihood of it being broken. The slight alembic shape formed after the ring seal serves the dual purpose of making the fabrication easier and also allowing complete drainage of unneeded solvent back to the distillation flask (Photo 7).

Photo 7

3. Condenser: The condenser needs adequate cooling capacity to completely condense the

solvent vapors and direct them into the collection reservoir. I used to favor the Friedrichs condenser because of its compact size. However, given the reactive and flammable nature of some solvent/drying agent combinations and the potentially thin wall of the Friedrichs condenser cold finger, a standard coil reflux condenser might be an equally efficient and safer choice (Photos 8 & 9).

Photo 8

Photo 9

4. Inert gas inlet: Although any standard gas inlet adapter may be used, it would be safest to use one without a valve. It is possible that the valve could be closed and yet it would still appear that inert gas was blanketing the reaction. This would also result in a closed system and possible pressure buildup.

5. Dry solvent collection: Like many solvent stills, this design has two possible ways to withdraw dry solvent. A stopcock and septum port allows a long syringe to be inserted and a small quantity of solvent withdrawn. Since many solvents dissolve common lubricants, the stopcock should have a Teflon plug. A 4 mm Teflon stopcock is stronger and it is easier to insert a needle through the 4 mm bore. Adapting the outlet side for a larger septum will allow more punctures without necessitating the change of the septum (Photo 10).

Photo 10

Photo 11

The standard taper joint allows a flask to be attached if larger quantities are desired. The drip tip and hose barb allow the flask to be evacuated and backfilled with inert gas if necessary. The brace rod provides additional support and lessens the chance of breakage (Photo 11).

6. Valve: The 120º Teflon stopcock is a good choice. It is easy to observe the proper orientation of the valve, either to collect dry solvent for use or to return unused solvent to the distillation flask. Although a T-bore or double oblique stopcock is often used, neither is as intuitive or user friendly as the 120º Teflon stopcock (Photo 12).

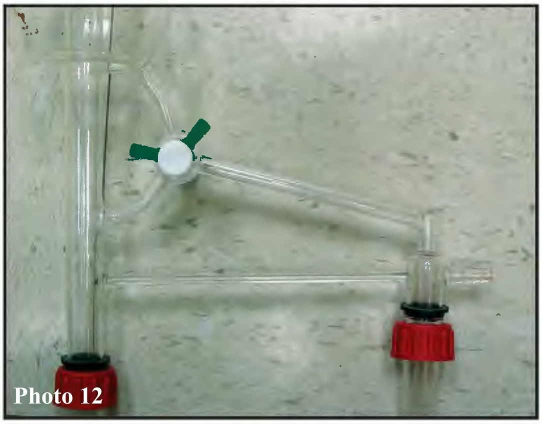
Photo 12

7. Other Considerations: Occasionally the distillation flask may "bump" upon heating. The standard taper joint at the bottom of the collection reservoir is a drip tip joint which has been extended, closed off, and a large hole blown in the side to function like a bump trap and prevent undistilled solvent from splashing up into the reservoir (Photo 13).


Photo 13

Photo 14

Since many solvents attack commonly used lubricants, Rodaviss® joints have been used in place of the standard ground glass joints. They do not require grease for a tight seal and the loosening feature aids in safe dismantling of the component parts. Glindemann PTFE Sealing Rings provide an extra layer of protection and also protect the Rodaviss® O-ring from potential exposure to solvent and possible swelling (Photo 14).

SOME OPERATIONAL CONSIDERATIONS


Photo 15

Due to the highly reactive nature of some of the drying agents used and the flammability of many solvents, the safe and proper operation of a solvent purification still requires good laboratory practice with extra precautions to minimize the possibility of an accident (Photo 15).

One important safety feature is a water flow monitor which will cut

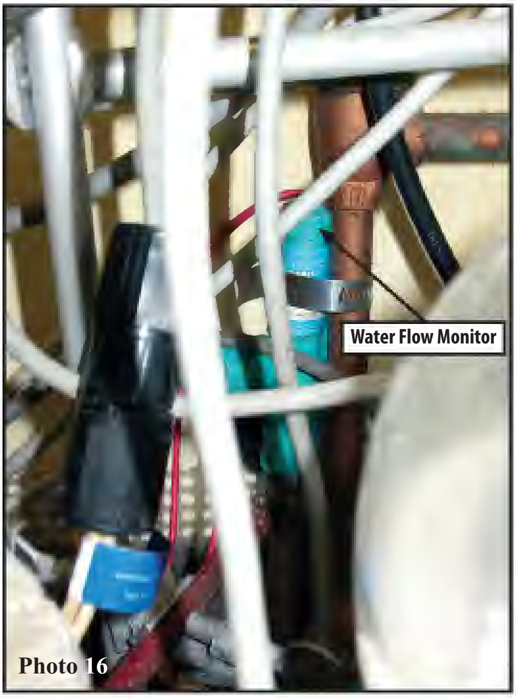

Photo 16

Photo 17

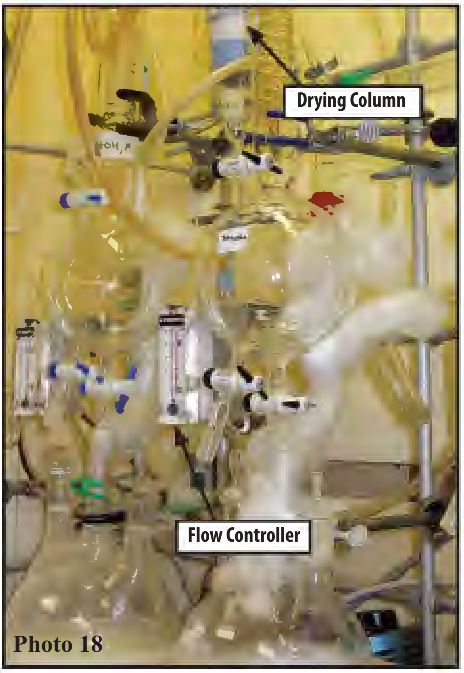

Photo 18

off power to the heating mantle(s) if the water supply is interrupted or the flow rate drops. (Photos 16 & 17)

Individual inert gas supplies with flow meters and bubblers to accurately measure and monitor the flow of the inert gas help prevent possible cross contamination and are a visual reminder that the still may be in operation (Photos 18 & 19).

Photo 19

Proper labeling of the contents of each still is critical, especially when there are multiple users (Photo 19).

Sometimes the inert gas itself may need to be dried and deoxygenated. A simple drying train may be set up for this function (Photo 18).

CONCLUSION

Solvent purification stills are fascinating in their many designs and functions. However, they are not the only way to obtain pure solvents. Sometimes merely storing the solvent over a molecular sieve will yield excellent results. Running some solvents through a column of activated alumina may work best. Thoughtful consideration must be given to safety, the quantity and the purity of the solvent needed. Fortunately, there are plenty of references and resources available to help you and your researchers make an informed decision.

ACKNOWLEDGEMENTS

I would like to express my appreciation to Kansas State University and the Department of Chemistry for their support of the scientific glassblowing facility and their continuing encouragement of my professional endeavors.

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Custom Sizes of Square, Rectangular and Elliptic Tubing

by

Doni Hatz[a]

ABSTRACT

Borosilicate glass tubing is softened and shaped over graphite forms. There are many sizes of square and rectangular tubing commercially available. Sometimes the researcher requires an off dimension size not readily available. When only a small amount of glass is needed for the project, drawing your own custom shaped tubing is easy to do. Depending on the researcher's objective for shape and size versus optical clarity, this method works extremely well for small projects. If optically clear glass is ideal, that is an entirely different method using borosilicate glass plate. Five sizes of custom shaped glass tubes are shown below.

PROJECT 1. Square Tubing

Graphite works well for a mandrel to shape the glass tubing. It offers good dimensional stability at high temperature because of very high thermal conductivity and a very low coefficient of thermal expansion. There are many different grades of graphite that vary in density and porosity. This graphite material is G10 quality but the description number may vary with manufacturer.

The graphite block is 2 inch outside diameter (o.d.) x 4 inch L (length). It has been drilled and tapped to about a 35 mm depth to fit a 3/8" stainless steel threaded rod (16 threads per inch). The threaded rod needs to be long enough to keep the pluro stopper from being heated, about a 12-15 inch length. A glass holder is made and slipped over the threaded rod to support the weight of the heavy graphite block.

The graphite support holder is made with 12 mm o.d. medium wall (MW) tubing in the middle (that sleeves the 3/8" threaded rod nicely) with two flares on each end. To make the graphite holder, seal the 12 mm tubing to 70 mm standard wall (SW) tubing. Fire cut the 12 mm tubing, about 3-4" length, then seal on another piece of 70 mm o.d. tubing. Trim the 70 mm tubing with the wet cut off saw on each side near the shoulder leaving 3-10 mm of the 70 mm tubing. Fire polish the saw cut ends and flare up slightly so the flare fits inside the 80 mm tube with extra room to wrap masking tape on the edge of the 70 mm flare to keep it from moving inside the tube. This also prevents the flare from cracking easily when slipping it inside and out of the tubing.

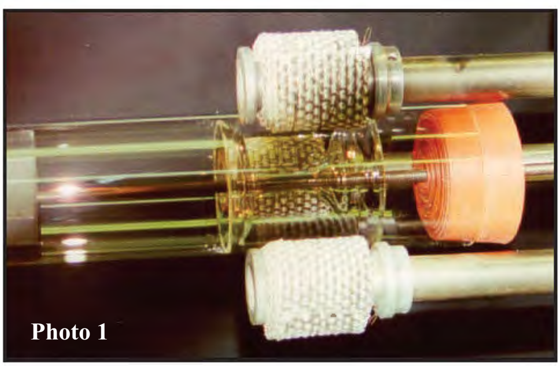


Photo 1

A two inch inner diameter (i.d.) square tube is made from 80 mm o.d. tubing. Insert the glass support with flared ends over the threaded rod, add the pluro stopper over the threaded rod and slip inside the 80 mm o.d. tube (Photo 1).

A blow hose is attached to the left

[a] The Procter & Gamble Company, Mason Business Center, 8700 Mason Montgomery Road, Mason, Ohio 45040. Email: Hatz.dj@pg.com.

side of the 80 mm o.d. tubing. Begin warming up the tube with a large torch, in this case a Carlisle CC blast burner (Photo 2).

Increase the heat with a cradle burner, to heat a larger cross section of glass. The cradle burner, also a Carlisle, consists of six heads, seven jets per burner head. Continue heating and shrinking the tubing, pulling it down on to the graphite mandrel from right to left. Be careful not to shrink the tubing completely around the right side of the mandrel or you cannot remove it easily (Photo 3).

The glass is shaped nicely around the graphite mandrel (Photo 4).

Once the tubing has formed around the graphite mandrel, flame anneal and remove the graphite form. Anneal in the oven (Photos 5 & 6).

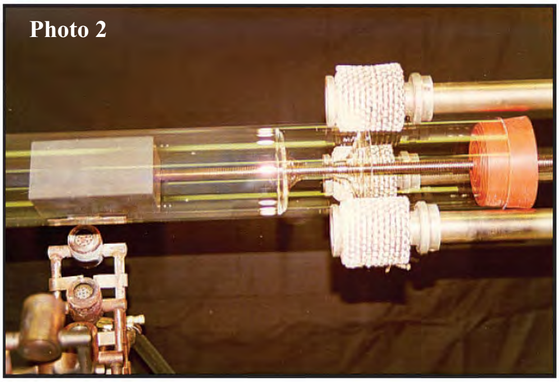
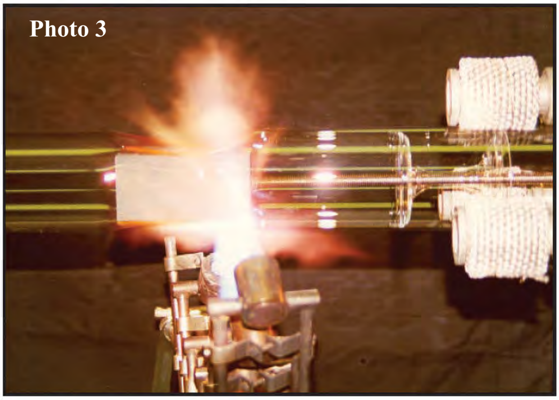


Photo 2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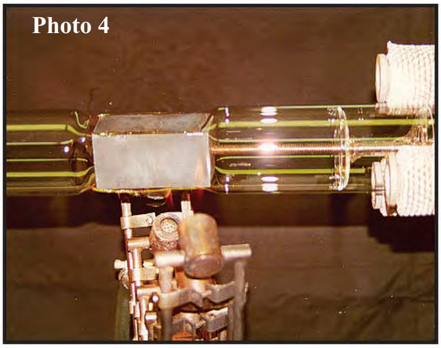
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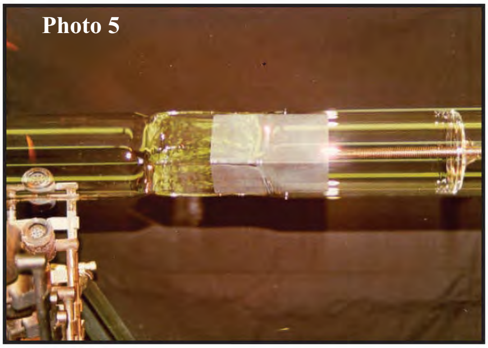


Photo 5

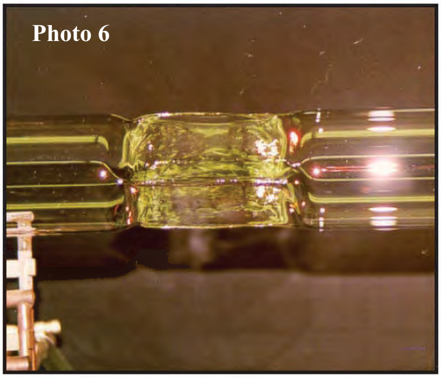


Photo 6

PROJECT 2. Rectangular Tubing

The rectangular tubing is made identical to the square tube. A 3 inch x ¾" o.d. graphite block is supported inside 85 mm o.d. tubing. A support holder for the graphite mandrel is made to fit inside the 85 mm o.d. tubing (Photo 7).

As the glass is heated right to left, pull the tubing slightly while you suck in the air to help the tubing slump over the graphite man-

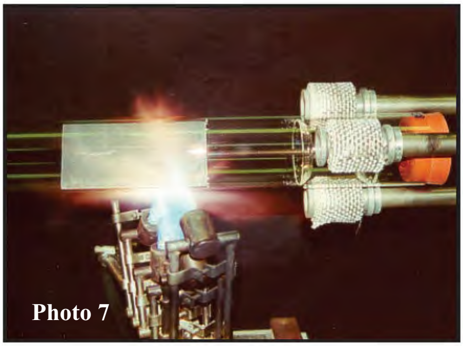


Photo 7

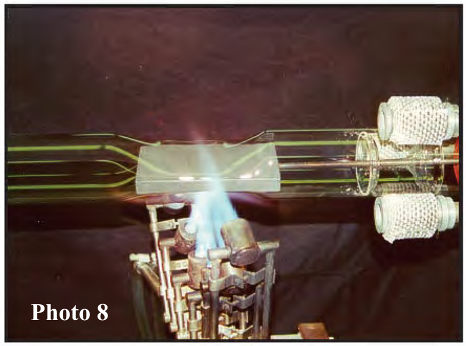


Photo 8

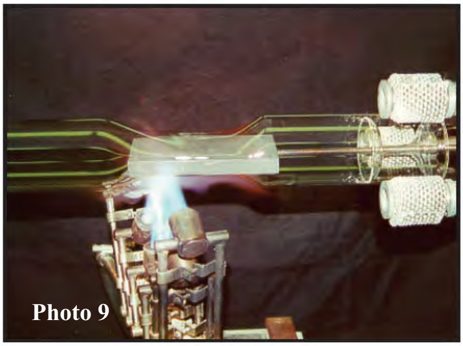


Photo 9

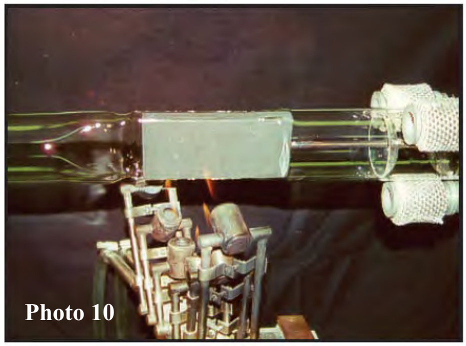


Photo 10

drel (Photo 8).

Since there is more glass to manipulate for the rectangular shape, it helps to continue pulling and thinning the glass tube until it slumps over the graphite to its final shape (Photo 9).

Once the tubing has formed around the graphite mandrel, flame anneal and remove the inside support (Photo 10).

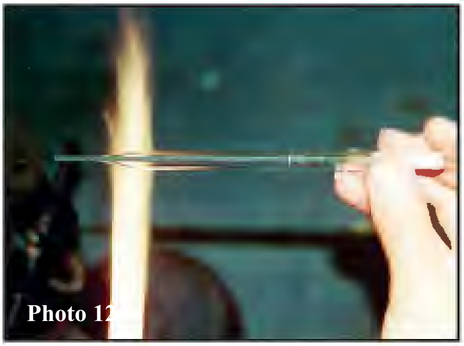
PROJECT 3: Small Square Tubing

The small square tubing is fabricated at the bench with a Carlisle CC blast burner, it is overkill for this project but this is my standard torch. On one end of a ¼" graphite rod, grind a two inch section into a 4 mm square with the wet belt sander. A fine finish can be achieved using an emery cloth. A slight taper on the graphite makes it easier to slip out of the tubing except for the final area of critical dimensions for the cell.

Photo 11

A glass handle is fused onto the end of the graphite mandrel. Once cool, masking tape is wrapped around the glass near the glass-graphite seal until it fits inside 14 mm o.d. SW tubing.

Begin heating the 14 mm o.d. SW tubing (Photo 11).



Pull and shrink the glass over the graphite mandrel (Photos 12 & 13).

Remove the mandrel as soon as possible. If it sticks, heat the tubing again with a soft bushy flame pulling on the mandrel until it loosens and slides out of the tube (Photo 14).

A stainless steel mandrel can also be used instead of graphite. Aerodag® G (a spray form of Aquadag®) is sprayed on the stainless steel to prevent the glass from sticking to the metal.

PROJECT 4: Elliptic Tapered Tubing

The elliptic tubing is fabricated similar to the square and rectangular but in this case it is demonstrated at the bench torch. The graphite piece was ground down at the belt sander to specific dimensions of 38 mm x 14 mm at the top end tapered down to 24 mm x 14 mm, a total length of 5 inches. A print-out of the elliptic shapes from the computer work well to size up the graphite. The graphite mandrel is drilled and tapped to fit a ¼" stainless steel threaded rod. The inside graphite support is made with 41 mm o.d. and 10 mm o.d. glass tubing.

The graphite mandrel with glass support is slipped inside 45 mm o.d. SW tubing secured with a pluro stopper over the threaded rod (Photo 15).

A point is pulled down in alignment with the bottom edge of the graphite mandrel. Cut open the tip of the point to let air escape (Photo 16).

Heat and shrink the tubing on the narrow end of the graphite mandrel, this keeps it from flopping inside the glass tube (Photo 17).

Continue to heat the tubing right to left, pulling the glass to slump onto the graphite (Photos 18, 19 & 20).

Once the glass is shaped, pull out the mandrel and flame anneal (Photos 21 & 22).

Photo 17

Photo 18

Photo 19

Photo 20

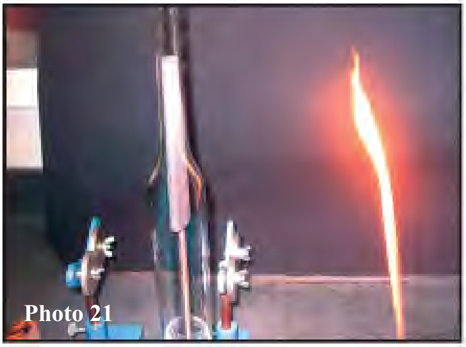


Photo 21

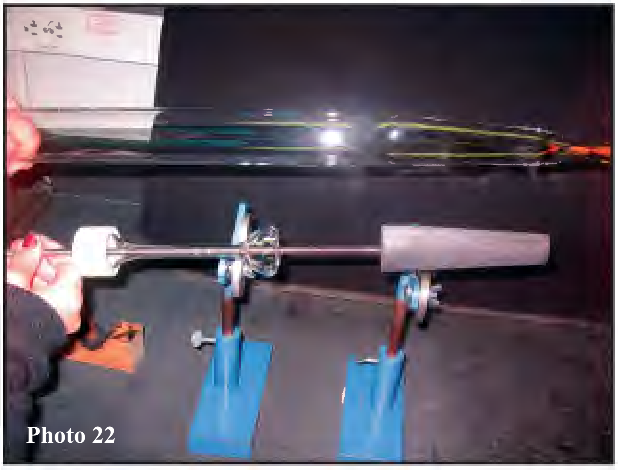


Photo 22

All pieces are oven annealed before trimming them to their final dimensions.

PROJECT 5: Rectangular Tray

By David Wedsworth, Johnson Controls, Indianapolis, Indiana

Similar use of a graphite mandrel is shown making a rectangular tray. In this case the graphite is drilled and tapped in the middle of one face of the graphite. The graphite holder is chucked into the lathe tailstock and a 5 liter round bottom flask is chucked into the headstock (Photo 23).

Photo 23

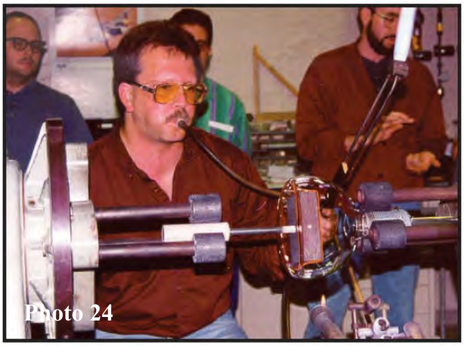


Photo 24

A blow hose is attached to the flask to control blowing the glass around the bottom and side surfaces of the graphite (Photo 24).

The graphite is removed quickly after molding it into the flask (Photo 25).

Flame anneal the flask, then oven anneal before trimming the excess glass to release the rectangular tray.

Photo 25

CONCLUSION

These methods work great for small pieces of custom shaped tubing. As with most projects, preparation and set-up of materials take more time than the glassblowing part. With this information you can try these simple methods. Please give me feedback of new and improved methods that you develop along the way so I can update the information.

ACKNOWLEDGEMENTS

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Photographs were taken by David Anderson (elliptic tubing) and by the artist.

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