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CONTENTS

Papers

Other Information

Papers

An Analysis of the Sodium Oxide
Deposition on Borosilicate Glass
Caused by Flame Working

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Introduction

When you heat a small region of glass with a torch, such as when creating a T-seal, one is likely to observe a whitish deposit about a centimeter or two from the worked area (Photo 1). Some glassblowers assume this is a quartz flume as it is rather similar to what is seen when working quartz. Although similar in appearance, the deposit is

Photo 1

not likely quartz as the working temperature of quartz is about 2300°C which is about 1000°C higher than the working temperature of borosilicate.[1]

A second inference for the approximate temperature of this phenomenon can be shown by the proximity of strain lines in glass in relation to the deposits (Photo 2). Here, the carbon rod is pointing to a deposit line. This provides an indirect measurement that the temperature in the location of condensed deposit is something greater than 565°C but certainly not 1000°C greater than 565°C.

Photo 2

Rather than silicon dioxide (SiO_2), it was the feeling of the authors that this deposit was sodium oxide (Na_2O). There are a variety of factors providing empirical evidence for this belief. When you heat glass with a gas/oxy flame, the sodium emission is easily observable. This is more so in borosilicate glass than say in aluminosilicate glass where the sodium content is 3.8%–4% versus 1.0% respectively. Didymium glasses are not necessary when working on aluminosilicate glass. In addition, this deposit is seldom observed when working aluminosilicate glass, typically dependent upon the glass's manufacturer and the type of flame used.[2] Also regarding the sodium emission, the emission's appearance varies when flame annealing in a previously-worked region (Photo 3). When looking over one's didymium glasses, one can observe a high sodium flame coming off glass that has not been struck by a flame, while in the immediate region around a worked area there is often a void. Another observation on a possible sodium link to the deposit is the evidence for sodium depletion in a heavily-worked region. This can be observed when glass has been aggressively overworked and where devitrification cannot

Photo 3

be removed by simply reheating the glass. It has been shown by Allan Brown that adding common table salt (NaCl), crushed glass, or sodium perborate ($NaBHO_3$) to the glass and flaming these materials into the overworked region helps remove the devitrification.[3,4]

Experiment

The obvious relationship between sodium and borosilicate glass is easily established. However, this relationship does not prove that the deposit is sodium oxide. A formal analysis of the deposit was required, and this was made possible via X-ray Photoelectron Spectroscopy (XPS) (Photo 4). Seen here, with co-author Michael Quinlan, is the experimental housing region. In addition, there are other parts of the equipment (not in view) about three times this size that house the electronics and computational parts of the device. Generally, X-ray Photoelectron Spectroscopy measures the energy of electrons ejected from an atom or molecule when a material is irradiated with photons. More specifically, the samples are placed on a carousel inside a vacuum chamber and brought down to a 10^{-10} torr vacuum (Photo 5). Within the chamber, an X-ray beam is aimed at the sample. The beam is sufficient to ionize an atom and thereby electrons are ejected. The emitted electrons are collected so their kinetic energies can be measured. The amount of kinetic energy of the electrons leaving t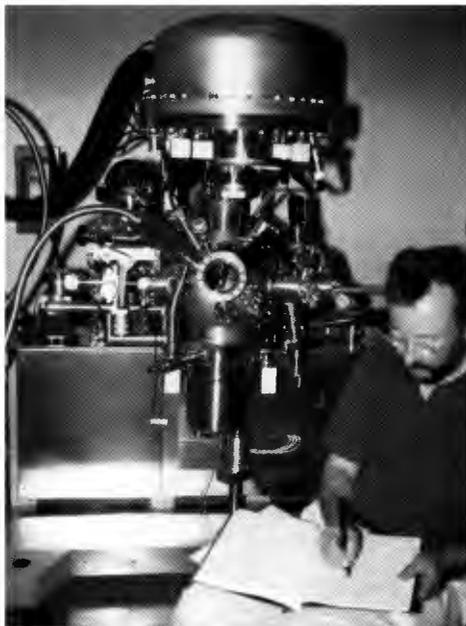he sample is directly proportional to their chemical bondin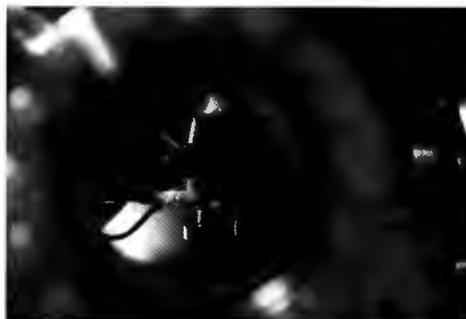g such that it is also possible to determine the chemical state of the analyzed species. Generally, the energies produced by this means allow for about a 5 nm depth of study for sodium and about a 10 nm depth for the anaylsis of the silicon.

Photo 4

Photo 5

Results and Discussion, part 1

The initial data was taken on pieces of an nmr tube. One control sample had nothing done to it. A second was heated in such a manner that a deposit was observed. Running these samples through the XPS showed the sample with the deposit having a significant increase in sodium content (Charts 1 & 2).
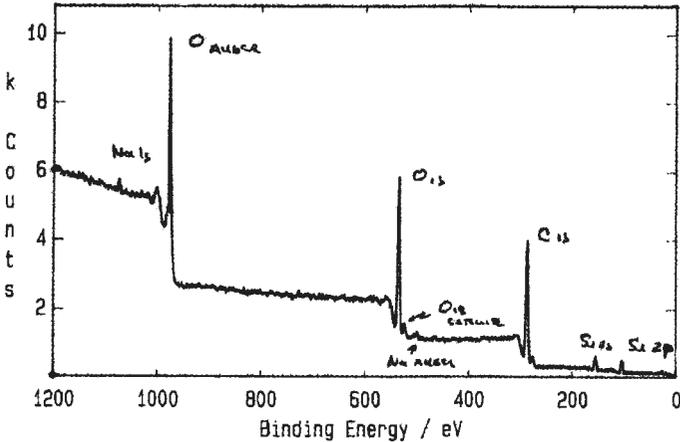
Chart #1 - Glass Standard

V.G.Scientific		XPS – Spectrum	V.G.Scientific	
13MAR02.DAT	Region 1 / 1	Level 1 / 1	Point 1 / 1	

Radiation
Al Kalpha
Max Count Rate
19691 CPS
Analyser
100 eV
Step Size
1.00 eV
Dwell Time
50 ms
No of Channels
1201
No of Scans
10
Time for Region
601 Sec
Acquired
11:26 13-Mar-01
Plotted
16:14 15-Mar-01

Untreated interior surface; flood gun o

Chart #2 - Heated Sample

V.G.Scientific		XPS – Spectrum	V.G.Scientific	
13MAR03.DAT	Region 1 / 1	Level 1 / 1	Point 1 / 1	

Radiation
Al Kalpha
Max Count Rate
34873 CPS
Analyser
100 eV
Step Size
1.00 eV
Dwell Time
50 ms
No of Channels
1201
No of Scans
10
Time for Region
601 Sec
Acquired
11:43 13-Mar-01
Plotted
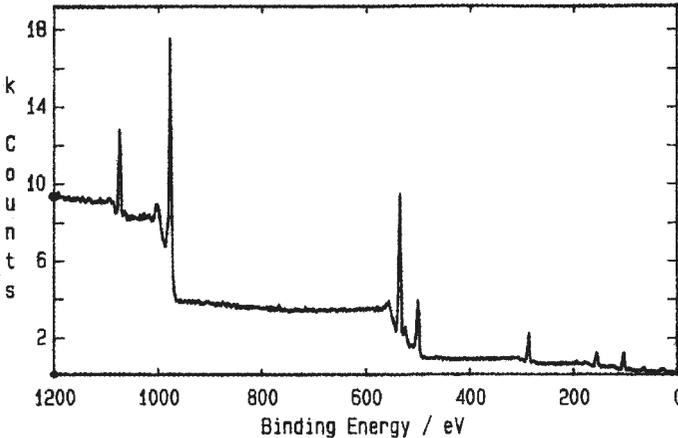16:23 15-Mar-01

Heat treated exterior surface; flood gu

Charts 1 & 2

Since the results of this test proved promising, five more samples were prepared. To prepare these samples, a ⅟₁₆ inch plate of borosilicate glass was cut into 1 cm strips. One strip was left as was as a Control Strip. The two samples were heated (*in situ* Samples 1 & 2) on one end so that a deposit was created on the other. One other sample was heated aggressively on one flat surface ("concave") while the other side remained away from the flame sample ("convex"). The final sample was collected in a different means entirely. When large tubing is firecut on the lathe, just before the fire cuts through the glass, a smoke-like substance is seen to appear on the inside of the tubing (Photo 6) streaming from the firecut region. There was suspicion that this was also a sodium compound and it was hoped that this set of experiments could provide some indication of the smoke's composition as well. A sample was prepared by placing a glass slide on the end of a glass rod. This was then placed through the head stock of a glass lathe and into the region near where some large glass tubing was in the process of being fire cut. Once the deposit coated the slide, it was prepared along with the other samples.


Photo 6

As shown in Chart 3, the results clearly show that sodium is continuously removed from the surface of borosilicate glass while it is worked with a torch. Both *in situ* samples show an increase in sodium levels above the level of the control sample. The "toasted sample" shows that the heated side (the concave side) is dramatically reduced in sodium concentrations while the opposite side (the convex side) shows higher levels of sodium than the control. As can be seen on the "vapor deposited sample," the suspicion that the smoke inside tubing during fire cutting was sodium was also correct.

Finally, a last test was performed on test samples where salt (NaCl) was added to glass that had been heavily heated with a torch. As seen in Chart 4, the sodium concentrations were significantly high indicating that a sufficient amount of sodium has been added to replenish the depleted sodium regions. In all cases of increased sodium concentrations found on the test samples, the binding energy is consistent with the interpretation of a film of sodium oxide.

Sodium content in near surface region
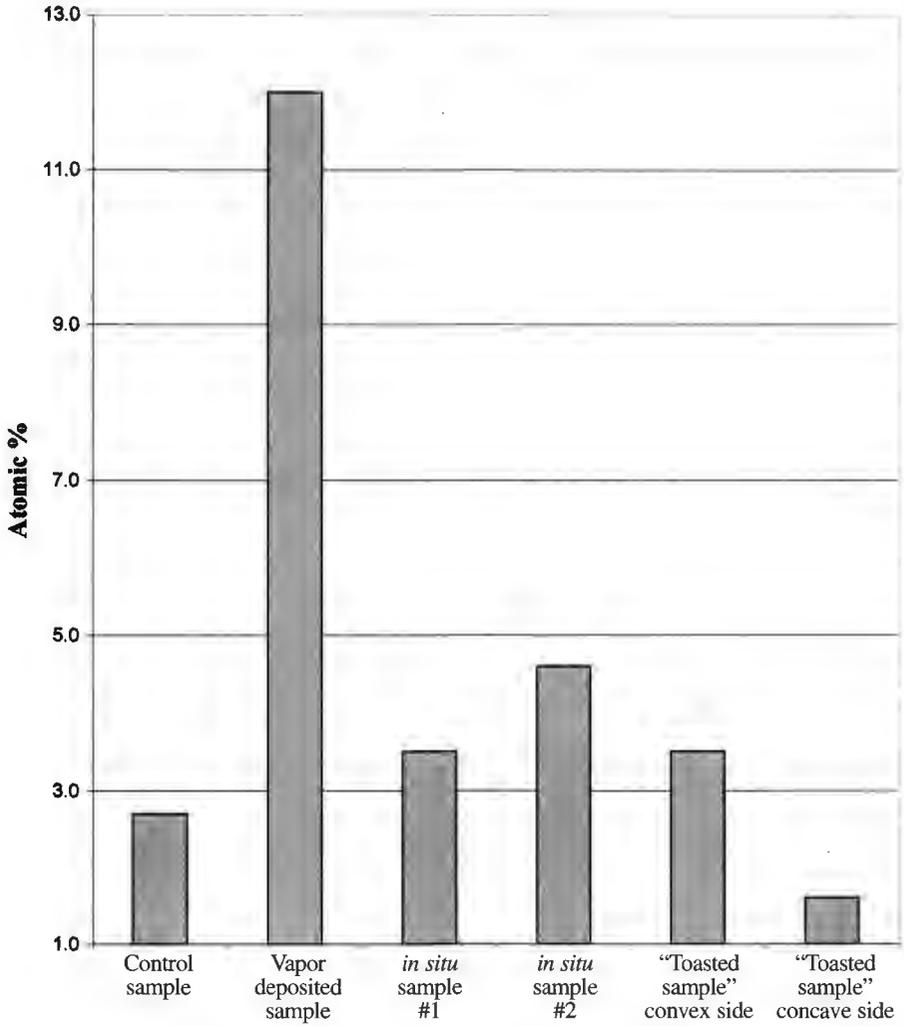
Sample Description
Chart 3
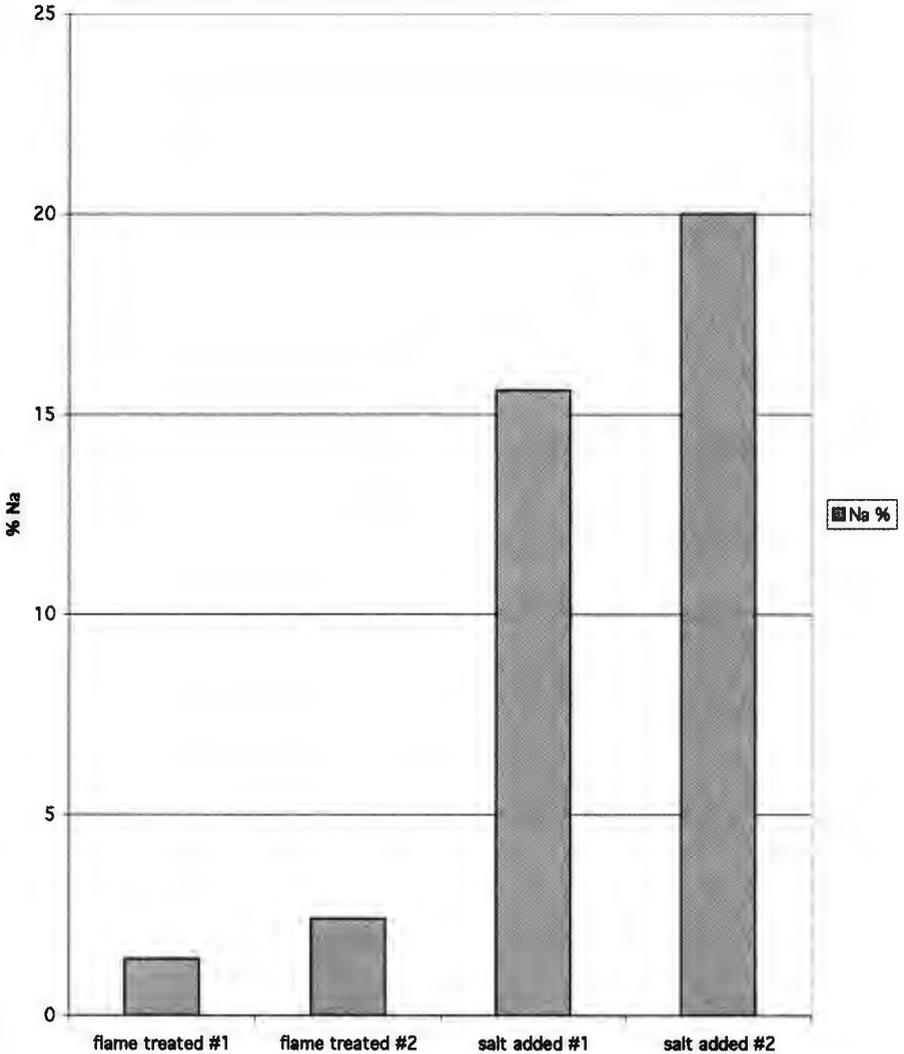
Chart 4

Results and Discussion, part 2

The next question is how does the deposit get to where it lies? The original supposition is that the means of travel was via air. That is, the torch's flame heats the sodium to such levels that it evaporates off the surface of the glass. The sodium that is not burned in the flame sublimes onto a cooler glass region where it collects as a deposit. My collaborator on this project suggested that the deposit was probably the result of surface migration. A simple experiment was devised to determine which approach was likely.

Photo 7

A metal washer was selected that just fit around a 10 mm glass rod (Photo 7). Just to one side of the washer, a torch flame was aimed at the rod rotating in a lathe. As is seen, the deposit was equidistant on both sides of the heated area, providing strong credence to the migration theory. If the deposit was airborne, it would not likely have appeared equidistant to both sides of the heated region. Note also that the strain lines again match up with the deposit lines (Photo 8).

Photo 8

The transport mechanism of the sodium demonstrates some of the unique aspects of glass structure and the function of the alkalis added to the silicon oxide matrix. From the beginning, sodium carbonate and other alkali metal oxides were added to glass as a means to lower the melting temperature and to achieve a lower viscosity of the molten glass. However, the silicon dioxide matrix is a very open structure leaving vast cavities for permeation of the materials through the glass. In addition, when heated, the sodium scoots across the surface like tumbleweeds until the glass is sufficiently cool so as to stop the migrational travel.

The migration of sodium from heated regions has already been established at temperatures of as little as 200–300°C.[5, 6] Similarly, focused heating via electron probe microanalysis (EPMA) can lead to a migration of alkali elements in glass away from a heated region.[7] It has been found that one way to prevent the migration of sodium during such analysis was by cooling the glass to minus 100°C.[8] Thus, when glass is sufficiently heated from one side, there is a migration due to permeation through the glass and, if the heat is great enough, the sodium will be expelled into the air. This is what is happening inside glass tubing when firecut. The difference between the inside and the outside while firecutting glass is that on the outside the majority of the airborne sodium is burnt up in the flame. On the inside, the sodium is left remaining to settle back onto any available surface. This is why the inside of large tubing tends to collect large quantities of the deposit.

Conclusions

It has been shown that the deposit surrounding any borosilicate glass region that has been worked by a gas/oxy torch is sodium oxide. It has also been shown that the smoke seen on the inside of glass and the deposit on the inside of glass apparatus is also sodium oxide. There may be other components to this material, but this was not examined.

It is beneficial to consider what the significance of these observations are for the glassblower. There are several. First is flame annealing: The sublimation temperature of sodium oxide (Na_2O) is 691°C — less than the softening temperature of borosilicate glass (821°C) but more than its annealing temperature (560°C). Keep in mind that annealing is a time-temperature based figure as glass can be annealed at a higher temperature for less time. The temperature for proper annealing is based on annealing a $\frac{1}{4}$ inch thick piece of glass for 15 minutes for no slumping to occur. The latter is important because we have no control on sagging inside an oven. However, we do have some control on over-heating while flame annealing and can respond accordingly. In addition to simply responding to sagging glass, we can use the deposit as a visual indication of approximate temperatures. Once the deposit has been burned off, we know that the surface of the glass has reached at least 691°C and the glass should be flame annealed.

Similarly, we can look over our didymium glasses and observe the sodium flame's irregular brightness at the beginning of the flame annealing process. Once we observe an even and regular sodium glare, we have an indication that the glass temperature is now generally uniform.

If we cannot remove the sodium because it is not possible to sufficiently heat the glass due to the dynamics of the apparatus, we can remove the deposit with nitric or hydrochloric acid. As has been shown by Jang, boiling glass slides in either hydrochloric or nitric acid significantly reduced the alkali metal concentrations at the surface of glass at a depth of up to 70 nm.[9, 10]

On a side issue this provides a likely explanation to several observed phenomena: when glass is dramatically overheated, it does not flow as well as the surrounding glass. The explanation for this phenomena could simply be that an extensive amount of the sodium has been removed from the glass so as to render the remaining material non-flowing. Thus, the practice of removing the errant strips and seeds of glass that no longer flow into the remaining glass is the best option.

Secondly, sodium depletion is also a likely explanation as to why you may find a small bit of strain that cannot be annealed away when one seals glass that has been fire cut to glass that has not been fire cut. Here, the change in sodium composition is probably changing the coefficient of expansion to a new, unknown value which is not likely to match that of the original glass.

End Notes

[1] R.M. Caldwel and J.D. Barker, *Lamp Glass Handbook,* © (1992 GE Components (Marketing & Sales Operation)): 55.

[2] Michael Souza, personal conversation, June 2001.

[3] Allan Brown, "Devitrification of Borosilicate Glasses; What Causes it? How can it be reversed?" Demonstration at 44th Annual ASGS Symposium, Princeton NJ, 1999.

[4] Allan Brown, "Correcting Devitrification Caused by Overworked Pyrex," *Fusion* 46.3 (August 1999): 59.

[5] R.J. Baird and R.J. Haeberle, "The effect of alkali ion migration on the adhesion of sputtered chromium metallizations to glass," *J. Vac. Sci. Technol.* A 4.3 (May/June 1986): 532–536.

[6] L.F. Vassamillet and V.E. Caldwell, "Electron-probe Microanalysis of Alkali Metals in Glasses," *J. of Applied Physics,* 40.4: 1637–1643.

[7] Jennifer Mass, Albert Ammerman and John A. Hunt, "Archeological exploration with the electron microprobe: The early history of glassmaking in the Venetian lagoon," *American Laboratory,* 33.8 (April 2001): 52–55.

[8] C.H. Nielsen and H. Sigurdsson, "Cryogenic Elimination of Sodium Loss in Glasses During EPMA Analysis," *Proceedings of the Microbeam Analysis Society, 1980, 15th annual meeting:* 149–142.

[9] H.K. Jang, Y.D Chung, S.W. Whangbo, Y.S. Lee, I.W. Lyo, and C.N. Whang, "Effects of chemical etching with sulfuric acid on glass surface," *J. Vac. Sci Technol.* A 18.2 (March/April 2000): 401–404.

[10] H.K. Jang, Y.D Chung, S.W. Whangbo, Y.S. Lee, I.W. Lyo, and C.N. Whang, "Effects of chemical etching with hydrochloric acid on a glass surface," *J. Vac. Sci Technol.* A 18.5 (September/October 2000): 2563–2567.

Comparison of New PTFE Joint Sealing Rings with PTFE Sleeves for Taper Joints

by
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Abstract

Here we compare the sealing performance of PTFE sealing rings, sleeves and stoppers for glass taper joints measured as vacuum leakage of apparatus and as solvent leakage of stoppered jars. We found that the stiff PTFE is a very effective replacement for the soft joint grease if used in the sophisticated shape of sleeves and stoppers with sealing ribs or as a slim PTFE sealing ring under a high sealing pressure. The lowest possible leakage can be performed using a new flat cylindric PTFE sealing ring which is less than 1mm wide, 0.1mm thick and weighing only about 5mg. The new sealing ring is high-vacuum tight (air leakage rate $10^{-8}\ldots10^{-6}$ mBar x liter / sec), solvent tight (loss of ethyl acetate out of jars < 0.1mg/day) and resistant to fluctuation of temperature (-196 to +300°C). The reusable PTFE sealing ring is thin enough to be used with Polyacetal or metal joint clamps and does not need to be fixed by a groove on the glass joint.

Introduction

Laboratory work with traditional taper jointed glassware is facing problems of joint freezing and, more seriously, of joint sealing (References 1-5). Joint grease can often not be used because it is not inert. PTFE joint seals are more inert and prevent joints from freezing. But there is a widespread misconception that PTFE is too expensive and too hard for a good seal of the rough glass joint surface. Unfortunately, little quantitative leakage measurement data has been published on the sealing performance of different shaped PTFE taper joint seals (References 1, 6). Frank (1) found that PTFE joint sleeves have no additional sealing effect. We found, however, that the shape and performance of the existing PTFE joint seals is very different and some of them offer an amazingly good and economic seal. Here we will examine the relation between the shape and the sealing performance of PTFE joint sleeves, stoppers and newly-invented sealing rings (References 5, 6).

Materials and Methods

<u>Materials</u>: The glassware contained German taper joints and stoppers NS 29/32 of good (smooth) standardized quality. The new sealing rings (Reference 6) were from Kimble-Kontes, USA, catalogue No. 676005-2942 for taper 29 needs no groove on the glass taper. The classic PTFE seals for joint 29/32 were: Disposable PTFE sleeve without sealing ribs (BRAND, Germany, No. 51422), PTFE sleeve (socket) with two outside sealing ribs (Bohlender, Germany, No. H 930-07), PTFE sleeve (socket) with two inside sealing ribs and grip collar (BRAND, No. 51467) and PTFE stopper with two sealing ribs (BRAND, No. 144458). The other materials used are very common in most catalogues.

<u>Standard sealing procedure</u>: The material was cleaned (degreased) by using ethyl acetate. The joint parts were uniformly pressed together at room temperature using an axial force of 3 kp for three seconds and one joint part was rotated around a 45° turn to overcome the dynamic friction. The time period between applying this <u>initial sealing pressure</u> and the leakage measurement was controlled as reported in the result section.

<u>The gas leakage rate</u> is a measure of the ability of apparatus to sustain vacuum or pressure. It is dependent upon the leaking gas. The gas leakage rate (detection limit for air 10^{-8} mBar x l / sec) was measured as air pressure increased in an evacuated and thermostated volume with the tested PTFE seal on a test joint (German DIN 12256 protocol (Reference 1)).

<u>The solvent leakage rate</u> is a measure of the ability of stoppered jars to contain volatile chemicals. The solvent leakage rate (mg/day) was measured as the weight loss of 2 g of ethyl acetate from 300 ml Erlenmeyer flasks with a taper joint 29 and a stopper 29 stored for 28 days under room temperature and atmospheric pressure. The leakage of vials was measured by the same method.

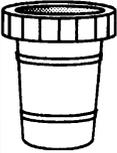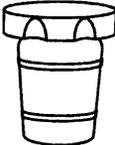**Results and Discussion**

<u>Gas leakage and solvent leakage</u>:
Table 1, Figures 1 and 2 show that the lowest possible leakage of all PTFE seals is performed by PTFE sealing rings. PTFE sleeves without sealing ribs have no significant sealing effect (see also Reference 1). PTFE sleeves and stoppers with sealing ribs reduce the leakage of PTFE sleeves. The statistical variation of leakage rates in Table 1 is small compared to the average values of each single type of PTFE seal, and is also represented in Figures 1 and 2. The gas leakage is reduced at a higher working temperature (Figure 1) when the PTFE becomes softer and, therefore, is providing a better sealing contact with the rough glass joint. The leakage is also reduced over the course of the time period after the joint was pressed together (Figure 2) because the PTFE needs time to flow under the sealing pressure to seal the leak (as shown in Figure 3D). The surprisingly low leakage of PTFE taper joint sealing rings can be explained by their small dimensions which provide a high sealing pressure while using only low sealing force (Figure 3). The low leakage under high pressure indicates that both gas convection between the PTFE surface and the joint and the gas diffusion throughout the micro-pores of PTFE are reduced.

<u>Comparison of sealing rings with vials and screw cap flasks (Table2)</u>: Taper joint flasks with sealing rings are as tight as glass vials but much more tight than plastic vials. Glass vials lose their tightness after the first penetration of the septum by a sampling syringe.

<u>Mass of PTFE (mg) used per seal</u>:
The small dimensions and low PTFE mass of the seal offer different advantages. The low mass of the PTFE seal (joint 29 PTFE ring 6 mg, sleeve 490...7000 mg, stopper 66000 mg, see Table 1) is not only inexpensive but is also environment friendly. A small seal ring is also only a small source of contamination. PTFE is indeed chemically resistant but it is a source of substance loss (by absorption in its micro-

TABLE 1
Comparison of PTFE sealing rings (references 6, 7), sleeves and stoppers for glass taper joints

Name of seal	Sealing ring	Sleeve with 2 ribs and grip collar	Sleeve with 2 ribs	Stopper (PTFE) with 2 ribs	Sleeve without ribs	Joint without sealing
Picture of seal						

Gas-leakage rate of air and statistical fluctuation (mBar l /sec)
(Increase of pressure in mBar per second in evacuated glass bulb, 1 liter, taper joint 29, room temperature)

Average (n = 10)	7.5E-07	1.5E-04	1.3E-04	2.2E-03	4.5E-02	4.4E-02
Standard-deviation	6.7E-07	1.7E-04	1.2E-04	1.3E-03	3.7E-02	3.4E-02
Minimum	2.5E-08	2.5E-05	5.5E-05	5.0E-04	1.0E-02	1.0E-02
Maximum	2.5E-06	4.5E-04	2.3E-04	6.0E-03	7.3E-02	7.2E-02
Improvement (without seal =1)	58667	293	338	20	1	1

Solvent-Leakage of Ethylacetate and statistical fluctuation (mg/day)
(Each 10 bulbs, 300ml with taper joint 29 and stopper 29, room temperature, atmospheric pressure)

Average (n = 10)	0.05	0.55	0.27	3.67	9.09	5.31
Standard-deviation	0.02	0.37	0.18	2.36	2.29	1.19
Minimum	0.01	0.10	0.12	0.54	5.47	4.17
Maximum	0.08	1.00	0.46	5.90	12.37	7.79
Improvement (without seal =1)	106	10	20	1.4	0.6	1

Other properties

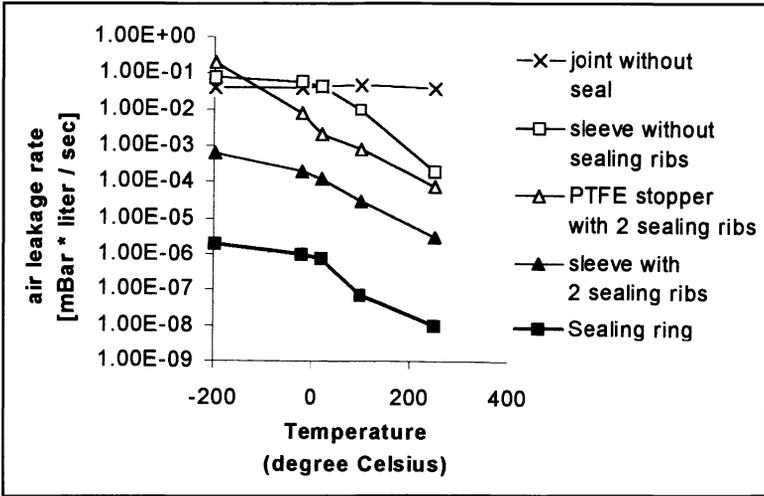|						
Mass of PTFE (mg) used per seal	6	7000	490	66000	425	
Upper temperature limit (°C)	300	260	260	260	260	450
Distance (d) of joint parts, mm	1.5	13	4	2.5	1.5	0
(matching KECK -clamps?)	(Yes)	(No)	(No)	(No)	(Yes)	(Yes)

Figure 1
Temperature dependence of gas leakage (air) of taper joint 29 with different PTFE seals

Before each of the temperature experiments (-196, -20, 20, 100, or 250°C), the initial sealing pressure was applied at 20°C. Then the joint was thermostated and the leakage measurement was started 0.1 days after sealing. The joint parts were separated (release of the sealing pressure) before a new measurement.
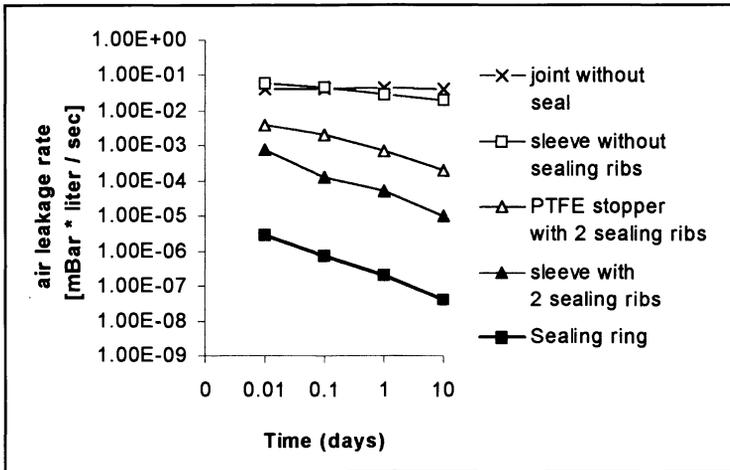

Figure 2
Time dependence of gas leakage (air) of taper joint 29 with different PTFE seals

The initial sealing pressure was applied at 20°C. The gas leakage was repeatedly measured at 20° in a time period up to 10 days after applying the initial sealing pressure.
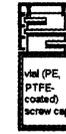
Jar	Reagent tube, Taper joint 14, 20 ml	Vial, Glass, 4 ml		Vial, Glass, 20 ml		Vial, PE 20 ml	Vial, PE+PTFE 20 ml	Flasc, Glass 250 ml
Closure	Stopper Glass	Screw cap Plastic		Crimp cap Aluminium		Screw cap Plastic	Screw cap Plastic	Screw cap Plastic
sealing element	Sealing ring PTFE	Septum (PTFE+rubber) Virgin	penetrated	Septum (PTFE+rubber) virgin	penetrated	Sealing rib in cap	Sealing rib in cap	Sealing rib in cap
	vial (glass), screw cap, septum		septum penetrated	vial (glass), crimp cap, septum	septum penetrated	vial (PE) screw cap	vial (PE, PTFE-coated) screw cap	nt, ss, ys
Solvent-Leakage (mg/day), average of each 10 jars								
Ethylacetate	0.05	0.02	11.90	0.70	12.30	9.40	10.20	0.10
Trichloromethane	0.05	0.05	28.80	1.50	11.50	10.20	6.60	27.40
Benzene	0.02	0.10	13.30	1.50	15.10	20.40	5.40	6.50

Table 2
Flasks with PTFE sealing ring (references 6, 7)
for taper joints compared to vials and flasks with screw caps.

pores) and of contamination (by desorption), for example, in trace analytical sampling. Also, the upper possible temperature limit is higher for low mass seals: the small sealing ring can be "baked" for cleaning together with the glassware at 300°C (short term at 330°C, handle with care as degradation of PTFE can cause lung edema). Heavy PTFE stoppers and sleeves can only be used up to 260°C because they produce far more toxic gases. This "baking" saves time in rinsing of the glassware.

Distance of joint parts and match of KECK-clamps:
A PTFE sleeve or ring of a thickness 0.05 mm separates the glass joint parts of a 1:10 taper at a distance of 1 mm or, more generally, 20 times that thickness (Table 1, Figure 3B). The thin PTFE sealing rings can be used in combination with KECK polyacetal or metal joint clamps whereas sleeves with sealing ribs are too thick for a good grip of the clamp. PTFE sleeves without ribs are thin enough to be used with KECK clamps, but they have no sealing effect.

A good and quick seal:
The extreme limit of greaseless taper joint sealing with PTFE was an air leakage $< 10^{-8}$ mBar x liter / sec and a solvent leakage < 0.03 mg/day at any temperature down to –196°C by using PTFE sealing rings (Reference 6). This low leakage can be achieved by heating up the PTFE or glass at 100…250°C before applying the initial sealing pressure (use a burner, lighter or hot-gun carefully). Extra low leakage was also achieved by making the glass joint surface smoother (rub the sealing ring by rotating one joint part until the glass looks shiny by impregnation with PTFE-micro-particles) or more slippery (addition of a micro scale amount of grease, solvent, water). As a comparison, an extra rough taper joint reduces the sealing power of PTFE.

Resistance of sealing against fluctuation in temperature:
Sealing rings showed no significant increase of leakage after five cycles of temperature change between –196°C and room temperature. On the other hand, PTFE stoppers will become loose during freezing (due to shrinkage of the PTFE). When these stoppers are fixed on the joint again at a low temperature, they can become stuck (see also Reference 5) after reheating or may even cause damage to the glass because of the expansion of the
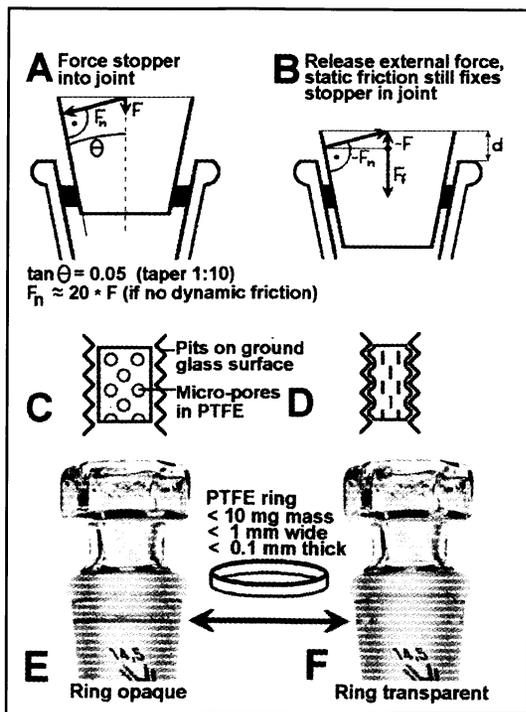
Figure 3
PTFE sealing ring before (left) and after (right)
pressing the two taper joint parts together

A) and B) Force leverage of glass taper, pressure and static friction: The force to provide the pressure necessary to make PTFE sealing (flowing) is limited because the female joint part should not break because of traction tension. If a glass joint 29/42 is initially pressed together using an axial force F of 3kp, and there is no dynamic fraction, then the normal force F_n vertical to the PTFE seal is 60 kg because of the glass taper leverage effect. The pressure (quotient force/area) onto a flat cylindric PTFE sealing ring (0.6 mm wide, 0.08 mm thick) is 118 Bar. Joint sleeves are under much lower pressure because of their higher contact area (sleeve with 2 sealing ribs each 1.5 mm width on one side only, 24 Bar; sleeve without ribs, 32 mm long, 2 Bar, virtually no flow of PTFE). If the external axial force of the joint is released, then the wedging caused by static friction force still holds a glass stopper and the sealing pressure is maintained. If the PTFE seal is thin enough, then the distance d in (B) is small enough for joint clamps (type KECK) to grip (see also Table 1).

C) and D) Deformation of PTFE: Microscopic model showing how sufficient pressure provides PTFE flow and compression to seal the pits on the rough glass surface of the joint and to reduce the micro-pores in PTFE. The PTFE and the ground glass pits are hooked up in (B, D) to deliver the important static friction which can fix a glass stopper without the necessary use of a joint clamp.

E) and F) Optical control of a good seal: A ground joint without a seal looks opaque because the passing light is scattered by the pitted glass surface. PTFE without pressure looks opaque or dull (E) because of its microporosity. A PTFE sealing ring under pressure looks transparent (F) as "grease" all around a good standard taper joint. It indicates a very compact glass-PTFE-glass contact, low microporosity and, therefore, is a good seal.

PTFE. Sealing rings are useful to fix glass stoppers on closed jars by wedging (static friction between ground glass and PTFE, see Figure 3B, D), making joint clamps unnecessary. The glass stopper can be separated easily by being moved back and forth, but it will not be inadvertently pushed out by the vapour pressure of the chemicals in the jar.

Conclusion:
The stiff PTFE is a very effective replacement for the soft joint grease if used in the sophisticated shape of sleeves and stoppers with sealing ribs or as a slim PTFE sealing ring under a high sealing pressure (References 6, 7). PTFE sleeves without sealing ribs have no significant sealing effect compared to a joint without any sealing means. Economical flat slim PTFE sealing rings (References 6, 7) can be used with outstanding advantage to seal greaseless standard glass taper joints. These rings are useful in the safe handling of sensitive or volatile chemicals and of analytical standards or samples in jars with a glass stopper or in glass apparatus.

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(7) Internet homepage: www.glindemann.net

Construction of a Quartz Cell Using Sealing Tape

by
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This paper will outline some of the things that I went through getting to where I now am in making quartz cells.

Sometime in the mid-to-late 1970's I, along with many others, was introduced to Vitta Sealing Tape. My initial attempts at using it were not successful. In 1981, in a conversation with one of our grad students, Ken Kosnick,[1] I found that he had also used it with some success. He stated that he had not followed the directions but had polished the ends of the tube, put the windows on, and then put the cell in the oven, which he ran to 900°C, pulling a slight vacuum during the sealing process. He opened the oven several times during the sealing process and felt this did not harm the cell. At this point, I would like to include the directions from the product data sheet:

After separating the release paper, the parts are placed on the adhesive layer of the glass tape. Subsequently they are pressed, using a pressure in the range of 50-250 psi for a period of no longer than 1 minute. It is recommended that an arbor press be used with rubber pressing plates having a durometer between 45 and 95. These factors will vary, depending on the individual application. After separation, the parts will have a sharp definition of the outline of the part and will separate only in the pressed areas.

Sealing

It is recommended that the following sealing operation be used:
1. *Start at room temperature.*
2. *Raise the temperature in approximately 1-1½ hour to the peak temperature of 900°C.*
3. *Keep temperature at 900°C for 1½ hours.*
4. *Cool slowly.*

Use a weight of approximately 10-12 ounces on the parts during sealing. Recommended surface finish for Quartz parts: 1600. No high polishing.
This sealing cycle gives only general recommendations. The time cycle, especially step 3, can be shortened in most cases without affecting the seal.[2]

Some of these things will make more sense as we go through the step by step for the cell and you will see how we deviated from these directions.

During this time, Rudy Schlott was also working with this tape and, in 1992, his procedure was published in *Fusion* under Questions and Answers. The method I have found to be the most successful is Rudy's as published.
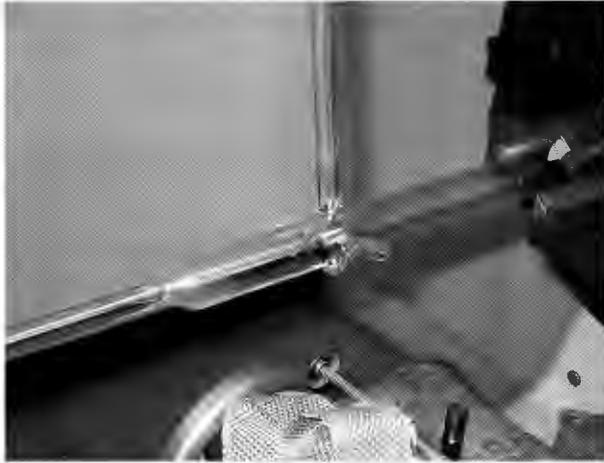
Figure 1

Figure 1 shows the cell body being constructed in the lathe. First a ring is blown up around the circumference of the tube, then the side tubes are sealed on. The large tube will go to vacuum. The two smaller tubes will be filled with sodium and any excess sodium will end up in the bubble at the bottom.
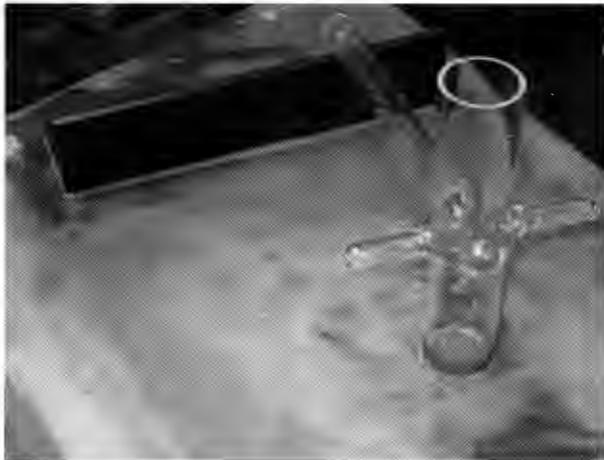

Figure 2

Figure 2 shows the tube being ground on a glass plate. In Rudy's directions, he says to grind it down to 500-800 grit. I did some experimenting with this going up to 800, but finally settled on 600 because that is what I had. You will notice that this is the first place I deviate from both the original directions that recommend grinding to 1600 and those of my original friend who polished the tube.

Figure 3 A & B

The next thing to do is cut a section of the tape material. Cut it a few millimeters larger than what will be needed. The material has a selvage edge (much like wallpaper if you have ever done that), so try not to go to the very edge. You will notice in Figure 3 A & B at the top, the tape did not separate cleanly. The tape consists of a waxy backing and a cellophane-like material that contains the adhesive material. If it does not separate cleanly as in this picture, this could be a sign that the material is approaching the shelf life limit or has passed it. Normal shelf life is nine months.

You will notice here that we are looking at a large rubber stopper; this is the work surface. I would normally have it covered with a clean Kimwipe. For the sake of these pictures, I have left that out. The tube is pressed against the tape (Figure 3C) using even hand pressure, another deviation from the instructions. I also like to cover the open end with a Kimwipe to keep hand oils off if I am going to put a window on the other end.
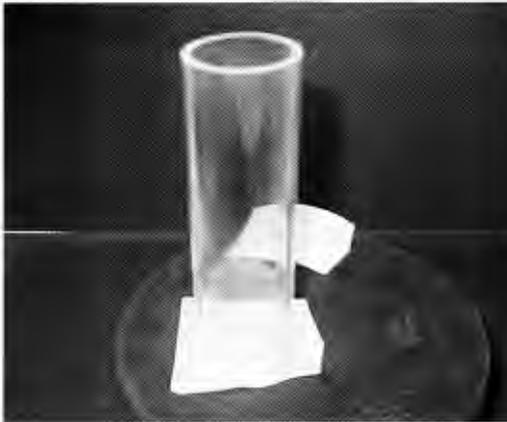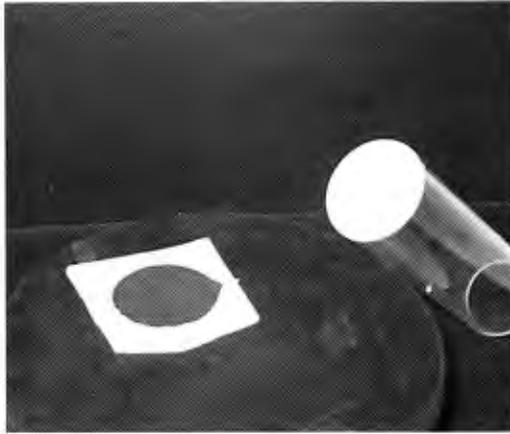

Figure 3C

Figure 3D

When you peel off the tape, you should have something like the picture in 3D. The rest can be trimmed off with a razor blade; I like to use exacto blades which you can buy in boxes of 100 at office supply stores. If the tape does not come off cleanly, wash if off with acetone; any crack in the surface of the tape will be a leak. Try not to get any additional flecks of material on the sealing surface, as these can also cause leaks. The blades themselves are hard to clean, so they become single use. And finally, even though this is going to fire on at a high temperature, if you get any on your clothes, it is hard to wash off and your clothes dryer will do a fine job of baking it on.

In the next figure (4), you will see the tube ready to be pushed to the window. Again, do this using uniform hand pressure. You can also see a couple of exacto blades in the picture. All of these steps are repeated to put the window on the other end, keeping everything clean and making sure you do not get trimmings from the second window on the first one. While I have been successful in pulling off a misaligned window and re-setting it with the same tape, I would recommend that you try and get it right the first time.

Figure 4

The cell is connected to a vacuum line (Figure 5) in the oven and a simple vacuum is applied, i.e. with a mechanical pump. The temperature is set to 1000°C and held for one hour. This is another deviation from the directions as they recommend 900°C for 1½ hours. Here Rudy recommends keeping the vacuum pump running until the temperature is below 500°C. Normally I run these overnight and then leak check them when I come in in the morning. Back to Figure 5: this is actually the look of the finished cell.
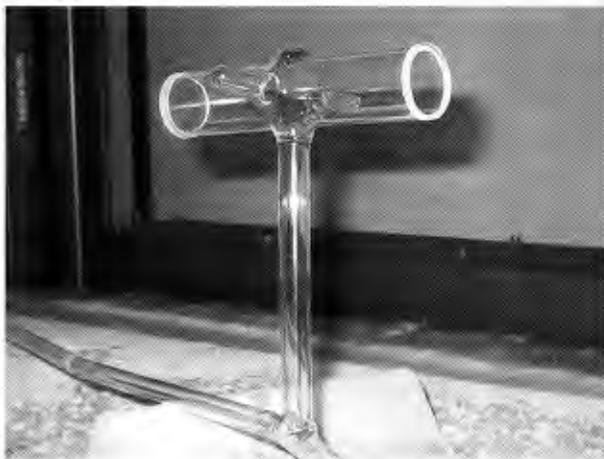

Figure 5

I am not involved with the sodium filling, as it is sent out to someone else. Insofar as extending the shelf life of the material is concerned, my original friend, Dr. Kosnick, placed it in a tube and pulled a slight vacuum on it. He said that this extended the shelf life, but I do not know by how much. If any of you look at Questions and Answers on the ASGS web site, you may have noticed the discussions on this last year and found that most people favored keeping it in the refrigerator, which is where my current batch has been for the last two years.

End Notes

[1] Dr. Ken Kosnick – personal conversation.
[2] Product Data Sheet, Bulletin G-05. Vitta Corp., 7 Trowbridge Drive, Bethel, CT 06801.
[3] Rudy Schlott, *Fusion* 38.1 (February 1992): 12-14.

The Glass Evaporator

by
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Abstract

I will discuss the use and construction of a glass evaporator unit designed for testing at the Savannah River Technology Center. The evaporator consists of three sections: 1) a modified kettle flask, 2) a three tier separator complete with overflow tubes, bubble caps and reservoir, and 3) a unique coil condenser. I plan to include all three units, with the main focus on the complexity of the construction of the middle section or separator. I will also describe the use and design of special fixtures needed to accomplish this task.

Introduction

Recently a chemical engineer came to me with an interesting challenge. It was to make a micro scale model, with a few modifications, of an evaporator presently being used on site. The evaporator would be used to study the reactions of certain chemical combinations and provide samples during the simulate test phase. This also provided engineers a unique opportunity to observe certain problems, such as foaming and scaling, which result from overheating and chemical breakdown of the solutions. With the simulate testing complete, the evaporator was utilized as a training tool for new operators. The test was captured on video, and has become an invaluable tool in providing a visual picture of the mechanics of the inside of the evaporator. Until now this was not possible. In addition to the visual capabilities, the most important objective was the ability to take and analyze samples during a test phase. This would provide valuable information and ensure proper simulate concentrations.

Bottom Section

The modified kettle bottom is made using a 4.5 inch flat o-ring flange, cut approximately 2¼ inches total length and sealed to a 10 inch section of 140mm medium wall borosilicate tubing. This is done by securing both pieces in the glassblowing lathe which is supplied with a lathe burner consisting of a minimum of eight fires and seven jets. During this step, the speed of the lathe would be used to expand and contract the glass seal, so a blowhose assembly is not required. The torches are supplied with fifteen pounds of oxygen and one half pound of propane. A gas and air annealing burner is used to slowly bring the two pieces up to temperature before making the seal. After approximately five minutes, the annealing burner is valved off and the lathe burners are ignited. Place the two pieces to be sealed close together so that the lathe burner heats both sections equally. Tool both ends to a 45 degree angle and seal. Once this seal is made and centered, seal the three side connectors, two #7 and one #15 internal threads, ninety degrees apart with a 15 degree pitch approximately

four inches from the bottom of the kettle. Once completed, flame anneal and allow the top to equilibrate using the gas and air annealing burner; this should take approximately five minutes. When cool, take the kettle out of the lathe and prepare to complete the unit. Secure the kettle in the lathe as before and round the bottom similar to a standard reaction kettle. After doing this, you have the option to flame anneal as in the previous step or place in a hot oven and anneal (Photo 1).

Photo 1

Middle Section

To start the middle section, make all of the internal parts first starting with the bubble caps. The bubble caps are constructed using 10mm borosilicate medium wall tubing cut 27mm long. A slot is cut in the top of the tubes ⅜ inch deep and ⅛ inch wide, approximately the width of a diamond cut-off blade. After completing the previous step, wash, dry, and fire polish the tubes on both ends and set aside for later use. Next take a two-foot section of 20mm borosilicate tubing and secure it in the lathe. Attach a blowhose assembly to one end of the 20mm tubing; the blowhose will provide control over the wall thickness and allow you to flatten the end cap. Once cooled, cut to 20mm total length using a cut-off saw, wash, and allow to dry. In preparation to sealing the slotted tubes, set the temperature of the annealing oven to approximately 500 degrees centigrade; the exact temperature during this step is not important. To seal the slotted tube inside the cap, use a small piece of quarter inch graphite plate with an 11mm hole drilled through it. This offers support during sealing and gives the proper depth below the cap that is needed in making the seal inside the evaporator unit. To make the bubble cap, place the slot end of the 10mm tubing above the graphite plate.

Then place the 20mm cap on top of the slotted tube. Use a small flame to slump the cap onto the tube when making the seal, such as a National Hand Torch with a #3 tip. When sealing the inner tube, pay special attention to ensure that no untouched or unsealed areas are present after making the seal. Place immediately into a warm annealing oven to prevent breakage. After sealing all of the bubble caps in this manner, run the oven through your normal annealing cycle (Photo 2).

Photo 2

Photo 3

Next you will need to cut two spacers, 20mm total length, using 22.2mm medium wall borosilicate tubing. These will remain open on both ends and, as before, wash, dry, and fire polish both ends before running through the annealing cycle (Photo 3). The last inside piece you will need is a 12.7mm medium wall tube with a maria push-up. Cut the 12.7mm o.d. tubing four inches and push-up a section 1¼ inches from the end of the tube. Once cooled, cut the top section ⅝ inch and fire polish both ends before running in the annealing oven (Photo 4).

Photo 4

The 4.5 inch flat ground flange, chosen for the bottom of the evaporator, will mate directly to the kettle bottom already assembled. To start, cut the flange 2¼ inches overall and set
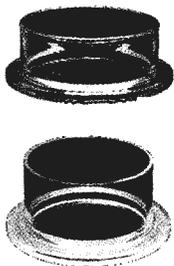
Photo 5

aside (Photo 5). For the sample ports, cut three 2mm Teflon stopcocks 1¼ inches and pull a 15 degree taper (Photo 6). The vents are made from three 10/18 outer joints cut 1¼ inches and also pulled to a 15 degree taper (Photo 7). Next cut two sections of 120mm standard wall borosilicate tubing 12 inches and fire polish both ends. These will be used to assemble the main body. After completing this step, run all of the pieces through the annealing oven to relieve stress.
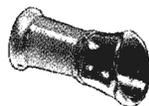

Photo 6

Photo 7

Photo 8

Photo 9

Before assembling the bottom tier, three special fixtures are needed: a 19/38 inner joint offset holder used to hold the open-end caps; a 12mm offset tube used to hold a 10mm graphite rod supporting the overflow tube; and the double bubble cap holder made from two 10mm shaker clamps incorporated into an offset glass holder (Photos 8, 9 and 10). This modified holder is used to hold two bubble caps at once, which ensures proper alignment and height while seals are being made inside the evaporator.

Photo 10

The lathe set-up for all three steps remains primarily the same throughout the assembly. On the head stock side of the lathe, a Litton HSJ six-jaw chuck is used along with graphite spools. The tail stock side uses an EC three-jaw chuck also equipped with graphite spools. The gas and air annealing burner is attached to a 12 inch Snake Support which travels with the fire carriage. The lathe burner, also mounted on the fire carriage, is an eight fire, seven jet assembly, supplied with 15 pounds of oxygen and one half pound of propane. Two hand torches used are: a National Hand Torch with a #3 tip and a Square Head Multi-Mix Hand Torch.

To assemble the bottom section, the 120mm tube is secured in the Litton HSJ six-jaw chuck along with the blow hose assembly. A scrap piece of tubing is chucked into the tail stock side and is used to seal off the 120mm section. The lathe burners are used to round the tubing while the blow hose supplies short puffs of air into the cylinder. This forces the glass against a flat carbon paddle to achieve a flat bottom. Once completed, the offset carbon holder is placed in the tail stock side in preparation for the next step. The carbon rod is placed close to the flat tube to gauge the location of the overflow tube. A National Hand Torch is used to heat a small section of the base to allow the carbon rod to form an impression in the glass bottom. Next, a hole is picked in the marked spot paying close attention to the size and shape of the opening. The bottom is heated again to maintain a constant temperature. The overflow tube is placed on the carbon holder and is centered in the hole before sealing into place. The National Hand Torch is used to seal around the maria while applying air through the main body with the blow hose.

After completing this step, the bottom is heated once more. The blow hose is removed and the 19/38 holder is secured inside the main body by using the inside chucks. Align the cup 180 degrees from the overflow tube, then seal into place using the National Torch. Heat the bottom section once more. Take out the 19/38 holder and reheat the bottom section while placing the two bubble caps into the holder assembly. Place the bubble cap holder into the main body and reheat the bottom section again. This will maintain a constant temperature on the flattened bottom and preheat the bubble caps to the necessary temperature. When aligning this section, center the caps between the two previous seals. Use the National Torch to heat both seals ensuring that all edges are touching. Use a small glass rod to pull the glass from the center of the bubble cap which will create a hole in the bottom of each cap. Flare the hole with a quarter inch carbon rod and heat the bottom section as before, repeating the above steps to seal the next set of cups in place. Place the ground flange in the tailstock side of the lathe and preheat for no more than two or three minutes. Use the lathe burner to seal the flange to the flattened section to create a ring seal. Next, pull a tapered hole 180 degrees from the overflow tube above the ring seal about the size of the flared stopcock. Reheat the bottom section for the last time by using the lathe burners and seal the stopcock at a 25 degree angle. Place in a hot oven, preferably 565 degrees centigrade, and anneal as usual. After annealing and allowing to cool, cut this section two inches measuring from the ring seal, and set aside (Photo 11).

Photo 11

The second section is sealed much like the first except for the addition of the 10/18 joint used as a vent. Place the 120mm tubing in the headstock and shape to a flat bottom as before. Seal the overflow tube, open-end cap, and four bubble caps as in the previous steps. Secure the bottom section completed in the first step in the tailstock. Begin by

Photo 12

Photo 13

preheating the outer edge of the tubing so as to slowly bring the ring seal up to temperature. Before sealing, make sure that the overflow is centered inside the open-end cap and that it is not touching the bottom as this will prevent the possibility of making the next ring seal. Once sealed, check the alignment of the tube to ensure that it is centered and that it is approximately half way up the cap. Pull a tapered hole below the ring seal 180 degrees from the stopcock which is located on the same section. Seal the 10/18 joint at 25 degrees and reheat the entire seal. Pull a tapered hole, as before, above the ring seal using the first stopcock as a guide. Seal the stopcock into place and reheat the ring seal. Place into a preheated oven, approximately 500 degrees centigrade, and allow it to soak for 10 minutes before continuing with the annealing cycle. After annealing, cut two inches, as before, and set aside (Photos 12,13).

The third section is assembled much like the first and second section, except that the 120mm tubing will be replaced by a 4.5 inch flat o-ring flange. Flatten the flange measuring two inches from the base of the flange. Repeat all of the steps in the

previous section and allow to soak at 500 degrees centigrade for 10 minutes before annealing. Once annealed and cooled, one final step is required. Take the completed unit and turn it upside down so the first section is in the upside down position. Place a 20mm cap on top of the slot tube and center. Place in the annealing oven and run a normal annealing cycle. Once the unit has reached 565 degrees centigrade, open the oven and seal the cap onto the slot tube. This should complete the middle section (Photo 14).
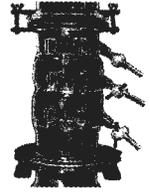
Photo 14

Top Section

To construct the top section, you must first assemble all of these necessary parts: a large glass coil, two #11 internal screw threads, a #7 internal screw thread, 2mm three-way Teflon stopcock, 4.5 inch flat ground flange, 16 inches of 120mm standard wall tubing cut straight and fire polished, and 12 inches of 38mm standard wall tubing.

First, you will need approximately seventy-two inches of 10mm medium wall tubing which will be used in making the coil. Then secure a three-inch o.d. stainless steel mandrel in the tail stock side of the lathe. Next, secure a one half inch o.d. off set glass rod on the head stock side of the lathe. Heat a four to five inch section of the 10mm tubing before starting the rotation of the lathe. As the lathe turns, apply constant heat on the tubing being coiled while maintaining a maximum spacing of 5 to 6 mm between each coil. After cooling, use leather gloves to remove the coil from the mandrel. Bend the bottom section to a right angle and the top to a vertical centered position. Cut the top section approximately 1¼ inches and flare. Gauge and mark the length of the bottom tube by comparing it to the inside diameter of the outside tube. Cut and modify the tube length before flaring to ensure a proper fit (Photo 15). Nex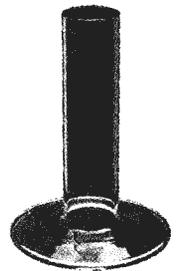t, secure the 38mm tube into the head stock chuck and flare to within 3 to 4mm from the inside diameter of the 120mm tube. Allow tube to cool before cutting and fire polishing to a total length of 6¼ inches (Photo 16).

Photo 15

Photo 16

Take the 2mm three-way stopcock and bend the top tube 90 degrees in the direction of the center tube. Make a push-up on the center tube and seal a small cup before bending 90 degrees resulting in a downward position. Cut the center tube one half inch below the bent section and fire polish. Use the cup as a gauge before cutting the top tube approximately 1½ inches and flaring. Cut the bottom tube as close to the stopcock body as possible and fire polish (Photo 17).

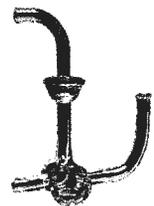**Photo 17**

Cut the 4½ inch flange to a total length of 2½ inches and set aside. Take a 30mm tube wrapped with corrugated cardboard and insert it inside the 38mm tube. This will be used as a holder during the ring seal step when connecting the flanged bottom. Secure

the 120mm tube into the outer jaws of the Litton six jaw chuck. Next, place the 30mm holder inside the outer tube by securing with the inside chuck jaws. Place the flat flange into the tail stock side of the lathe and secure. Preheat using the gas and air burner for no more than five minutes. Place the lathe burner under the outside tube and begin heating. Seal the inner and outer tubes together to form a Dewar seal. Next, position the lathe burners between the Dewar seal and the flat flange. Seal the two sections together and allow equilibration. Place the stopcock next to the ring seal to gauge the location of the holes needed to seal the stopcock into place. Pull both holes and reheat the ring seal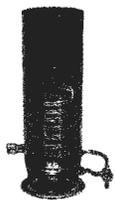 before sealing. Seal the stopcock into place and remove the 30mm holder from inside the 120mm tube. Then, place the coil inside the outer tube and hold in place using three equal sections of Hi-Resist tape. Align the bottom coil inlet 180 degrees from the stopcock sealed in the previous step. Reheat the ring seal before slumping the outer tube onto the coil inlet. Seal the coil into place and pull excess glass from the center which will form a hole. Seal the #11 internal thread to the coil inlet and reheat. After heating the ring seal, use a reducing flame to allow the seal to cool slowly. After proper annealing, allow the section to cool to room temperature (Photo 18).

Photo 18

Secure the top section into the head stock and begin to heat slowly. After heating for approximately 10 minutes with the gas and air burner, use the lathe burner to heat the outer tubing. When the proper temperature is obtained, shape the top section into a rounded bottom. Seal the coil's cente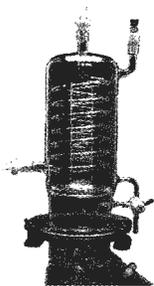r outlet in the middle of the rounded bottom and pull the glass from the center to create a hole. Flare the hole using a carbon rod and seal the #11 internal thread. Next, pull a small hole in the outer tube in line with the stopcock sealed in the previous step. Once again, heat the top section before sealing the #7 internal thread to the side of the outer tube. Seal the #7 thread and reheat using a reduced flame before cooling with the gas and air burner. Once it is completed, run it through a normal annealing cycle (Photos 19 and 20).
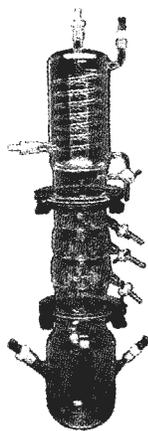
Photo 19

Photo 20

Acknowledgments

How I Fabricated a Right Angle Dewar

by
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Several years ago, I received a request from a UIC professor for a dewar that I had not made or even seen before. Explaining to me that he had used a dewar in the shape of a right angle where he previously worked, he informed me that it was indeed possible.

The obvious procedure I immediately imagined required immense firepower and skill to bend both the inner and outer tubes simultaneously. While I have floated many an inner tube of a vacuum trap and drip tip assemblies suspended by the ring seal for centering, bending the jacketed tube with and through its outer jacket just seemed beyond my skill and equipment levels. This was proven to me after I tried it. Bending a pair of tubes left me with nothing other than a twisted double wall of abstract glass sculpture. I had to come up with another method. In an effort to spark some creative solution, I bent a length of tubing and inserted half of it into a larger sized tubing and stared at it for a while. I was stumped and felt that I had reached a sort of glassblowers' block as do writers. Finally, I decided to solicit Joe Gregar's input. Today, of course, I have the high-tech option to go on-line to the Q & A mail list and solicit the entire membership's knowledge and advice. Before I reveal what Joe had suggested, I also feel I learned why I may not have considered all the possibilities because of a sort of prejudice on my part. To elaborate is sharing the insight that I personally dislike machining glass. With my apologies to our abrasive suppliers, drilling holes, saw-cutting and grinding seems to be brutal treatment of my favorite material. Forcefully breaking away fragments, sometimes chipping and cracking, accompanied by noise and liquid coolant just does not compare with my controlled coaxing of the hot molten glass coordinated ambidextrously with the forces of gravity. Perhaps this viewpoint is a basis for why so many people consider our practiced, developed skill, an art.

Now for a solution to fabricate a dewar around a corner. Saw-cutting the outer tube at a 45 degree angle and fusing the halves together over a bent tube was a viable possibility.

The larger diameter tube was cut at a 45 degree angle, and the *abused* ends were acid cleaned and fire-polished. A round bottom was shaped to length on this smaller diameter tube and then it was bent near a 90 degree angle.
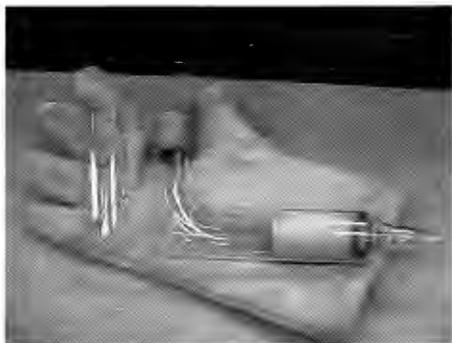
Picture 1

Picture 2

Picture 1 shows the bent tube supported inside one of the angle-cut outer tubes. I used an "Ace-type" elastic bandage here for packing. Regardless of how tightly this material is wound, it remains porous enough to blow air through it for the dewar end seal later. The end was roughly tapered down for a blow-hose connection eliminating the need for a stopper that could fall out at a most inopportune moment. Also shown here is some blue braided fiber, heat-resistant material supporting the other saw-cut leg.

The view in Picture 2 shows the cut angles matched up. Although they were a perfect match before I flared them, at this diameter my bends are not. At first I even used a ribbon burner but the bend was too gradual. The sharpest bend I could perform still left very little inside clearance. I brought a sample and a reject which illustrates this. The clearance was too tight and the outer tube made contact at the bend. I was unable to seal the entire circumference simultaneously, so I first partially tacked the fuse.

Picture 3

Picture 4

Then I began fusing the inside of the angle and worked the outer surface of the angle again (Picture 3). Having some gaps to contend with, I needed to resort to using a sharply pointed tungsten rod for complete contact of the circumference to close the seal and to establish inside blow pressure. I realize that using the tungsten rod is controversial and sometimes frowned upon (Picture 4). However, by adjusting the alignment, the gaps are small and I did not need to fill in the spaces by adding any glass with a rod, which I consider even worse.

Picture 5

Picture 6

I rounded the outer angle more, striving to eliminate sharp corners, working the seal more with the help of gravity (Picture 5), then finally back to the inside of the angle, evaluating wall thickness (Picture 6) until I eventually decided against attempting any further aesthetic improvement. Satisfied with the structural soundness of the seal, I removed the blue packing carefully and quickly with a pointed wire hook (Picture 7). I then moved to the lathe for the dewar end seal. This could certainly have been done in the ring stand, but not with my hands.

Picture 7

Picture 8

Flaring the inner tube up to meet the outer tube (Picture 8), I completed it by blowing it round also for structural strength (Picture 9). After flame-annealing (which occurred periodically without me repeatedly mentioning it), I scored and thermo-shocked the end open so that it made for easier removal of the elastic bandage packing with a better view of the pointed wire hook I mentioned earlier. A slight twist and this elastic material is out unlike the stiffer stuff (Picture 10). Perhaps there is a new product here for our suppliers or an entrepreneur in the group to consider: some sort of heat-resistant elastic hybrid cross. A disadvantage is that the bandage sheds a bit. You can see this in one of the rejects I brought as it was not cleaned. It also was not silvered for better viewing.

Picture 9

Picture 10

Making the Right Angle Dewar

Picture 11

Picture 12

In order to deliver, I had to make several of these. In one attempt, the packing loosened up and I would like to show you how I tried to salvage it. I wound blue packing on a tube of quartz and inserted it inside the bent tube (Picture 11). This allowed the flare to be supported (Picture 12) as the quartz is not softened by the level of heat that softened the lower temperature borosilicate glass (Picture 13). Picture 14 shows final fusing of the dewar end seal which is completed by blowing it round.

Picture 13

Picture 14

Cleaning and oven annealing followed. Chucking it back into the lathe and without setting up inside blow pressure, I pulled the side wall with a rod to thin it out and then opened it for the 18/9 ball joint tip-off tubulation to be sealed to the jacket (Picture 15).

Picture 15

Picture 16

Picture 17

Picture 18

To chuck the project into a 3-jaw chuck, I temporarily bent this tube away (Picture 16). The outer tube is collapsed and the final round bottom is formed (Picture 17) all the while monitoring the inside space between the two round bottoms (Picture 18). I had reservations about transporting an evacuated dewar so the sample here is not. The tubulation was then re-straightened, attached to a lab vacuum line for evacuation (Picture 19), and briefly flame-annealed locally at the tip off (Picture 20). The best method is to have a vacuum line inside an annealing oven, which my shop does not have. I have heard some people even fuse a small blob to the end where a pinhole could be.

Picture 19

Picture 20

When Jim asked me to present a paper, I simply could not turn him down when I remembered all the section meetings when he has helped me. Giving back to the Society, instead of just taking, is really quite rewarding. When I realized I could not reveal some ground breaking discovery, I decided that my objective was to share the alternative method I used to support the researcher who came to me for help. This bent dewar may well be a rudimentary procedure for many of the experienced and very talented people in this room, but if some of you picked up one minor tip from all of my work here, or if juniors not able to attend this Symposium learn by reading about this in the future *Proceedings,* then my objectives have been accomplished.

Why this shape?

As I understand it, the right angle dewar shape is ideal because when it is used, it is immersed into another dewar with nitrogen. The nitrogen flows out and across a microscope stage continuing to cool the sample even while it is being viewed. Another advantage is that the entire set-up is also portable; this allows the researcher to easily transport it across the lab. The purpose of the dewar was to hold samples of temperature-sensitive compounds under a cold, dry stream of nitrogen for mounting in X-ray crystallography studies. A "shelf" for the sample was fused inside the final instrument later.

I would like to express my appreciation to my employer, UIC, for support in this presentation. I must also thank my friend Pedro Bueno for his professional assistance and his dependability.

Improved Snyder Column

by
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It is often necessary to separate mixtures by distillation in various techniques. Distilling columns are commonly used for this purpose. The Vigreux Column, Oldershaw Column, and columns using various packing are all used for this purpose. This paper will focus on improving the durability of the Snyder Column. In the conventional Snyder Column (Figure 1), a vertical glass column is constructed having any number of constrictions around the circumference of the column forming a series of chambers. A hollow glass ball with a downwardly directed stem is deposited inside the column at each constriction forming a valve seat. As vapors of the various volatiles pass upward through the column, the ball valves will lift allowing vapors to pass from chamber to chamber. As the heavier fractions condense out, the resulting liquid will flow downward as the ball valves open. This process is known as fractional distillation.

One of the most common uses of the Snyder column is the Kuderna-Danish technique as a concentration method for pesticide analysis.[1] The Kuderna-Danish technique using the Snyder column is commonly used by environmental testing laboratories. These columns are used on a daily basis and there may be a dozen or more distillations taking place at the same time. In the regimen of daily use, cleaning, assembly, and disassembly, many columns are broken.

I have repaired several hundred Snyder columns. When one sees that many broken columns, a pattern of breakage becomes very obvious. The most glaring weakness of the column is that a constriction is used for the valve seat (Figures 1&2). Many times the column is snapped apart at the constriction. This occurs for two specific reasons:

1. The diameter of the column is reduced in size at this point.
2. The abrasive action of the bulb impinging the surface of the waist creates flaws on the surface of the constriction.

The combination of these two conditions creates an ideal area for a fracture. For example, when the Snyder Column is used in certain environmental testing applications, the EPA forbids the use of grease or Teflon* sleeves on the ground glass joints. In many cases this causes the joints to stick together. In an effort to separate the joints, the force applied on the column causes the column to snap at the constriction.

The next problem that occurs frequently involves the indentations that are in the Snyder column preventing the bulb from rising too high. They are typically made very pointed and thin. The upward action of the bulb, during the operation of the column, will cause thinly constructed indents to break (Figure 2).

The final problem exists with the bulbs. The bulbs are agitated in a very vigorous

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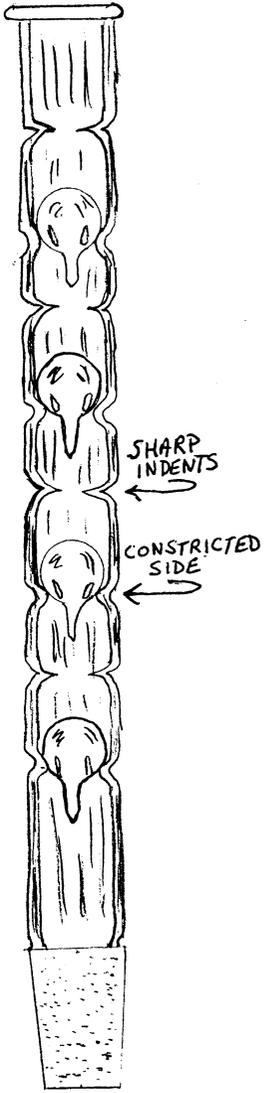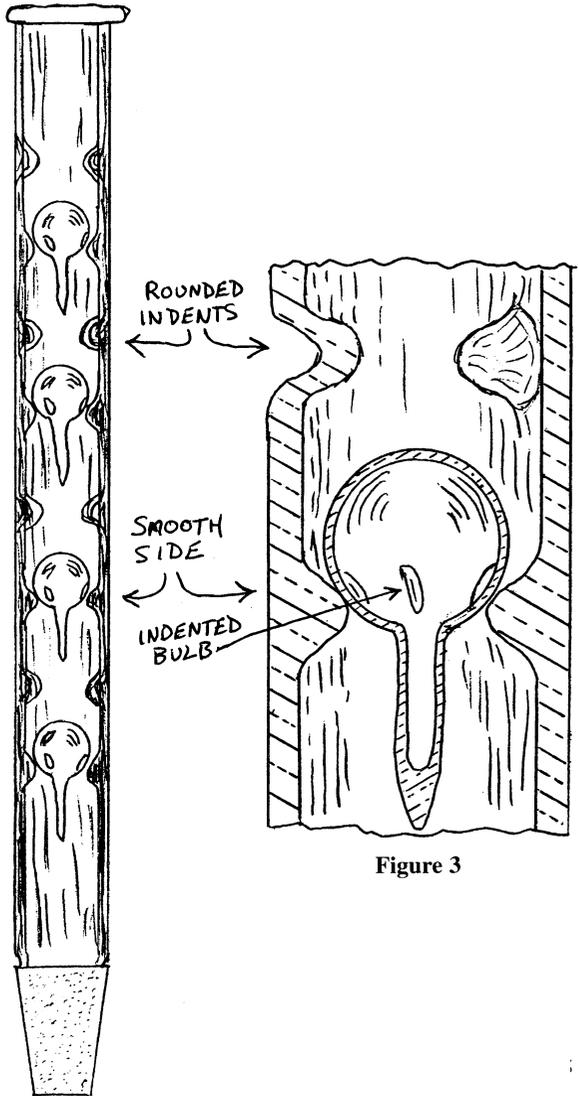* Teflon is a registered trademark of E.I. Dupont

ROUNDED
INDENTS

SHARP
INDENTS

CONSTRICTED
SIDE

SMOOTH
SIDE

INDENTED
BULB

Figure 3

Figure 1   **Figure 2**

manner by the up and down surges of liquid and vapor in the column. Many bulbs fail because they are simply constructed too thin. I have also seen many bulbs in Snyder Columns that have filled with liquid due to failure to seal the stem of the bulb properly.

To solve the problem of the weakness in the constricted portion of the column, I have designed the Snyder Column to maintain the same outside diameter throughout the entire length of the column. This is typically 28mm (Figure 3).

I create the valve seat by gathering glass on the inside diameter of the tubing where the seat is to be located. The glass tube is rotated in the lathe and heated with a torch at the site where the constriction is to be formed. The tailstock of the lathe is then slowly moved twenty millimeters toward the headstock. A flat carbon is laid on the rotating tubing while blowing into the tube to create enough pressure to produce a smooth surface on the outside of the glass tube.

When the newly fabricated seat has cooled enough to lose all of its red color the glass bulb is inserted in the tube. When the glass bulb is inserted in the rotating tube, the bulb will find its way into the seat. The process of creating additional chambers is done by measuring up the tube sixty millimeters making a reference mark and repeating the previous procedure for as many chambers as required.

Three equally spaced indents are required in the chambers to keep the bulbs from rising too high. I use a ¼ inch rounded end graphite rod to make the indent. Heating a spot on the glass tube half way between the valve seats and pushing the molten glass in slowly with this tool makes the indent much stronger than when pushed in with a sharply pointed tool.

To construct the bulbs, I use ¼ inch medium wall tubing. After closing and rounding off the open end of the tube, I gather as much glass as I can and blow the bulb up to nineteen millimeters. After the bulb is blown, three equally spaced indents are put in the area of the bulb that contacts the valve seat.[2] This will prevent the bulb from sticking to the seat during operation of the column. The bulb is sealed off from the ¼" tubing stem by heating about 15 millimeters below the bulb and pulling the bulb away from the tube. Do not cut the stem and seal the bulb by heating the open end with a torch. This may result in a tiny capillary being formed in the stem and the bulb may fill with liquid during operation.

The Improved Snyder Column with straight sides, thick walled valve seats, rounded indents, bulbs constructed with ample wall weight, and properly sealed off, will outlast the traditionally constructed column many times over. It takes additional time to construct this improved column, but it is well worth the effort.

End Notes

[1] Gunter Zweig, ed., <u>Principles, Methods, and General Applications</u>, Vol. 1 of <u>Analytical Methods for Pesticides, Plant Growth Regulators, and Food Additives</u> (New York: Academic Press, 1963): 95-96.

[2] United States Patent Number: 4,511,435. Date of Patent: April 16, 1985. Inventor: Rudy Strohschein.

Preparative Electrophoresis Chromatography Apparatus Operation/Design/Construction

by
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Abstract

One of the groups in our Chemistry Department needed an inexpensive method for purifying ribonucleic acid (RNA). Commercial models are available but they were prohibitively expensive. Electrophoresis gel chromatography is the method used for this process. The chemist and I worked in cooperation to design and construct a glass apparatus for this purpose.

This paper will provide an overview of preparative electrophoresis chromatography as it relates to an ongoing research project at the University of Wisconsin Chemistry Department. It will also cover:

- Background information
- The process of building, testing and refining the glassware used for this project

The glassware design went through three iterations. At each stage, the glassware was tested with a dye sample and modifications were made. The latest version (No. 3) has been successfully tested with dye but has not been run with a preparative RNA sample.

Background Information

A brief discussion of the fundamental chemistry involved will provide an understanding of the basic workings of the electrophoresis apparatus.

Chromatography is the general term used for any experimental technique used for the separation of organic compounds. Gel electrophoresis is one form of chromatography used for separating charged molecules. A gel column is saturated in a buffer solution between two electrodes. The molecules to be separated are placed on the gel and a high voltage is passed between the two electrodes. Positive ions will migrate towards the negative electrode and the negative ions will migrate towards the positive electrode.

There are two types of electrophoresis:

1) Analytical: The evaluation of molecular velocity vs. electrical charge of an unknown sample against a "known" sample. Similar samples will have similar molecular velocity for a given electrical charge. Analytical

electrophoresis is commonly performed as a thin layer chromatography between plates.

2) Preparative: This is molecular separation (purification) based on differences in molecular velocity vs. electric charge.

Preparative electrophoresis is the process we used for purifying RNA.

Molecular separation is influenced by the shape and size of the molecules, their electrical charge, and the characteristics of the gel through which the molecules are moving. The gel is a uniformly porous material and its composition will determine the porosity through which the molecules will flow. The gel can be thought of as a molecular sieve: certain size molecules are able to travel freely through the gel while different shaped molecules may still be able to work their way through the gel but at a slower rate. When the system is working properly you will get the separation of molecules into discreet horizontal bands in the gel. Two types of gels are commonly used: aragose and polyacrylamide. Aragose gel is used for long chain molecules. Polyacrylamide gel is used for smaller length molecules. Polyacrylamide gel electrophoresis is used for RNA purification.

No attempt will be made to discuss RNA chemistry other than mentioning the following two characteristics: it has a net negative charge, and it will absorb ultraviolet light. Commercially available RNA comes in two forms: pure product and a raw (preparative) product that requires further purification.

Building, Testing, Refining

A student came to the glass shop looking for help in building an electrophoresis device. He had seen commercial models available but these were prohibitively expensive for his needs. Knowing the underlying concepts and theory of the behavior of the molecules he was trying to isolate, we were able to design and build an original apparatus out of borosilicate and quartz glass for preparative electrophoresis.

Each prototype was tested with a negatively charged dye of two molecular sizes. The visual formation of two discrete horizontal bands and their behavior in the apparatus was the basis for evaluating the apparatus' performance. Based on this performance, modifications or adjustments to the design were made. The successful running of dye through the apparatus was used as a proof of concept before using the preparative RNA sample.
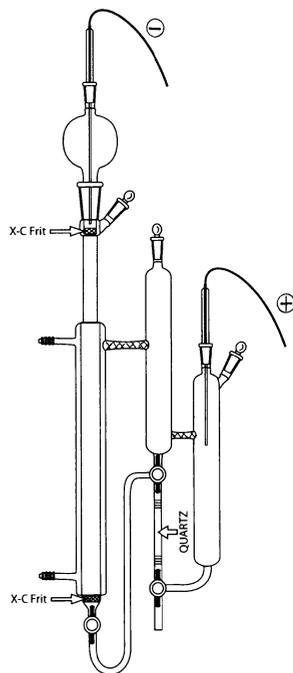
Design No. 1 is shown in Figure 1.

Figure 1
Electrophoresis apparatus,
version 1.

The intended theory of apparatus operation is as follows. Polyacrylamide gel is placed in a water-cooled chamber and polymerized. The preparative RNA sample is placed on the top of the gel. A buffer solution is placed in the 100ml flasks and reservoirs to saturate the entire system. The 3-way stopcocks are positioned so there is an open path between the two electrodes. The Electrodes are inserted. The power source (2000 volts) is turned on. The negative RNA ions travel through the gel towards the positive electrode and separate into horizontal bands depending on their size and shape. Since the sample is impure, several bands will form. A uniform temperature is required throughout the cross-sectional area of the gel to keep the integrity of the bands as they move down the column. The bands will continue to move all the way down the gel.

Since the RNA sample will not have a visual indication of its path, a method to detect its movement was employed using a technique called UV shadowing. Quartz tubing does not absorb UV light and RNA does absorb UV light. A fluorescent plate is placed behind a quartz section of tubing. A UV light in front will illuminate the plate unless it is absorbed by RNA. There will be a dark outline if RNA is present. Drain the sample. Continue until RNA is present again. These unknown samples can then be characterized with a mass spectrometer or compared with a standard reference molecule using analytical chromatography with polyacrylamide gel.

Glassware Construction

This is a straightforward construction with two areas of interest for the glassblower. These are the borosilicate-to-quartz seal and the borosilicate-to-platinum seal used for the electrodes.

Borosilicate-to-quartz seal: A 10mm diameter, 100mm length quartz section was sealed between the two 3-way borosilicate stopcocks. Schott sealing glasses 8228, 8229, and 8230 were used for the intermittent seals. Starting from the quartz end, wrap the 8228 rod around the 10mm diameter for three wraps. Keep the area hot but do not fuse in. Continue with the 8229 for three wraps and with the 8230 for three. Seal on the borosilicate tube and then fuse the sealing glasses. A No. 2 tip on a National hand torch was used for this. Starting from the quartz tube, a sharp flame was concentrated on the tube to soften and seal in the adjacent (8228) glass. This procedure was continued until all the glasses were sealed in and had uniform wall weight. This procedure was done using the lathe. See Figure 2.
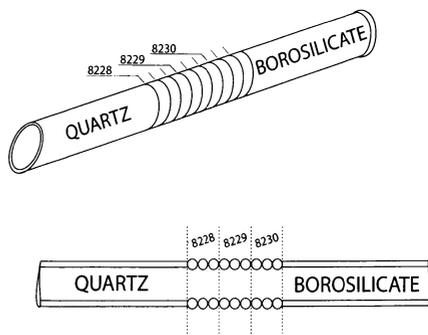

Figure 2
Quartz-to-borosilicate graded seal.

Borosilicate-to-Platinum seal: The two electrodes were made on the drip-tip ends of 10/30 standard taper joints with 6" drip-tip extensions. Since this is a non-vacuum application, a Housekeeper-type seal was used to seal 0.5mm diameter platinum wire

through borosilicate. The platinum was flattened close to one end. The borosilicate was tapered down with an opening to accept the widest part of the platinum. A pin vice was used to hold the platinum in the tailstock of the lathe. A small diameter carbon rod was used to paddle the glass onto the platinum. The glass was heated to make the seal complete. Remove the platinum from the pin vice and cut the platinum about a 7mm length from the end of the seal. A hot flame was used to ball the platinum up against the seal. Borosilicate rod was used to make three (3) protective legs around this platinum ball. Soft solder chunks were placed inside the tube. The electrical cord was inserted inside the tube and the soft solder was heated to melting to make the electrical connection through the borosilicate.

Running the dye through this apparatus revealed the following shortcomings of the design.

1) The large diameter inner chamber for the gel was creating a temperature differential across the gel (tubing) cross section similar to that shown in Figure 3.

2) The frit at the bottom of the gel chamber was making clean-up difficult after a run.

3) There was dye hold-up in the bottom of the bend up to the first stopcock.

4) Too much buffer solution was required for the positive electrode chamber.

Of these three items, the temperature variation across the gel was the most critical. This directly influences the purity of the sample.
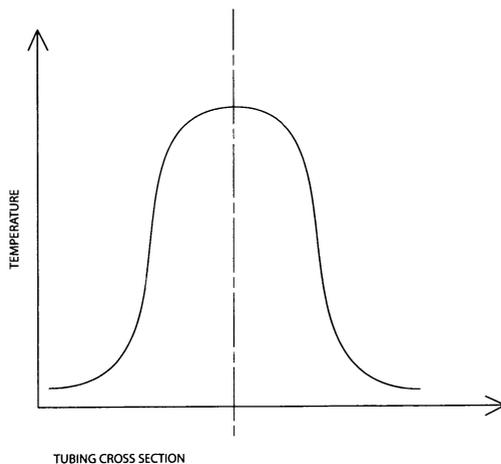

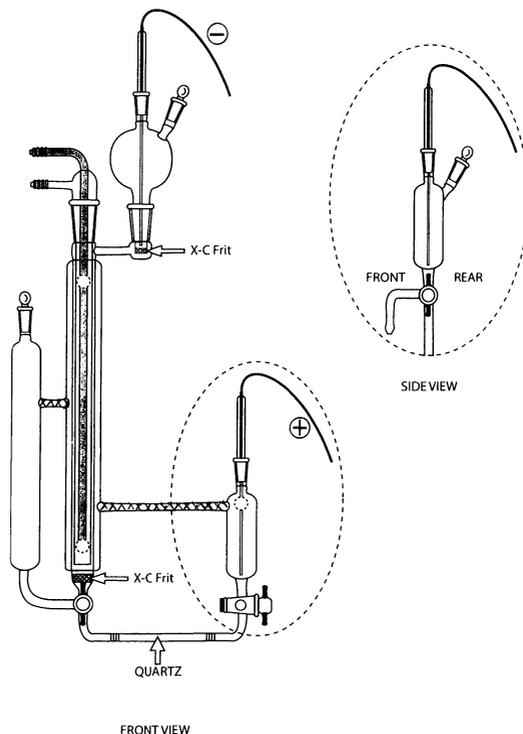**Figure 3. Cross sectional temperature variation of version 1.**

Figure 4. Electrophoresis aparatus, version 2.

Design No. 2 is shown in Figure 4. The changes for this version included:

1) Rebuilding the 300mm length condenser section to include an inner cooling finger. The 15mm (12.6mm i.d.) inner condenser tubing of version 1 was replaced with 22mm (19mm i.d.) tubing. A 16mm o.d. tube was used for the inner cooling finger. The gel gap was reduced from 12.6mm in version 1 to 3mm.

2) Lowering the gel column frit below the ring seal.

3) Repositioning the quartz tubing horizontally.

4) Reducing the volume of the positive electrode buffer solution chamber.

Testing this version showed that the dye moved in discreet horizontal bands, which means that there was a uniform cross-sectional temperature in the gel. We noticed that the frit at the bottom of the column was still causing cleaning trouble. The frit was removed and a side port through-seal was added. Also, the bands did not move uniformly once they passed through the 4mm 3-way stopcock and expanded to the 10mm o.d. quartz tubing. The quartz tubing was reduced to 5mm o.d. Design No.3 is shown in Figure 5. The side port will allow the addition of a high density liquid on which the gel can polymerize. Once the gel is polymerized, the high density liquid is drained and is replaced with buffer.
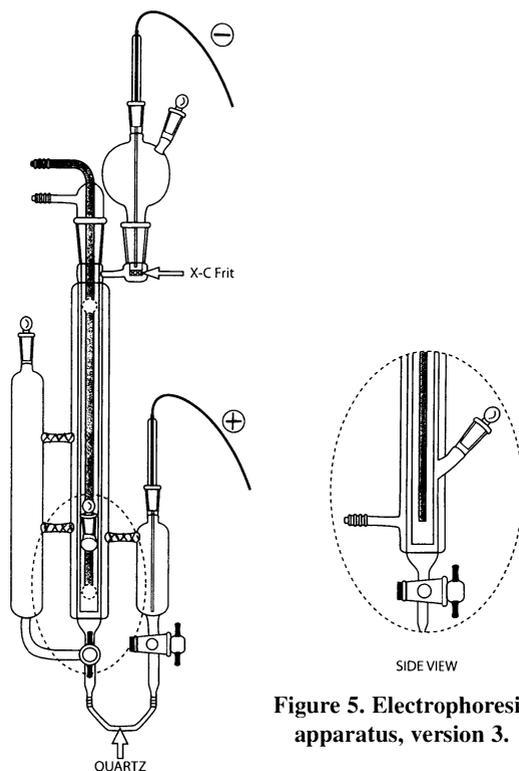
43

Figure 5. Electrophoresis apparatus, version 3.

SIDE VIEW

QUARTZ

X-C Frit

Construction details: The sequence for the small diameter tubing through-seal is illustrated in Figure 6. Two blowhoses are used. This third version was run successfully with dye.

Due to the ongoing modifications throughout this project, the final version, if made from scratch, would not necessarily be configured like this final version. Further changes could include a smaller volume of the positive and negative electrodes buffer solution, and the side joint at the top of the gel column could be constructed to facilitate the inserting of the needle for addition of dye/RNA. These changes are for convenience and do not affect the fundamental operation of the apparatus.

Conclusions

An original low-cost glass alternative for polyacrylamide gel electrophoresis was successfully run with a negatively-charged dye. The next step in this process is to verify that RNA can successfully be run through this apparatus. It is possible that testing with RNA may lead to further refinements.
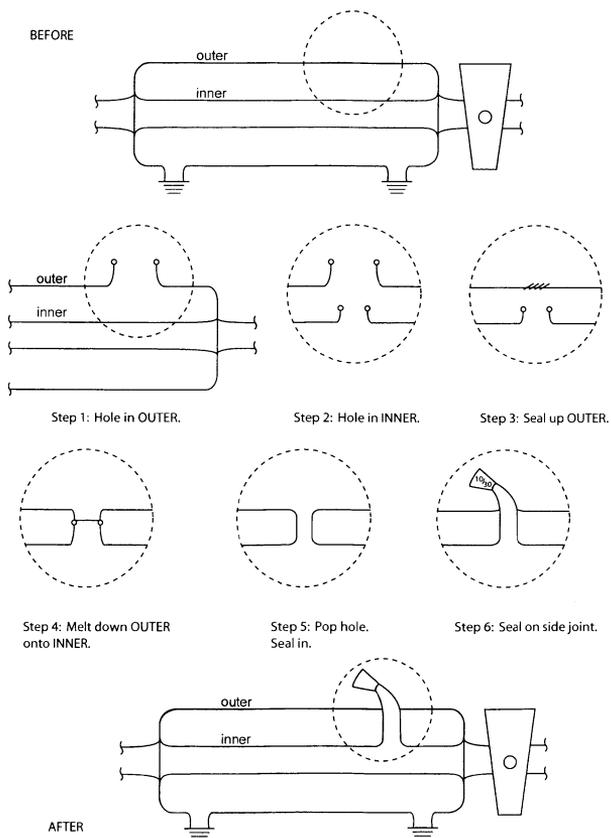
Figure 6. Through-seal technique.

Acknowledgements

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Unique Tools for the Glass Shop

by

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This paper consists of three unique tools for the glass shop. The first is a fixture for grinding circular windows. The second is an air gas torch used for preheating larger tubing or apparatus. The third is an air and vacuum supply made from a Ford smog pump; other smog pumps should also work.
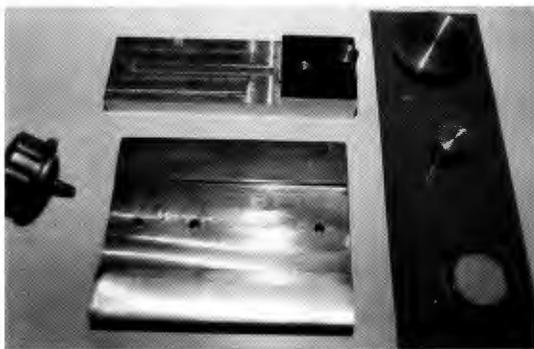

Photo 1

The first tool is a fixture for grinding circular windows (a detail drawing is shown in Figures 1 and 2). Photo 1 shows the four main parts of the fixture which are:
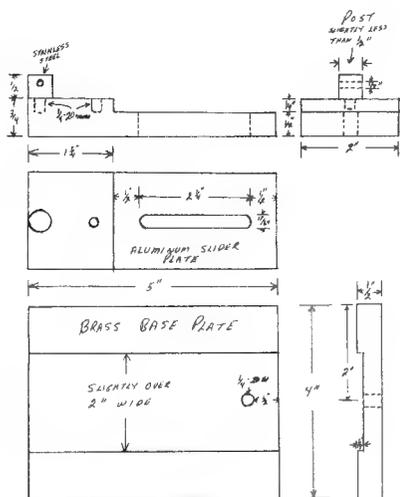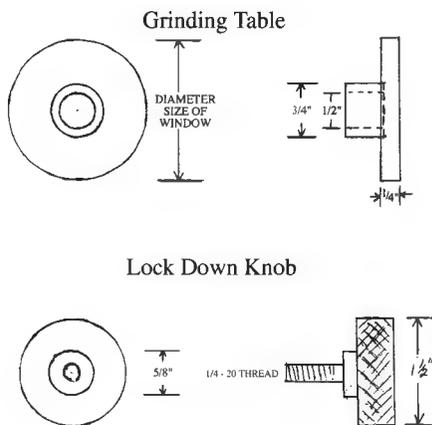

Figure 1

Figure 2

1. Brass base plate with a threaded hole for the stainless steel knob to screw into. The extra holes in the brass base plate are due to experimentation. Check drawing.
2. Aluminum slider plate with a stainless steel post. There is a second screw hole to reposition the stainless steel post for larger brass tables.
3. Stainless steel knob to hold the slider plate stationary.
4. Circular brass tables.

Photo 2

Photo 2 shows a rough cut window set up for grinding with the belt sander. The window is held in place with a thin layer of double sticky tape. A space should be left between the two strips of tape (which I forgot) in order to allow the insertion of a small spatula to release the window.

Photo 3

Photo 3 shows a completed window.

Photo 4

Photo 4 shows grinding a 45 degree bevel on the window. This will allow faster sealing of the window thus preventing warping.

Photo 5

Photo 5 is the completed window.

The second tool is an air gas torch used for preheating large tubing and apparatus. The torch is made from brass and stainless steel tubing.

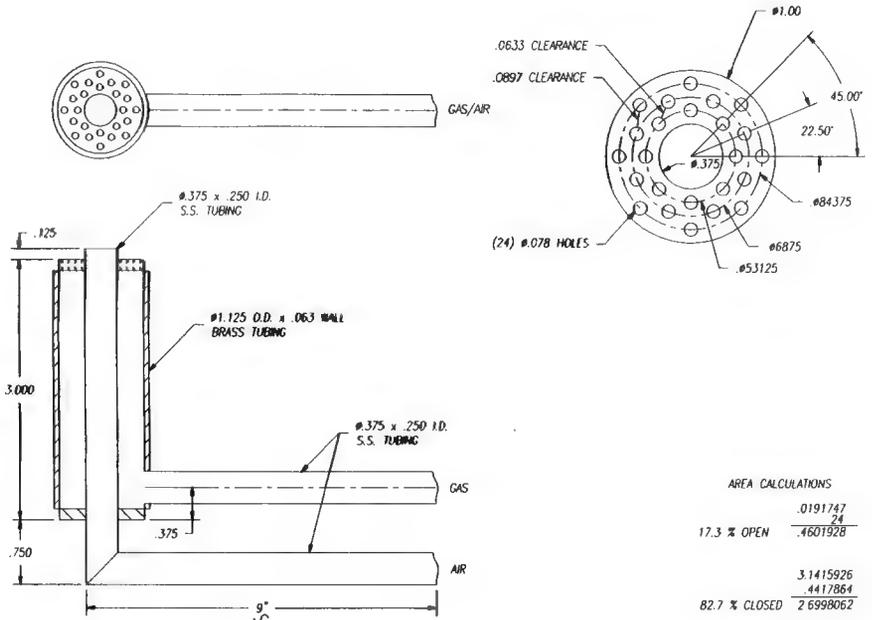Figure 3 is a detailed sketch of the air gas torch.

Figure 3

Photo 6

Photo 7

Photo 6 shows the torch burning only propane.

Photo 7 shows the torch burning a mixture of propane and air.

The third tool is an air or vacuum pump using a Ford smog pump. The smog pump will generate up to 8 pounds of pressure, and can also pull enough vacuum for holding a window in place while sealing on. The smog pump supplied the air for the air gas torch and all other torches in Photo 7.
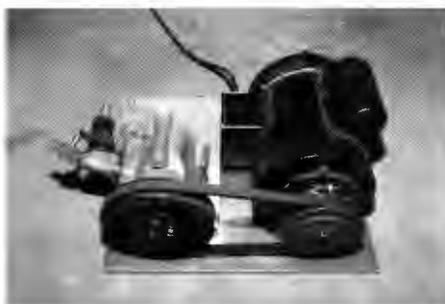

Photo 8

Photo 8 shows the smog pump mounted on an angle plate then fastened to a ¼ inch base plate. A 115 volt, ¾ horse motor is being used. It can be run in clockwise or counter-clockwise direction depending on if you want air or vacuum.
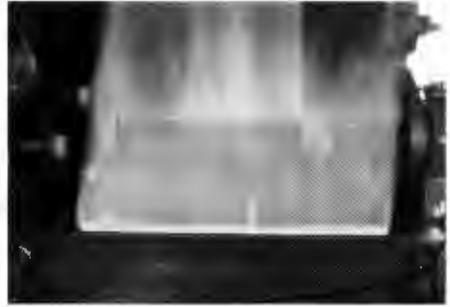
Photo 9

Photo 10

Photo 9 shows a veriflo hand torch with a number 2 air tip. The torch is burning propane and air. The air is supplied by the smog pump.

Photo 10 is a 16 inch ribbon burner operating with propane and air supplied by the smog pump.

Photo 11 shows Jennifer Partlow holding a 6 inch square by ¼ inch thick piece of plate glass with a 1½ inch o.d. vacuum chuck.

Photo 12 shows a 4 inch by ⅛ inch window being held by a vacuum chuck using vacuum generated by the smog pump. If you want to use a smog pump for both air and vacuum, get a reversible motor and wire it into a double pole double throw switch then to the on/off switch. Locate the switches so you do not accidentally change from forward to reverse while running.

Photo 11

Photo 12

The pump I am using is quite noisy. I am told that it should not be so noisy and that it may have bad bearings. If it is too noisy, put it outside and run a hose inside through a window.

I do not know if using the smog pump as a vacuum pump will shorten its life or have any other negative effects on it. Check it out for your own safety.

Up-to-Date Technology on Annealing Ovens for Quartz and Borosilicate Glass

by
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Programmable temperature controllers, heating elements, electrical power controllers, and new annealing oven designs: how they have affected the important process of annealing glass and Quartz.

In the early days of annealing, we were required to be a bit flexible because the technology of the equipment left a lot to be desired. Temperature controller accuracy would fluctuate, heating elements would burn out, and some annealing oven designs were quite cumbersome. This presentation will show how manufacturing technology has improved the required task of annealing.

Of all the technological improvements in this area, I feel that the most important would be in temperature controllers, and heating elements. Most would agree that solid state technology has played a major role in the advancement of the world today, and this is true for temperature controllers. Today's latest technology provides the capability of not only being able to program rate to temperature, soak, and rate of descent, but also the ability to log each run with a computer interface, easily change programs, and monitor and repair potential problems within the equipment before it becomes a problem. Many of today's manufacturers supply these products at very competitive prices, and I would advise shopping around to best suit your own personal preference. Although calibration of all temperature controllers is still very important, today's technology has also made this task more reliable and easier.

Heating elements for annealing Quartz reached a milestone in about 1985 with the development of a material called Molybdenum Disilicide. This has proven to work quite well under conditions of rapid heat-up and cooling conditions associated with the process of annealing Quartz. Other materials such as Silicon Carbide do work well, although they have not proven to be as economical and reliable as Molybdenum Disilicide.

Last but not least, annealing oven designs have vastly changed over the years along with the improvements in insulating materials and components to keep up with the demands of the industry. Instead of the front loading designs and the coffin type which are still extremely effective in some cases, the options of rotary hearth, vertical step conveyor, clam shell and bell designs have proven to be quite effective in today's world.

Violet Ray Devices
The Design and Fabrication of Glass Vacuum Electrodes

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Lightning has always fascinated mankind. In 1757, Benjamin Franklin flew his famous kite. In the early 1800's, Mary Shelley's story of Frankenstein postulated that its force might even restore life. It is not surprising that when it became possible to generate and control electricity, people sought ways to use it to improve health. In the early 1900's, violet ray[1] devices became quite fashionable for their purported healing properties. The following treatise will discuss their design and construction with an emphasis on the glass electrode portion of the devices.

History

There were a number of key discoveries and inventions that led to the development of violet ray devices. In 1748, Sir William Watson used an electrostatic machine and a vacuum pump to make the first glow discharge. His glass vessel was three feet long and three inches in diameter, the first fluorescent light bulb. In the mid 1800's, Heinrich Geissler, a German glassblower experimented with the discharge tubes that bear his name. About the same time, Heinrich Hertz demonstrated that it was possible to generate alternating current. At the end of the 19th century, Nikola Tesla sought to find a way to transmit electrical energy through the air using high frequency alternating current. One result of his experimentation was the development of the tesla coil.

A violet ray device is basically a combination of the tesla coil and a modified Geissler tube. They generate a high frequency, high voltage alternating current that is transmitted to the skin through evacuated glass tubes. The output might be described as a continuous static charge. Some of the original devices used hand held tesla coils similar to those we use to test for vacuum leaks (Figure 1). Others used larger high

Figure 1

[1] Violet Ray was the brand name of a particular unit, but violet ray came to be used as a generic term for the units.

Figure 2

frequency generators that could be used for longer periods of time without overheating (Figure 2). Most of the glass electrodes were evacuated tubes of various shapes, although some of them had metal feed-through wires.

There was a particular fascination with scientific inventions in the early part of the 20th century. Great advancements were being made in the field of electricity, and there was the general feeling that it was the destiny of mankind to overcome every obstacle. It is not surprising that the inventions of science would be turned to cure the human condition. As an example, the electrotherapy, or diathermy, provided by violet ray devices was claimed to be good for just about every physical ailment one might have from heart disease to hemorrhoids. The ozone created by the recombination of oxygen atoms into O_3 after the sparks tear apart the oxygen molecules in the air were thought to be good for purifying air and water. More recently it has been suggested that the ultraviolet given off by the sparks might have a sterilizing effect. Unfortunately there is only anecdotal evidence for any health benefits; no control group or comparative studies have been done.

Basic Design

The important features of the tesla coil are that it produces a high frequency (200,000-400,000 Hz), high voltage (30,00-50,000 volts) alternating current (Figure 3). This gives an output that resembles a static charge spark such as one might generate by rubbing one's feet on the carpet and then approaching a grounded surface, except that there is continuous sparking rather than a single occurrence.[2] The high frequency, high voltage also results in what is called the "skin effect;" the charge tends to run on the surface of the conductor or skin rather than through it.
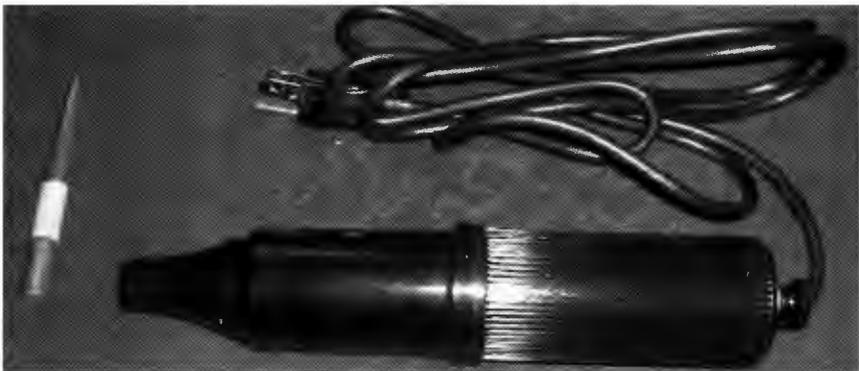

Figure 3

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[2] It takes 30,000-50,000 volts to cause a 25mm spark in air.

The glass electrodes serve as a means to distribute the charge produced by the tesla coil (Figure 4). The tube has a low-pressure gas in it that is ionized by the high voltage; this ionized gas then carries the current to the point of contact with the skin. The relatively poor conductivity of the glass diffuses the energy so that a single, hot spark that would burn the skin is not produced. The color of the discharge is determined by the type of gas in the tube.

Figure 4

The original electrodes were made of soft glasses (soda-lime, lead, and uranium) and they were evacuated until they could sustain a discharge. The gas inside was primarily nitrogen with some oxygen which gave a purple-white or violet glow. Some of the first tubes had feed-through wires, but most were just sealed, evacuated tubes. A few had inclusions of metal wool or ribbon. End caps were put on the end that plugged into the tesla coil with plaster. Electrodes came in many shapes, each particular contour designed to fit a certain portion of the human anatomy (Figure 5).
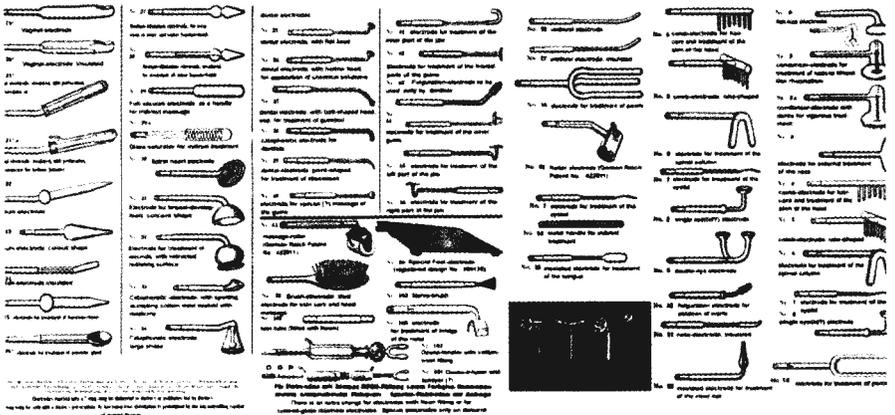

Figure 5

Design Considerations

Glass

The glass of choice today is borosilicate. The majority of us are proficient in fabricating shapes using it and familiar with its working properties. Soda-lime, lead (clear or colored), uranium, or most any other glass could be used since the glass itself does not have a major effect on the operation. Fused silica could even be used. The choice of a colored glass would affect the discharge color observed. Fused silica would be the only one that might let some ultraviolet radiation from the discharge escape the tube, but the amount of UV radiation generated is quite small to begin with.

Shape

The most important aspect of shape is the exterior contour. This is governed by the particular area of the anatomy to which the tube is expected to conform. Constrictions of the interior of the tube have the effect of making the discharge brighter in the constricted area (Figure 6).
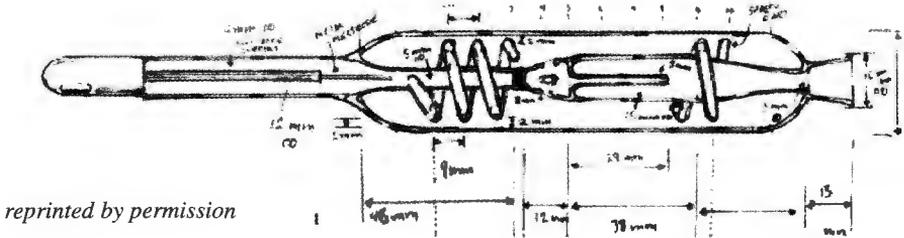

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Figure 6

Feed-through wires

Metal electrodes put into the tube have the effect of carrying the charge to a particular part of the interior of the tube giving a more intense glow at that place (Figure 7). The whole tube will still glow some, but discharge will be more concentrated between the end of the electrode and the glass that is in contact with the skin.
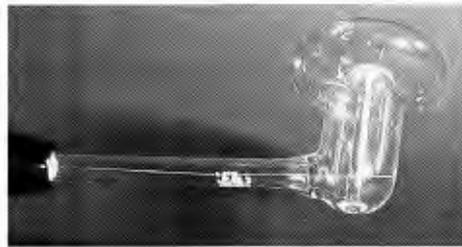

Figure 7

Inclusions

The placement of metal wool, ribbon, or foil into the tube has an effect similar to feed-through wires (Figure 8). There tends to be very little glow in the area of the foil or wool but very intense discharge from the metal to the glass in the area that is in contact with the skin. The feel on the skin is not noticeably different when inclusions or feed-through wires are added to the tube.
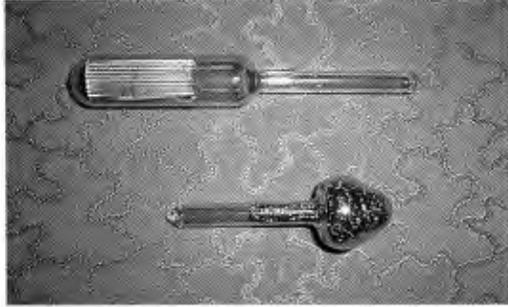

Figure 8

Interior Gas

The most apparent effect of the choice of gas is the discharge color: air gives a pale violet-white; argon is pale blue; neon produces a red-orange that appears as a more intense discharge since the human eye captures that particular frequency more readily. There are blends available from neon supply companies that can also be used. There is no reason other than cost and availability that would prevent one from using other gases.

Coatings

Coatings on the inside of the tube, particularly phosphors used in the neon industry, have the effect of altering the color and brightness given off by the tube. They also tend to produce a more uniform glow from the tube but obscure the internal discharge. Phosphors were not used in the original tubes.

End Caps

It is difficult to find properly-sized end caps today and it is prohibitively expensive to have them made in small quantities. Thin-walled brass or copper tubing is a possible option. Epoxy is a modern day option for adhering the cap. End caps have no particular effect on the operation of the tube and are only necessary as protection for the glass as it is plugged in and removed from the coil.

Construction of the Electrodes

Fabrication of the Tube

There is no unique trick to producing the glass for the electrodes. 11 mm tubing is the right diameter to fit most tesla coils and is the end that is eventually sealed off after filling. A constriction should be made at this point. Heavier walled tubing is better for

mechanical strength and does not affect operation. The working end of the tube depends on the particular contour desired and is formed as determined by that choice (Figure 9).

Figure 9

Evacuation/Filling

The electrodes are very much like neon tubes except that they do not normally have electrodes mounted in them. They are essentially processed like neon tubes with the exception that they are not 'bombarded'. The evacuation and filling station is designed from a typical neon filling station. A mechanical pump for the vacuum is sufficient, but a good high vacuum system is always better, particularly if you want to be able to more closely control the parameters for reproducible results or electrode research. The electrodes can be filled until the discharge 'looks good' when excited with a tesla coil or they can be filled to a specific pressure. The latter is better for quality control and repeatable results (Figures 10 & 11).
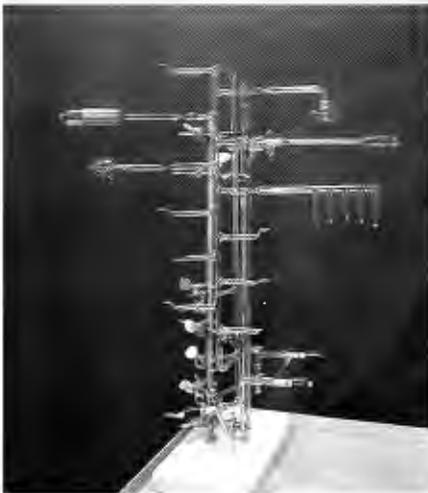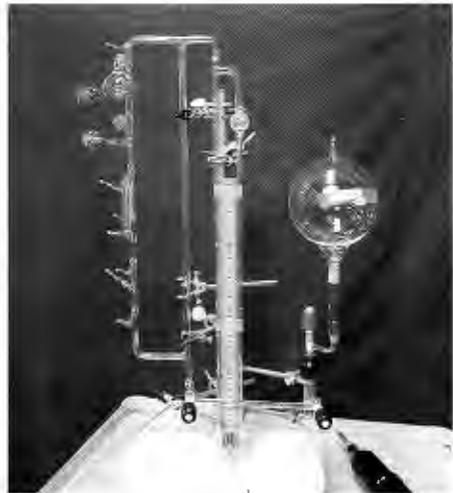

Figure 10

Figure 11

Filling Station Diagram Notes

Numbers 1-4 indicate stopcocks or high vacuum valves with teflon plungers and o-ring seals. #5 indicates a threaded o-ring fitting which may have either internal or external threads. The U-tube indicates mercury, but butyl phthalate or even silicone oil could also be used as in many neon fill systems. The U-tube is needed if you want to quantify the pressure in the filled electrodes.

Gas Flask Preparation

Many gases used in the neon industry can be purchased in one-liter flasks that come with a break seal. It is possible to get these flasks made of borosilicate glass, but the most readily available are made of soft glass. For the soft glass flasks, add a graded seal to the tube on the flask, place a 25mm length of 6-8mm rod in the tube and attach the valve. Open the valve and evacuate. Close the valve and shake the flask so that the piece of glass rod fractures the break seal (Figure 12). Connect the flask to the system using the threaded o-ring fitting. This approach is useful since it allows for changing gas flasks with a minimal loss of gas from the flask being removed or from the one being added.
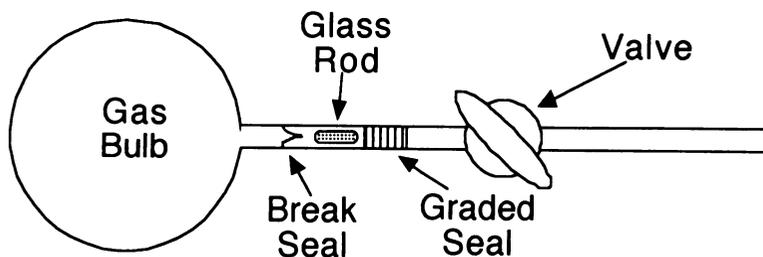

Figure 12

Basic Fill Procedure (refer to the filling station diagram, figure 13)

1) Open valves 1, 2, and 3. Valve 4 remains closed.
2) Seal the electrodes to be filled onto the system. (The electrodes can be annealed prior to this step to ensure that they are clean.)
3) Start the vacuum pump and allow the system to be evacuated.
4) Close valve 1.
5) Close valve 3.
6) Open and then close valve 4. (This introduces a small volume of gas into the area indicated by 'A'.)
7) Close valve 2 at this time if the pressure of the gas being introduced into the electrodes is to be measured.
8) Slowly open valve 3 to introduce gas into the system while monitoring either the discharge in the electrodes or the pressure using the U-tube. Close valve 3 when the proper amount of fill is achieved. If insufficient gas is introduced, repeat steps 5 & 6 and return to step 8. This approach of 'ladling' gas from the flask to the
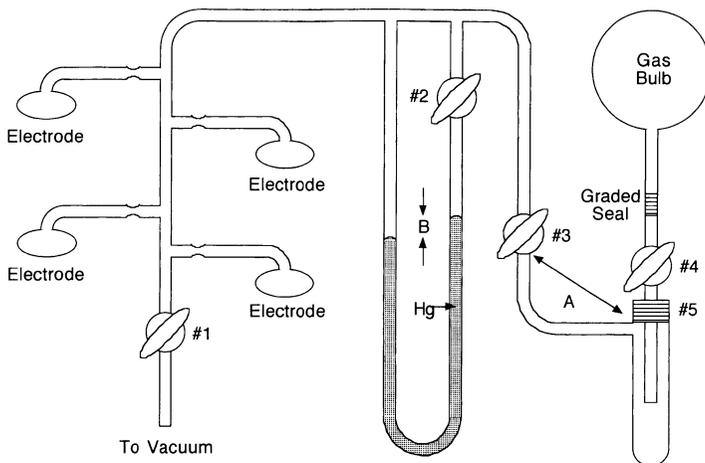
FILLING STATION DIAGRAM - figure 13

Figure 13

system allows for finer control of the amount of gas introduced into the electrodes, protects the gas in the flask from contamination, and reduces the amount of gas used in each fill process. (Normally pressures between 7mm and 20mm of mercury are good. 13mm of mercury as measured at 'B' works well and is the difference in height between the two columns of mercury. A meter stick mounted between the two legs of the U-tube makes this measurement easier to monitor.)

9) If the electrodes are properly filled, they are now sealed off at the constriction and annealed (Figure 14).

10) (Optional Flushing) To make sure that the gas in the electrodes is pure, it often helps to flush the system with the gas to be used one or two times before the final fill. This is more useful when using a mechanical pump. In a system using a good high vacuum pump, flushing would not be as important. Flushing is accomplished by the following steps:
   a) After step 6, leave valve 2 open and open valve 3.
   b) Open valve 1 and evacuate the system.
   c) Close valve 1.
   d) Close valve 3.
   e) Open and then close valve 4.
   f) To flush again, return to step (a). To do the final fill, return to step 7.

Post Production Problems

An obvious problem that develops with an electrode is that it looses its vacuum, usually from a leak. These are commonly the result of pin holes or cracks in the glass that can be repaired after which the electrode can be refilled. It is unlikely that out-gassing will be a problem since the vacuum is not that low and the pressure is not that critical. In addition, the electrode is not in a discharge situation for any extended length of time.
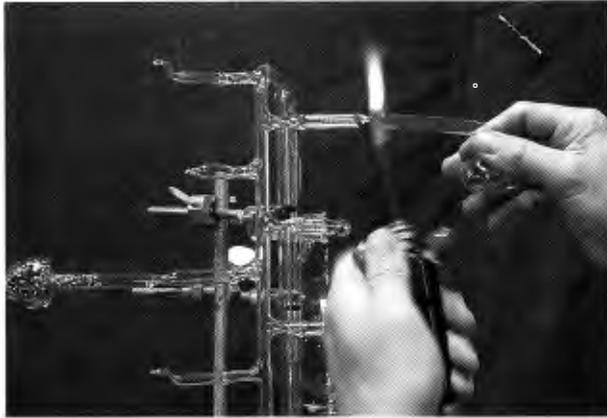
59

Figure 14

A less apparent problem is exterior dirt. These electrodes are in contact with skin and often used with other greases. These greases and skin oils coat the glass and interfere with their performance. An electrode will look fine but not support a discharge. Since these greases and oils are organic, a quick run through an oven 'ashing' cycle (400°C) or annealing cycle will restore them to original condition.

Conclusion

The production of violet ray electrodes is not difficult, nor does the filling process require stringent control. For the glassblower, there is a lot of room for experimentation with the design, the glass, and the fill gas. The possibilities are hindered only by one's imagination.

Further scientific research is required to determine their efficacy in curing or alleviating physical ailments. Well-documented, controlled construction of electrodes coupled with control group, comparison medical studies might answer the question of whether there is any benefit and, more importantly, the how and why of the benefit if it exists.

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James Klein
Gary Koopman
Georges Kopp
Jack Korfhage
Timothy Kornahrens
Charles Kraft
Jill Kraft-Santa
Tim Krantz
Barry Lafler
Andrew LaGrotte
Manfred Langer
Andrew Ledden
Jane Lewandowski
Robert Lewandowski
Donald Lillie
Douglas Little
Janeille Litton
Michelle Litton
Charles Litton, Jr.
William (Brad) Logsdon
Andy Lorenz
Mike Lozanoff
Ronnie Lymburn
John Macgown
Arnedo Malit
Glen Martin
Wilbur Mateyka
Victor Mathews
Larry McCollum
Elaine Meints
Frank Meints
James Merritt
Sharon Merritt
Patricia Miller

Dan Mindrum
Les Mintz
Keki Mistry
Steven Dean Moder
Arleen Molodow
Marvin Molodow
R.C. Monares
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Ned Morgan
Sabrina Morgan
Diann Morris
Michael Morris
Rita Morris
Roxanne Morris
Peter Moss
Eric Mueller
Danny Murphy
Thomas Murphy
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Peggy Nichols
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Diane Norman
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J. Norman
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Mary O'Brien
Tim O'Brien
Donald O'Brien, Jr.
Anne Marie O'Brien- Murphy
Patrick O'Neal
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Susan Olsen
Frank Ondrey
Kenneth Owens
James Ownby
Edward Padeway
Michael Palme
Jennifer Partlow
Joseph Partlow

Mary Partlow
Beth Paulison
E. Victor Pesce
Toni Pesce
Carol Peters
Jeff Petrie
Ken Petrie
Robert Platt II
John Plumbo
Lynnette Ponton
Melissa Ponton
Richard Ponton
Robert Ponton
Edwin Powell
Sally Prasch
Dolores Reppert
Jay Rice
Bill Roach
Monty Roach
Hans Rohner
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Ron Ross
Steve Russo
William Sales
Astrid Salvesen
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Carol Scalfani
Brian Schwandt
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David Searle
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William Showacre
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Jan Singhass
Sharon Skenandore
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Katie Smart
Gordon Smith
Richard Smith
Jeannene Smythe
Patrick Smythe
Michael Souza
Christopher Sprague
Thomas Stefanek
Dennis Steffey

Michael Stevens
Parker Stowman
KylerDean Strand
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Phil Surdam
Walt Surdam
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Kevin Teaford
Neal Trent
Ron Trent
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Jordan Vandenhoff
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Dennis Wargo
Jack Watson
Jim Weber
David Wedsworth
Patricia Wehoe
Peter Weier
Jeffrey White
Michael White
Mark Wicker

Daniel Witt
William Wilt
Kathryn Withrow
Donald Woodyard
Gregory Woolman
Fred Wright
Adam Yates
Christopher Young
Damon Young
Ryan Young
Tracy Young
Mohamed Younus
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