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Toledo, Ohio

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CONTENTS

THE BLASCHKAS AS SCIENTIFIC GLASSBLOWERS

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The Ware Collection of Glass Models of Plants, Harvard University's celebrated Glass Flowers, represent not only an elegant expression in glass artistry, but also an extreme in scientific accuracy. The more than 3,000 models, originally commissioned to teach botany, were created by the father and son team Leopold and Rudolph Blaschka. Tales of secret and fantastic processes circulated from the time the first models were exhibited in 1887, and have continued to this day. It is the intention of this paper to discuss some of the lampworking and jewelry making techniques used by the Blaschkas. There is no "magic" needed to make glass flowers. The Blaschkas employed standard techniques which continue to be used today; however, their understanding of botanical detail and their keen observations of natural history were paramount for expressing life-like forms in glass.

Figure 1: Rudolph (left) and Leopold (right) Blaschka standing in front of their home/studio in Hosterwitz, Germany. Circa 1895

Background

At the turn of the century, even a general education included botany, but finding flowering material to study during the New England school year was often problematic. The Director of the Botanical Museum, George L. Goodale, and the Ware family, who financed the undertaking - decided glass models were far better than the wax and papier-mache ones commercially available, and convinced Leopold Blaschka to make a few models of plants. The contract was extended through his son's retirement in 1936, 49 years later. By that time, nearly 780 species of plants, in 164 plant families, were created, each with a realistic life-size model, magnified cross and longitudinal sections of the fruit (ovary), and enlarged anatomical or morphological

sections of plant parts characteristic for that species. Together, the models illustrate the plant kingdom, including pollination by various insects, carnivorous plants, algae, fungi and plant diseases.

The Blaschka's were well educated and particularly interested in natural history. They used standard floras and other botanical references for the size ratios, along with fresh material or dried pressed specimens. Some common species grew in Hosterwitz, near Dresden, Germany where the Blaschkas lived and worked. Others were sent by the Harvard botanists as seed which the Blaschkas cultivated in their garden. They also had access to a greenhouse where more exotic & tropical species flourished. In addition, Rudolph traveled to the Caribbean and made two trips to the USA; he recorded the flora in elaborate drawings complete with the studies of plant parts, their relationships, as well as notations on color and surface texture. Interestingly, the drawings did not contain notes on assembly.

The Blaschkas as Practical Businessmen

Leopold had a definite philosophy about talent and training as evidenced in this 1889 correspondence.

> "Many people think that we have some secret apparatus by which we can squeeze glass suddenly into these forms, but it is not so. We have tact. My son Rudolph has more than I have, because he is my son, and tact increases in every generation. The only way to become a glass modeler of skill, I have often said to people, is to get a good great-grandfather who loved glass; then he is to have a son with like tastes; he is to be your grandfather. He in turn will have a son who must, as your father, be passionately fond of glass. You, as his son, can then try your hand, and it is your own fault if you do not succeed. But, if you do not have such ancestors, it is not your fault."

Combined with this philosophy and "tact" was their astute sense of business. As with any production work, their approach had to be practical. The Blaschkas used economically feasible materials and methods, often model parts were made from clear glass, probably single strength plate glass scored and cut to the approximate petal/leaf size, heated and then pulled into shape before adding base, apex and marginal details.

All like-parts were made at the same time (e.g. all the petals of similar species). On models where the fusion of delicate parts was precarious, the details were made separately and then glued to the body of the piece. For example, the spines on the cacti seem to have been pulled from what would become button-like bases and then adhered spirally to a cactus stem.

Techniques: A String of Beads & Coloring

It seems the Blaschkas's greatest innovation was the use of a wire support structure. Since their heritage included lampworkers, glass artisans, and jewelry makers, they were comfortable manipulating metal and glass.

To appreciate the technique, one must first visualize a string of beads where the string is made of a central wire and the beads are glass tubes of various lengths. The tubes form the stem sections (internodes), while each leaf or branch, fused at the base to its own copper or platinum wire, was wrapped around the central wire at the juncture between the tubes. In species having tendrils, fibrous roots, or leaves with stems (petioles), the structures are made of wire covered either with paint or glass and then attached.

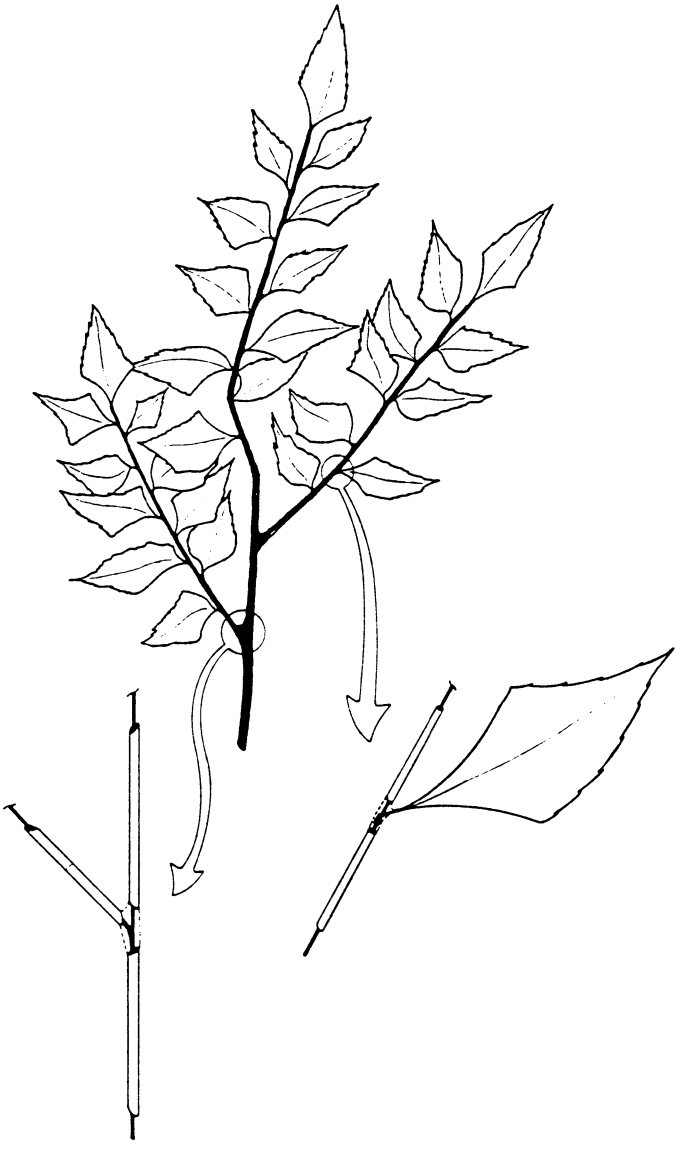

Figure 2: Exploded view of a fern leak showing the wire underpinnings and assembly (Illustrator, Wayne Myers)

In nature, the area where a leaf or a branch emerges (the node) is swollen; the Blaschkas took advantage of this. They disguised the attachments between the stem sections (probably filling it with frit) without concern for the nodal bulge, while cleverly adding to the model's realism.

The wire underpinnings seem to occur in all the assembled models, except a few early models which appear to be supported by a woody material. The resulting combination enables the model to withstand more physical stress than glass alone. This may have been especially important during shipment from Germany to the States by steamship.

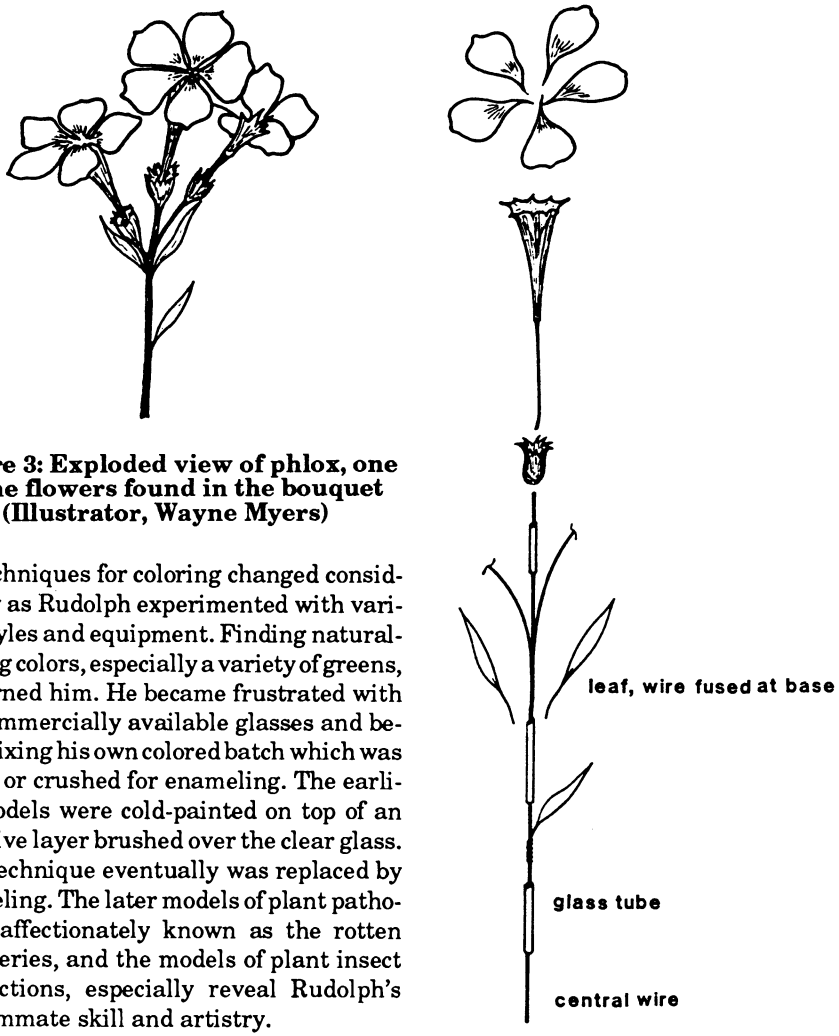

Figure 3: Exploded view of phlox, one of the flowers found in the bouquet (Illustrator, Wayne Myers)

Techniques for coloring changed considerably as Rudolph experimented with various styles and equipment. Finding natural-looking colors, especially a variety of greens, concerned him. He became frustrated with the commercially available glasses and began mixing his own colored batch which was blown or crushed for enameling. The earliest models were cold-painted on top of an adhesive layer brushed over the clear glass. This technique eventually was replaced by enameling. The later models of plant pathogens, affectionately known as the rotten fruit series, and the models of plant insect interactions, especially reveal Rudolph's consummate skill and artistry.

Conclusion

We are just beginning to appreciate the variety of techniques used by the Blaschkas to create the Glass Flowers. A number of conservation issues (too broad to delineate here) have become apparent over the years. A preliminary project, begun this spring, should determine the colorants, the adhesive material, and the glass types. Ultimately we plan to repair the damaged models and redesign the exhibit so visitors are given a fuller understanding of the collection, botany and glass as an artistic medium.

Credits

I would like to thank Dave Gover, Bill Ryan and Wayne Myers for their initial contributions, and particularly, Erich Moraine and Ed Mitchell for their continuing involvement and interest.

Figure 4: Bouquet given to benefactors by the Blashcka in 1889

THE TRAINING OF SCIENTIFIC GLASSBLOWERS IN THE U.K. IN THE 90'S

Michael Oliver
Glass Training Ltd.
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May I first wish you all at the Symposium a very happy and successful time. Unfortunately, I am unable to be present to deliver this paper, but, I am sure Mr. Mateyka will be able to express the contents equally as well as myself.

I would also like to take this opportunity to send to the American Society best wishes from the members of the British Society with a hope that you will go from strength to strength in promoting the art of Scientific Glassblowing.

In dealing with the changes envisaged in the future training of Scientific Glassblowers in the U.K., one must appreciate the impact which the European Common Market will have on U.K. manufacturing skills, particularly in the Glass Industry and consequently in Scientific Glassblowing skills. We must first acknowledge that a Common Market throughout Europe will bring fierce competition for the U.K., as well as Continental Markets and the British Glass Industry will have to be ready to meet this challenge.

The Government has already appreciated this of course and their concern has established the need for National Vocational Qualifications in almost all industries, where no formal qualifications in trades exist. This concern has resulted in the formation, in October 1986, of the National Council for Vocational Qualifications, whose purposes and aims are to achieve a framework for vocational qualifications that relate directly to a person's competence in employment. These qualifications and the standards associated with them are essential to the economic performance of the country and to individual job satisfaction.

One of the first jobs of the National Council has been to set up Lead Bodies and Working Parties for all aspects of Industry, including various working parties for each aspect of the Glass Industry. Here a working party has been formed to discuss and agree on the levels of skill in Scientific Glassblowing which will in future enable individuals, young and mature, to graduate from unskilled to highly skilled craftsmen. If they attain the necessary standard of competence they will receive a nationally recognised certificate showing the competences in which they have qualified.

It is at this point that I wish to mention the Lead Body for the Glass Industry in the U.K., "GLASS TRANING LTD." who have their headquarters in Sheffield. This training establishment has worked hard to organise and co-ordinate the various working parties in the Glass Industry and it is due to their efficient handling of problems and administration difficulties that the schemes have progressed so well. Their help and advice, which has been so freely given, has resulted in the Working Party for Scientific Glassblowing achieving first class results in determining the various levels of skills.

The Working Party consists of skilled representatives from the following areas: Scientific Glassblowing Companies, Research and Development, Education and members of both the British Society of Scientific Glassblowers (BSSG) and Glass Training Ltd.

This Working Party meets regularly to formulate and discuss the levels of skills required. Four levels have been formulated, ranging from elementary to highly skilled; the latter including further skills in the ancillary operations connected with Scientific Glassblowing.

At Level 1 the trainee glassblower will need to be competent in manufacturing a range of elementary glass pieces, for example, blowing round bottoms tubes, making connecting pieces in the shape of T pieces and Y pieces and making simple internal seals in the shape of Leibig and Alihns Condenser, etc.

Graduating through each level, the glassblower undergoing training will at Level 4 be a highly skilled individual who will have experience in various glass compositions ranging from soda-lime through to silica. The trainee will be able to manufacture both advanced and complex apparatus at the bench or at the lathe. He or she will also have experience in manufacturing methods in the ancillary operations of grinding, graduating, vacuum technology, advanced metal to glass seals and glass to ceramic seals.

To back up this competence in the work place will be further education in the area of Glass Technology associated with glass manufacture. Various further education centres throughout the country who offer courses in both practical and theoretical Glass Technology will handle this side of the training. An Open Learning Pack consisting of audio-visual material and information is made available through colleges as an alternative to regular attendance for those who need to study at home.

Qualified Assessors will undertake the assessment of various skills at each level. The Assessor will be a competent member of a company's staff following approval by the National Council. Skilled members of the BSSG will be asked to qualify as Assessors, as well as supervisors and key personnel in the various companies and further education centres. The four levels of skill and competency mentioned above have resulted in a Standard Manual of Training on which all competencies will be based and assessed.

It is envisaged that when a Glassblower graduates through Level 4, he will not only have a highly skilled knowledge of Scientific Glassblowing and its ancillary crafts, but, will also have the necessary skills to organise and plan the layout of a Workshop and manage its production.

Finally, the benefits of such a scheme over the coming years.

To the Individual Scientific Glassblower

At long last a chance to train and obtain a national award in Scientific Glassblowing. This can be obtained at each level from elementary through to highly skilled status. With the scheme being purely voluntary the individual may wish to obtain qualifications at a certain level only without advancing to the top level. This is permissible.

To the Company

Training with the Company and in further education centres will result in a highly skilled work force, which in turn will bring economies in production, increased quality and standards and enable the company to be more competitive; an increasingly important factor with the introduction of the Common Market in 1992.

To the Nation

This will enable the British Glass Industry - particularly in the production of Scientific Glassblowing - to be a highly skilled Industry, with the ability to hold its own in Continental markets where competition will be exceedingly tough.

EXPERIMENTAL METHOD FOR DETERMINING STRAIN TRANSFER EFFICIENCY IN EPOXY SEALING

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Epoxy sealing is sometimes seriously contemplated, even by glassblowers, and concerns emerge relevant to induced stresses especially when materials of dissimilar expansions are involved. An experimental procedure with a sandwich seal is described by which strain transfer efficiency of epoxies can be determined. Data are presented for two glasses differing in expansion by 54.5 x 10^{-7} /C joined by Uniset E-2510 epoxy.

1. Introduction

Conversations with scientific glassblowers have confirmed that not all sealing in the systems that they work with can be accomplished by hot glass fusion. Indeed, due to temperature limitations or other constraints, the glassblower must find another avenue for sealing. Epoxy is a medium that can be considered since it offers a strong bond that can be attained at a relatively low temperature.

The investigation report herein involves two glasses that were considered for bonding together with a thermal setting epoxy. Indeed, some product tests were conducted that resulted with a high percentage of breakage. A test sandwich seal was devised to evaluate stresses developed and effective strain transfer of the epoxy on the cooling portion of the thermal setting temperature cycle. It provides a simple, straightforward test format for evaluating sealing candidates, providing one of them is a transparent glass with a known stress-optical constant.

2. Study Glasses and Epoxy

The two glasses in this study are Codes 5390 and 7251 manufactured by Corning Incorporated. The physical properties of these glasses pertinent to the seal test are given:

	Code 7251	**Code 5390**
Elastic Modulus	9.2 x 10^6 psi	9.2 x 10^6 psi
0-300°C mean expansion coefficient	3.8 ppm/°C	9.25 ppm/°C
Stress-optical constant	0.262 nm/cm/psi	-

The thermal setting epoxy used is UNISET E-2510. The recommended curing is 15 minutes at 160°C (320°F).

3. Preparation of Test Seal

The seal requires three rectangular cross-section flat beams, 0.213 x 1.59 x 4.46 cm in size, two of Code 5390 glass and one of Code 7251 glass. Glass beams are prepared by diamond saw cutting and fine mill grinding. Both 0.213 x 4.46 cm surfaces of the Code 7251 beam are polished to facilitate the measurement of optical retardation.

Two strips of masking tape 1" wide are placed at the center on both sides of the Code 7251 glass beam with the tape edges running parallel the ends of the beam. Epoxy is then doctor-bladed to the four glass surface areas thus provided at the beam ends, using the tape thickness to control the epoxy thickness and uniformity. The two Code 5390 beams are then attached congruently with modest normal pressure. The final assembly is shown in Figure 1.

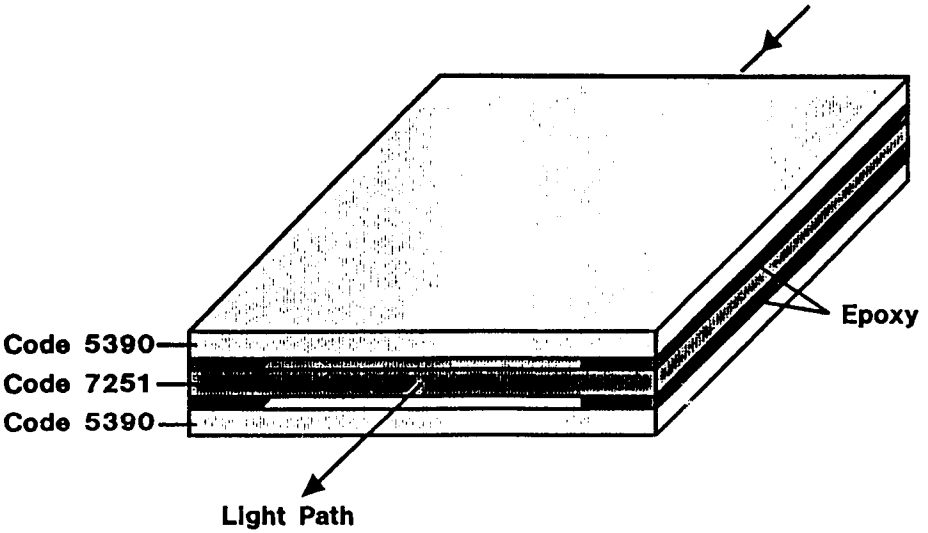

Figure 1: Epoxy Bonded Space Sandwich Seal

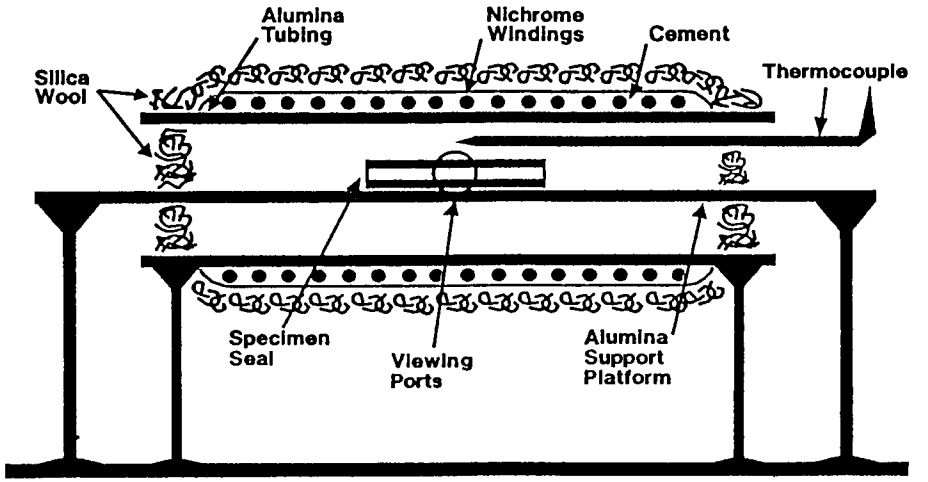

Figure 2: Low Heat Capacity – High Power Furnace For Photoelastic-Temperature Measurements

4. Furnace and Optical Bench

An optical bench with a furnace to house the specimen seal provided the necessary experimental apparatus. The optical bench has the required components for a Friedel polarimeter as specified in ASTM Designation F218, "Analyzing Stress in Glass". Filtered white light peaking at 522 nm is used.

The furnace is shown in Figure 2. It has nichrome windings for electrical power, controlled by the operator through judicious adjustments of an auto-transformer. With a little practice, very acceptable conformity to a specific thermal schedule can be attained. Due to high power, 1500 watts, and a minimum of insulation, provided

by a 1/2" layer of silica wool, the furnace is very responsive both on heating and cooling. Open viewing ports, 3/8" in diameter, were core drilled at opposite ends of a diameter in the alumina furnace tubing before wrapping and cementing the nichrome windings. The test seal is supported on a 1/8" x 3/4" alumina beam. A type S thermocouple, the junction of which is placed just above the center of the seal, is connected to a DORIC TRENDICATOR 400A with direct digital output in degrees C.

5. Experimental Procedure

The test seal is carefully centered in the furnace on the alumina support beam. The furnace is then powered to follow the epoxy curing cycle.

Optical retardation is measured at the center of the Code 7251 seal beam periodically through the thermal schedule and logged as degrees rotation of the Friedel polarimeter analyzer along with specimen temperature and time of observations.

Derived thermal expansion mismatch is calculated from the equation below:

$$\delta'_T = \frac{\lambda A}{180PK}\left[\frac{1}{E_1} + \frac{t_1}{2t_0E_0}\right]10^6$$

where: δ'_T + Experimentally derieved mismatch, ppm
λ = Wavelength of light, 522nm,
A = Friedel polarimeter analyzer rotation, degrees,
P = Path length (seal width), 1.59cm,
K = Stress-optical constant of 7251, 0.262nm/cm/psi,
E_1 = E_2 = Elastic moduli, 9.2 x 10^6 psi, and
t_1 = t_0 = Seal member thicknesses, 0.213cm.

Substituting values: $\delta'_T = 1.14A$

6. Experimental Results

Figure 3 is a plot of the thermal schedule attained for curing the epoxy and experimental determination of effective mismatch. Following the hold at 160°C, the power to the furnace was simply shut off and retardation readings were made during free cooling. During cooling compressive stress develops in Code 7251 glass and tensile stress in Code 5390 glass.

Resulting expansion mismatch data calculated from equation 1 are plotted on Figure 4. Three notable features of these data are:

(1) No retardation is present initially upon cooling from the hold at 160°C. This shows that the viscosity of the epoxy up to this point is low enough to permit full stress release.

(2) There is a significant slope change at 118°C. This is the transition temperature, Tg, analogous to the glass transition temperature found in the vicinity of the strain point. As with glass, the epoxy becomes more rigid below Tg, supporting more stress.

(3) The slope below Tg is linear, as expected, since the difference in expansion between the two glasses is a constant and change in the elastic properties with temperature is negligible.

During the data analysis, the writer found that a meaningful parameter can be abstracted, which is defined therein as effective strain transfer. A fully rigid seal will yield a mismatch-temperature slope equal to the difference in expansion between the two glasses. A lower value slope means that the epoxy is viscously and/or elastically absorbing strain. Expressed mathematically:

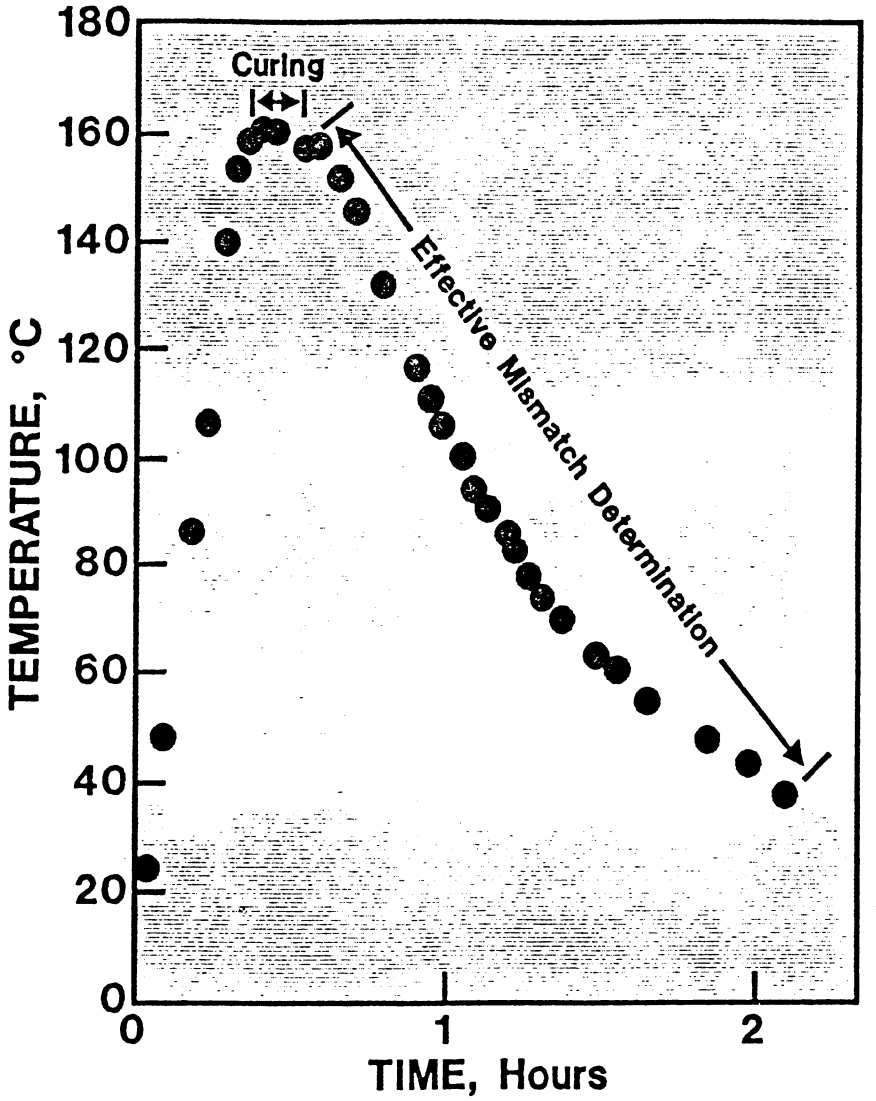

Figure 3: Thermal Schedule for Epoxy Curing and Effective Mismatch Determination

$$\epsilon = \frac{\frac{d\delta'_T}{dT}}{\frac{d\delta_T}{dT}}$$

where: $\frac{d\delta'_T}{dT}$ = Slope of experimentally derived mismatch — temperature curve, 4.85ppm/°C.

$\frac{d\delta_T}{dT}$ = Slope for full strain transfer, $(\alpha_{5390} - \alpha_{7251})$ = 9.25 – 3.80 = 5.45ppm/°C

$$\epsilon = \frac{4.85}{5.45} = 0.89\ (89\%)$$

Also shown on Figure 4 is the 100ppm mismatch level, generally imposed as a conservative limit for high reliability seals. It is notable that the seal fractured at a level above 400ppm before the seal reached room temperature. The fracture originated in the Code 5390 Glass, as expected (being in tension), at the inside junction of the epoxy seal. Obviously, UNISET E-2510 epoxy is too efficient for sealing these two glasses.

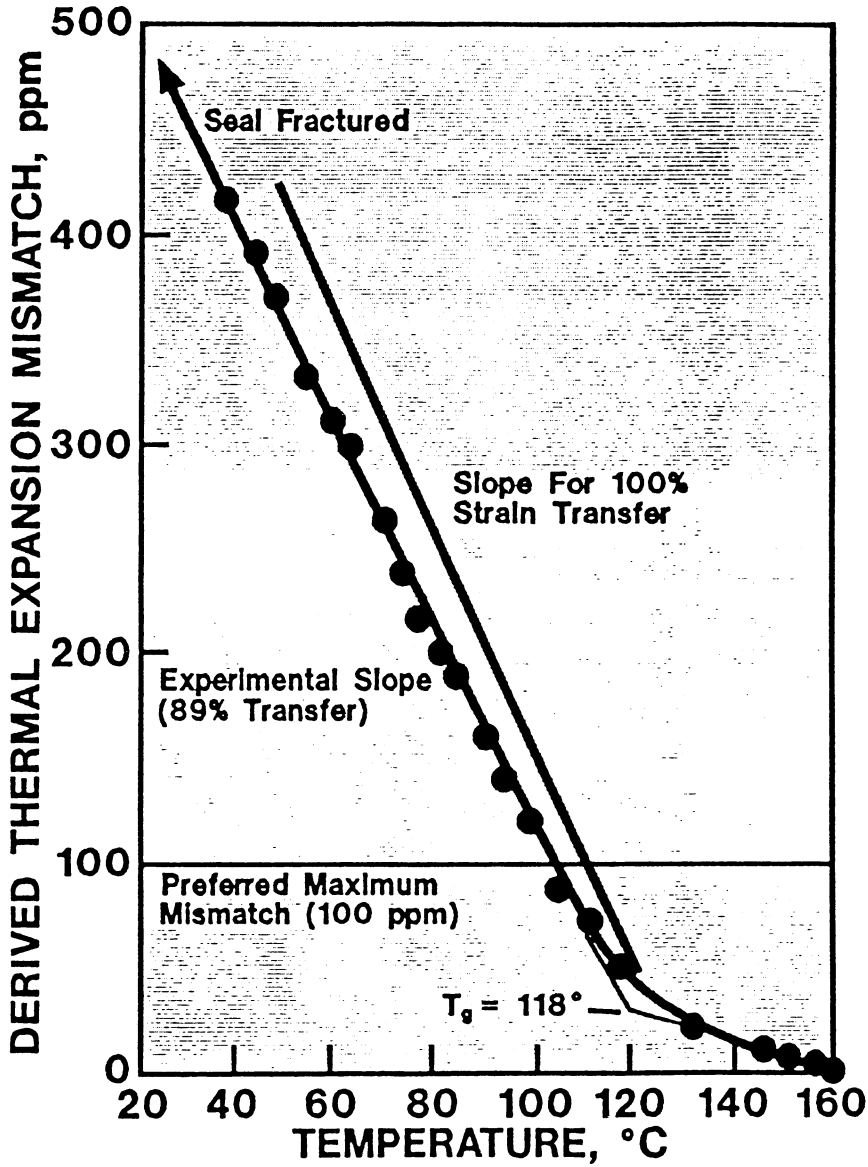

Figure 4: Photoelastically Derived Mismatch Vs. Temperature for Code 5390-Code 7251 Epoxy Seal

Following curing, the thicknesses of all epoxy layers were measured as 0.003".

7. Conclusions

A simple technique for evaluating strain transfer efficiency of epoxy resin cements has been described. A transparent glass with known stress-optical constant is required for the central seal member. Outer seal members may be other glasses, metal, or ceramic. Thermal expansions and elastic moduli must be known for both seal materials, but these are generally available from the manufacturers.

Strain transfer efficiency is important to thermal expansion matched systems, since stresses develop under use conditions which impose temperature gradients. For such an application, a seal can be made with the glass intended against a glass, metal, or ceramic of differing expansion. Expected stresses in the actual application due to temperature gradients can be used as limits during testing of the seal by limiting the temperature excursion of the seal.

It strikes the writer that epoxy manufacturers could use this technique for evaluating and qualifying compositions. Room temperature cured epoxies can be evaluated by heating and/or cooling a seal after curing.

For the case study presented, the epoxy was too efficient. These changes would have moved epoxy sealing towards more favorable conditions:

(1) Less expansion mismatch between glasses.

(2) An epoxy with lower Tg.

(3) An epoxy with lower elastic modulus and/or viscosity (creep).

(4) Thicker epoxy layer.

CONSTRUCTION OF A QUARTZ REACTOR WITH INTERNAL HEATER

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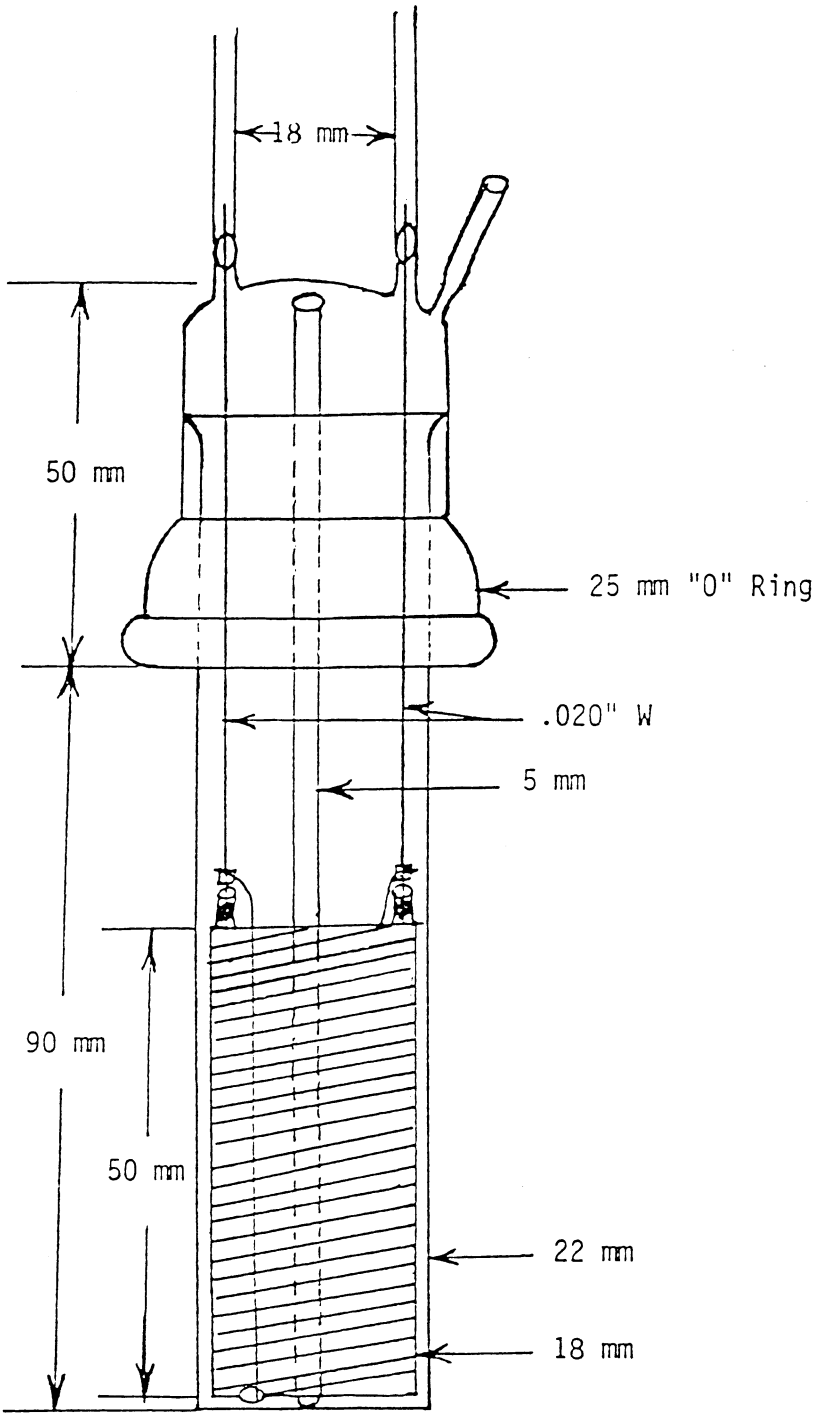
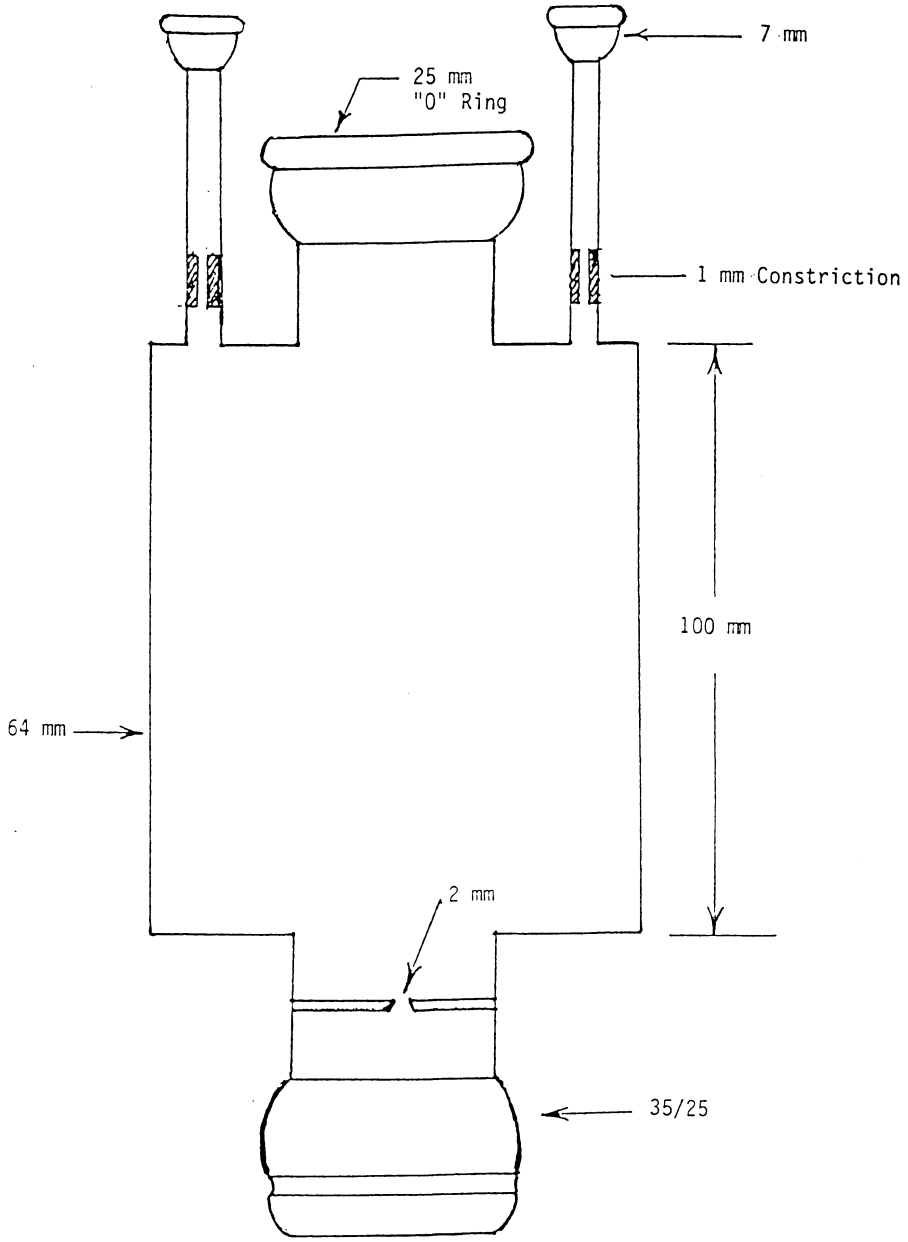
When constructing this apparatus, I started with the inner section first. That being the 18mm tube that the heater wire is wound on. I set the back stop of my wet saw at an angle that I feel comfortable with and then starting with a length of 18mm Quartz Tubing longer than the finished piece cut about 1/3 of the way into the tube and set my stop. At this time you are ready to begin. With a continuous motion, begin turning the tube against the wheel and advancing it at the same time keeping the grooves as close as possible without running over the previous one. It may help to practice your technique on Pyrex tubing first. Also I use a diamond wheel because you don't have to worry about the wheel flexing or the depth of the groove changing due to loss of material on the wheel. After I've grooved a section of the proper length it is cut off square and a small notch put in the bottom, into the first groove. This is then close by fusing on a small section of rod. At the top end fuse on 2 short sections of 5mm tubing 180° apart. These will accommodate the beaded Tungsten rods. I now cut the Tungsten rods to length and clean them. The beading is done in a lathe running at high speed using a hot flame, heat the Tungsten and with one motion as you see the oxide leave wipe on a coating of #1 grade glass. If necessary this bead may be built up to a larger size by heating and adding more #1 grade glass. A good seal will be bright and shiny. The rods are beaded at both ends. The bottom being beaded all the way to the end and the top leaving enough to clip on to. The beads are then sealed into the 5mm tubes of the cut section taking care to keep them parallel. We are now ready to wind the heater wire on. Through some trial and error we finally arrived at .008" Tungsten for the heater itself. One end is slipped through the small hole at the bottom, then up through the center tube and spot welded to the larger Tungsten rod using a small piece of nickle ribbon in between, after which the wire is carefully wound around the grooved tube taking care to keep it tight and then spot welded to the other electrode. This part is set aside and the 25mm "O" ring is taken up, this I seal off and blow a round bottom of the correct length. I then flare the 22 mm tube slightly so that it is a close fit inside the "O" ring. The next step for me is simplified because I have a Litton 6 Jaw Chuck, I hold the 22 mm tube in the rear Jaws and the "O" ring in the front Jaws to make the ring seal. After the ring seal is made, I seal on the two parallel 6mm tubes indicated here as 18mm apart for reference. The important thing is that they match the distance of the Tungsten rods on the heater, they are left longer than needed and the evacuation tube is sealed on. After cooling the heater is slipped in and pushed all the way up out of the way. You can now finish the bottom to the required length. The heater was then pushed down to rest on the bottom. With a little practice you can carefully score and crack off the 6mm tubes at the center of the bead allowing you to seal in the Tungsten rods. At this point the thermal couple tube is prepared. A hole is blown and it is sealed in, it may take some adjustment of the tube to get it to fit all the way to the bottom. The tube is now evacuated to a hard vacuum. The outer chamber was made by pre-making the 7mm "O" ring tube, here I used small sections of 1mm capillary tubing because the researcher wanted accurate knowledge of the size. The 25mm "O" ring was sealed to the 64mm tubing keeping it as short as possible and the 7mm "O" rings are added. Turning the tube around I closed the end to the proper length and sealed on a section of tubing the same diameter as that of the Ball Joint. After cooling it was cut off short and fire polished. The Ball Joint was closed and a flat bottom was made on the end.

The hole was made by first drilling a small hole in the center then drilling from the inside of the Joint. A tapered diamond drill was used until the proper taper was achieved and the hole was enlarged to 2mm. At this time the final seal was made by mounting in the lathe and using a ring burner.

In conclusion I would like to thank Jude Frances of the USC Chemistry Department, whose project this was and his advisor Dr. S. Benson.

(See following pages for diagrams)


18 mm
50 mm
25 mm "O" Ring
.020" W
5 mm
90 mm
50 mm
22 mm
18 mm


7 mm
25 mm
"O" Ring
1 mm Constriction
100 mm
64 mm
2 mm
35/25

HEALTH HAZARDS IN THE GLASS SHOP

G. E. Myers and J. S. Gregar
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Argonne, IL 60439

Since the beginning of the Occupational Safety and Health Administration, commonly referred to as OSHA, there have been fewer resources devoted to safety in the small workplace than to workers in larger plants and factories. Many glassblowers are among those who must educate themselves about safe work practices through special organizations and individual interest.

As glassblowers gain years of experience, they generally are instructed by a supervisor or co-worker to attain skills in glass cleaning and glassblowing techniques. Often it takes a few years until they develop their own very special and unique housekeeping practices. Many materials frequently used throughout our exclusive industry have recently been found to be hazardous. Many commonly used articles and chemicals found in the glass shop may not be safe even though you have used them for years.

As we started putting together a list of different items, it became so complex that it was soon apparent that we could not include everything in this talk. We decided to concentrate on a few of the more important concerns.

We will begin with solvents. All organic solvents are toxic to some degree. They tend to affect either internal organ systems such as the liver, or the central nervous system, or both.

In the work environment, the most common route of exposure is through inhalation of solvent vapors. The American Conference of Governmental Industrial Hygientists, or ACGIH, has formulated what it calls Threshold Limit Values, or TLVs, as exposure guidelines. The TLV for a substance is the airborne concentration, expressed as parts of vapor per million parts of air or milligrams of vapor per cubic meter of air, to which it is believed that most workers can be exposed, eight hours per day, five days per week, for a working lifetime, with no ill effects. Inherent in the TLV concept is the belief that the body can deal with hazardous materials, up to a point; in other words, there is a threshold for damage, both immediate and long-term.

OSHA has promulgated a list of Permissible Exposure Limits, or PELs. PELs are similar in concept to TLVs, and in fact the most recent PEL list was taken verbatim from the 1988 TLV list. PELs often are higher than the corresponding TLV, because TLVs are more up-to-date.

Skin contact is also a common route of workplace exposure to solvents. Some solvents can be absorbed through the skin in dangerous amounts, and nearly all organic solvents can cause dermatitis, that is, skin damage that can range from temporary irritation to long-term painful and even debilitating damage.

All solvents should be used with care, but there are two organic liquids which should NEVER be used as cleaning solvents. The first of these is benzene; note that this is b-e-n-z-E-n-e, C_6H_6, not b-e-n-z-I-n-e, which is another name for petroleum ether.

Benzene is a proven carcinogen which causes leukemia, as well as aplastic anemia. The OSHA PEL is one part per million, which is an indication of the severity of the hazard. The odor threshold for benzene is well above one ppm, so if you can smell it, you are exceeding the TLV.

Toluene and xylene and close relatives of benzene, but they are considerably less hazardous. However, be aware that commercial grades of these chemicals have been found to be contaminated with small amounts of benzene. Reagent grade materials are essentially pure.

The other organic liquid which should never be used as a cleaning solvent is carbon tetrachloride. It is a potent liver toxin which also is a suspect carcinogen. It has a TLV of five parts per million, which, like benzene, is lower than its odor threshold.

Trichloroethylene is a kidney and liver toxin which causes cancer in animals. Although it is less hazardous than carbon tetrachloride, it should not be used as a general cleaning solvent unless necessary, and then only with care to avoid inhalation and skin contact. Its TLV is fifty parts per million.

There are some relatively safe organic solvents. We reiterate that no organic solvent is COMPLETELY safe. Acetone is of fairly low toxicity, although its odor is irritating and it can cause dermatitis from prolonged or repeated skin contact. Ethyl alcohol also is of relatively low inhalation hazard. Isopropyl alcohol, which also is known as "rubbing alcohol," is more toxic than ethyl alcohol, but still safe to use with appropriate care. Methyl ethyl ketone, or MEK, also is safe for use with appropriate care even though it is more hazardous than isopropyl alcohol. Kerosene, painters' naptha, and petroleum ether all are of relatively low toxicity. Turpentine has a surprisingly low TLV, 100 parts per million, but it is of low hazard because it has a low vapor pressure. It is important to emphasize that we have been talking about toxicity — all of these solvents are flammable, so they present a real fire hazard.

If it is necessary to use a chlorinated hydrocarbon solvent, the safest choice is 1,1,1,-trichloroethane. This chemical also is called methyl chloroform, and it is sold under the trade name "Chlorothene®."[1] It is easy to confuse this material with trichloroethylene, which is much more hazardous, so make sure you have the right solvent before you use it.

In general, you should not rely on your sense of smell for warning that there are solvent vapors in the air. Odor thresholds vary widely between chemicals and between individuals, and we have seen that odor is not sufficient warning for highly toxic materials like benzene and carbon tetrachloride. Ventilation always should be provided when working with solvents.

Dilution ventilation is nothing more than the supplying of fresh air to an area, to dilute the solvent vapors to an acceptable concentration. Dilution ventilation is not a reliable hazard control, since a vapor concentration gradient always exists, and the solvent user normally is positioned in the highest concentration. In addition, dilution ventilation is inefficient and expensive, since large volumes of heated or cooled air must be exhausted to the outdoors.

The ever-present canopy hood is a poor device for control of solvent vapors. These hoods are designed to remove heated air that is rising, and have very poor capture of cooler air and contaminants. Furthermore, captured contaminants would have to rise through the breathing zone of the user in order to be exhausted.

Local exhaust ventilation is the preferred method for control of air contaminants. With local exhaust, air contaminants are drawn into the ventilation system at or near their point of generation, and before they can be dispersed into the room air. Local exhaust is an effective contaminant control, and it exhausts relatively little conditioned air. A chemical laboratory fume hood is ideal for working with solvents.

Although occasional brief skin contact with solvents probably will not cause harm, it should be avoided whenever possible, and minimized when necessary. Rubber

gloves are the classic method for preventing skin contact, but it is important to understand that glove materials must be chosen for specific solvents. Some solvents actually can diffuse through certain glove materials and be trapped against the skin, thereby increasing the hazard. This can occur with no visible damage to the glove. All reputable glove manufacturers and distributors can provide selection charts for their gloves, to help you pick the right glove material for the solvent you wish to use.

Mercury is a highly hazardous material that is commonly found in glass shops. Metallic mercury is hazardous only through inhalation of vapor, which is invisible and has no odor. Prolonged or repeated overexposure to mercury vapor can cause permanent nerve and brain damage. The TLV for mercury is 0.05 milligrams (50 micrograms) of vapor per cubic meter of air.

Freshly-spilled mercury can break up on impact into a myriad of tiny droplets with an enormous total surface area, and vapor generation can be significant. After a few days, surface contamination and oxidation may reduce vapor generation; however, any disturbance of these droplets will produce fresh surfaces and an increase in vapor generation. Mercury spill cleanup kits are available from a variety of sources. Spilled mercury should be cleaned up immediately. Mercury which has fallen into cracks or is otherwise inaccessible can be treated with a sulfiding agent such as HgX®.[2]

Glassware that is contaminated with mercury should be carefully cleaned before it is heated. One method is to rinse it repeatedly with nitric acid until all visible traces are gone. If a laboratory fume hood is available, it should be used for all work with mercury.

The increasing use of fused quartz in the scientific glass shop brings with it increased concern about silica exposure. Inhalation of silica particles can cause silicosis, a fibrotic lung disease that drastically decreases the victim's lung function. It is permanent and often disabling.

The respiratory system has numerous built-in defenses, most of which act to prevent particles in the inhaled air from reaching deep into the lungs. Large particles more than a few micrometers in diameter are trapped in the upper airways and removed when one "clears his throat." Particles smaller than a few micrometers are not trapped efficiently, and can penetrate deep into the most vulnerable lung tissues. Small silica particles thus can be deposited in the most sensitive parts of the lungs, and the result of repeated or prolonged exposure can be silicosis.

At one time it was believed that only crystalline silica was significantly hazardous, and that amorphous, or glassy, silica was far less harmful. We now know that crystalline silica is indeed more strongly fibrotic, but we have learned that amorphous silica is more hazardous than once was believed. Almost by definition, glassblowers work with amorphous silica. The TLV for crystalline silica is 0.1 milligrams (100 micrograms) of silica per cubic meter of air, and the TLV for amorphous silica is twice that, or 0.2 milligrams per cubic meter.

Silica dust can be created by cutting or grinding, but most of the particles thus generated are quite large and are intercepted by the respiratory defenses. Cutting and grinding should be performed wet to minimize dust generation, and all cleanup materials should be disposed of while still wet.

Silica working temperatures are so high that silica fume, an aerosol of extremely tiny particles, is generated. Studies have shown that glassblowers using canopy hoods are exposed to silica fume at concentrations normally below TLV, while those working without ventilation frequently are exposed to concentrations greater than TLV. This is one instance where canopy hoods are effective at reducing hazards. In order to be effective, canopy hoods (and, for that matter, ALL ventilation systems)

should be engineered and installed by competent, experienced personnel. All ventilation systems for the control of airborne health hazards should meet the design requirements of the ACGIH Industrial Ventilation Manual.

Asbestos once was a staple in glass shops, and unfortunately, it is still found in some. The aerodynamic behavior of fibers is dependent primarily on their diameter; in other words, a very thin fiber is more likely to penetrate the respiratory system's defenses than is a thicker fiber of the same length. Asbestos fibers are unique in that they split lengthwise into thinner fibers when they are torn or abraded. This is believed to be the property that makes asbestos so hazardous.

Asbestos is a proven carcinogen which causes both lung cancer and a rare, always-fatal cancer called mesothelioma. Asbestos simply should not be used at all in the glass shop. Fibrous glass and fibrous silica are far less hazardous because their fiber diameter is relatively large, and because their fibers break crosswise into shorter fibers of the same large diameter.

Ceramic fibers can be manufactured with a very small average diameter, and there are many who believe that small-diameter ceramic fibers may have a hazard potential similar to asbestos. Indeed, manufacturers of small-diameter ceramic fiber materials warn of the tumorigenic properties and suggest an exposure limit that is equal to the old OSHA PEL for asbestos. The use of small-diameter ceramic fibers should be avoided whenever possible. Ceramic fibers also can be manufactured with a large average diameter, and these are not believed to be significantly more hazardous than glass fibers.

Mineral wool, sometimes called rock wool or slag wool, contains some small-diameter fibers. Some studies have shown an excess of lung cancer in mineral wool workers. Mineral wool is, however, less hazardous than asbestos, since only a small fraction of the fibers are of small enough diameter to be of concern.

Organic material like Kevlar®[3] and Nomex®[4] are not known to be hazardous by inhalation. These materials are of large fiber diameter, and the fibers are soluble in body fluids so they do not remain in the lungs for long periods of time.

Less is known about carbon and graphite fibers, since these are relatively new materials. Recent studies have reported that the fiber diameter of these materials is sufficiently large that they should be of less concern than asbestos. There is at present no convincing evidence that these fibers are hazardous.

The glassblower who is confronted with the repair of a piece that is covered with molded-on asbestos sheet simply should refuse to do the job. If the piece is too valuable to discard, the asbestos should be completely removed by someone who is trained and equipped for asbestos removal. If the glassblower is incautious enough to perform the removal, he is then faced with a major disposal problem: the removed asbestos cannot safely or legally be disposed of in any easy fashion.

This has been a necessarily brief overview of some selected health hazards that may be found in a scientific glass shop. It is not intended to be complete, either in scope or in depth; rather, our intent was to alert you to some of the more common potential hazards that you may face in your daily work activities. It simply is not possible to tell you everything you need to know in these few minutes. We will conclude with some suggestions for sources of further help and information.

The OSHA Hazard Communication standard requires chemical manufacturers to supply Material Safety Data Sheets (MSDSs) with their materials. MSDSs can be helpful, but we have found them to be of variable quality: some are good, some are bad, and some are worthless! They are getting better, however. Newer sheets are likely

to be more informative than older ones, so be sure the ones you have on file are the latest. If possible, compare sheets from different manufacturers or suppliers, and follow the most stringent recommendations.

If you work for an employer that has an industrial hygiene department, consult with your industrial hygienist. Professional industrial hygienists are knowledgeable about health hazards and control measures, and they have access to the latest health information. Industrial hygienists who meet the educational and experience requirements of the American Board of Industrial Hygiene, and who have passed Board exams, are called "Certified Industrial Hygienists" and are allowed to use the initials CIH after their names.

The ASGS Safety and Hazards Committee is a valuable resource. Contact the Committee if you have health and safety concerns.

Government agencies are chronically overworked and understaffed, but they can be of valuable assistance. Check with your local or county public health department. If your state has an occupational health or environmental protection agency, call them. Your regional or local Federal OSHA office may be able to help you, at least with some information.

Finally, for those of you who must "do it yourself," there are several books which may be available in libraries.

The most well-known and comprehensive work is *Dangerous Properties of Industrial Materials* by Irving Sax.

Clinical Toxicology or Commercial Products by Gosselin and co-authors lists many of the hazardous ingredients in a large number of commercial products.

These two references are likely to be of the most practical use to you.

Patty's Industrial Hygiene and Toxicology, edited by George Clayton, is quite technical but can provide a wealth of knowledge for those who are willing to dig it out.

The National Institute for Occupational Safety and Health publishes a massive work known as *Registry of Toxic Effects of Chemical Substances*, which also is highly technical.

We hope that this brief introduction to common health hazards that can be found in many glass shops has given you a useful overview. It is important to realize that although many of the materials that we work with are potentially hazardous, they can be used safely if appropriate cautions are known, understood, and followed.

Footnotes:

1 Chlorothene® is a registered trademark of Dow Chemical Company.

2 HgX® is a registered trademark of Acton Associates.

3 Kevlar® is a registered trademark of E. I. du Pont Nemours & Co.

4 Nomex® is a registered trademark of E. I. du Pont Nemours & Co.

Thanks to Argonne National Laboratory's Environment, Safety and Health Division for their assistance in gathering information for this paper.

DEWARS I HAVE KNOWN

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This presentation is intended to show practical application and construction technique concerning specialty dewars. Building flexibility into specialty dewars allows researchers to use the same dewar - cells for IR, UV and, in some cases, electrochemical applications. It is my intent to show a pair of dewar - cells used by multiple groups for various applications (see fig. 1). Also covered will be a simplified method for constructing NMR transfer dewars.

For many of us, our first encounter with dewars was when we were in grammar school. Mom would put our soup in a "thermos" bottle and send us off to school. When we dropped that lunch box, there'd be a soft pop and soup leaked on the sandwich and cookies. Unfortunately, today's grammar school students carry plastic. As a consequence, our young people are denied this unique experience.

Dewars have been used in research laboratories for over one hundred years. Sir James Dewar, British chemist and physicist invented the dewar flask in 1872. His research into the liquefaction of the gases hydrogen and helium led to the development of this vessel. Figures 2 and 3 show examples of Dewar's early work. The main concept of the dewar flask has changed very little, it remains a jacketed flask evacuated and silvered. Modifications to Dewar's original design include the four walled liquid helium dewar and the addition of optical windows.

Construction of the thin film, low temperature Raman dewar:

This cell was built in two pieces using a number 50 "O" ring joint to join the halfs (see fig. 4). The over-all length of this cell is 12 inches. The dewared section is 7" in length and fabricated from 35mm and 45mm standard wall tubing. The bottom two legs of the inside piece are the "business" end of the cell (see fig. 5). 10mm standard wall tubing was flattened with a carbon paddle and then ground flat and smooth with 600 grit carborundum powder using a wet wheel. The liquid protein sample is held against this flat surface by means of an aluminum holder with two quartz plates pressed together. Between the two quartz plates is a 1/2mm thick teflon washer (see fig. 6).

The external windows were mounted with number 20 "O" ring joints. This was done so the windows could be changed to meet the needs of differing experiments. As shown the cell is utilizing commercial quartz in the windows and the plates holding the sample protein. By changing the windows and plates to suprasil quartz, borosilicate or KBR salt, this cell can be used for near and far UV in infrared work.

The multiple tungsten feed through portion on the top was installed to allow for electro-chemical applications not yet utilized. The stopcocks mounted on the top are used for evacuation and in some experiments for inert gas flow.

Construction of the high-low temperature IR dewar - cell:

This second dewar - cell has the basic same design as the low temperature Raman dewar (see fig. 7). It, too, was built in two pieces, this time using a 45/50 standard taper joint as the connection. This cell is used exclusively for IR work. The sample pellet is held in the rod frame just below the dewar section.

This cell can be used to elevate the temperature of the sample or lower it to chrogenic levels. To use at low temperature simply fill the reservoir with liquid nitrogen or dry ice - acetone. There was also a need to run the sample at a slightly elevated temperature. Dewars tend to be viewed primarily as chrogenic vessels. They

are equally useful in maintaining above ambient temperature. This was accomplished by feeding a heating coil made of nichrome wire through the reservoir (see fig. 8). The next step is to fill the reservoir with silicone oil and apply current to the heating coil.

The windows on this cell are also mounted with "O" ring joints. Because this cell is used exclusively for IR work, the window material is never changed. They do, however, need to be removed and repolished periodically. A second reason for the use of "O" ring joints to secure the windows is that we needed to install a thermo-couple wire near the sample pellet (see fig. 9). By stripping an inch of the insulation from the wire, it can be easily inserted through the center of the "O" ring. Pilot holes are made with a standard sewing needle. Lightly grease the thermo-couple wire and slide the wires through the pilot holes (see fig. 10). When the joints are clamped together vacuum integrity is maintained. This cell is used routinely in the $5x10^{-6}$ torr range.

NMR Transfer Dewars:

NMR transfer dewars are a common site in most research laboratories. The liquid nitrogen transfer dewars that came as original equipment with the last two NMRs our univeristy brought had 90° bends in each dewar. After inspection it became apparent that the dewar was constructed in two pieces and joined at the 90° angle. I reproduced the transfer dewar using this method, but found it too time consuming. This method also leaves the possibility of a leak at this joint.

I find it much easier and faster to make the dewar straight in the lathe and bend both the inner and outer at the same time (see fig. 11). To help in the construction, fuse three support posts equally spaced around the inner tube. These should be added on both sides of what will become the bend. These support posts serve two purposes. The first, to help support the inner and outer tube while sufficient heat radiates to the inner tube allowing it to bend smoothly. It also serves to hold the inner tube centered while you make the dewar seals on each end. Make sure these are far enough from the bend to be out of the heat zone when bending. You do not want the support posts to stick or fuse to the outer tube. This could cause stress problems later on and would also serve as a hot spot for the liquid nitrogen.

Using this method both speeds up the construction process and eliminates one area of potential leak.

I'd like to thank Diane Gerlach of Kenosha, Wisconsin for her slides of Sir James Dewar's early work.

This paper has not been presented elsewhere, nor has it been presented for publication elsewhere.

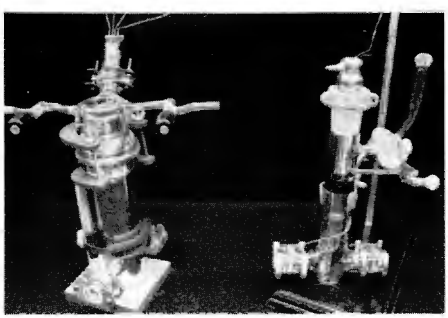

Figure 1

Figure 2

Figure 3

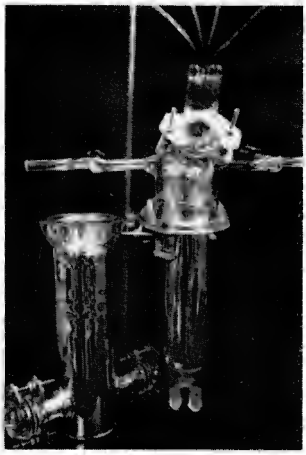

Figure 4

Figure 5

Figure 6

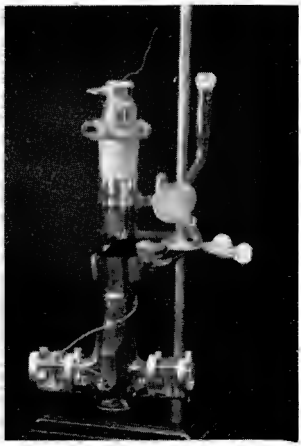

Figure 7

Figure 8

Figure 9

Figure 10

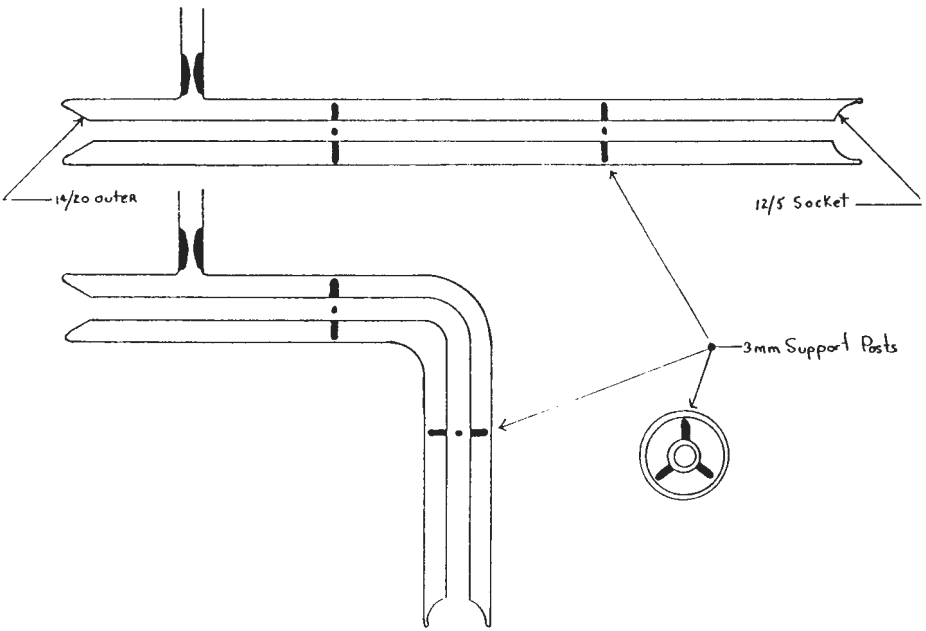

Figure 11

A FUSED QUARTZ DIAPHRAGM GAUGE FOR THE PRESSURE-CONTROLLED CRYSTAL GROWTH OF INDIUM PHOSPHIDE*

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Introduction

Indium phosphide, a semiconductor used in electronic and optoelectronic devices, is synthesized in sealed fused quartz ampoules by solidifying the In-P melt formed by the reaction between molten In heated to temperatures above 1000°C and P vapor produced by heating solid red P to temperatures in the range from 450 to 500°C. The P pressure, which determines the composition of the In-P melt, cannot be adjusted reproducibly by setting the temperature of the solid P, because the vapor pressure vs temperature curve for red P can vary significantly from batch to batch and even from time to time during the same synthesis run. In several runs that we have made using the conventional technique of heating the synthesis ampoule in air, the ampoule has exploded violently even though the P pressure expected from the published pressure-temperature curve was well below the estimated yield strength of fused quartz, which is estimated to be about 15 atm at the temperatures used in synthesis.

We have recently developed a horizontal gradient-freeze method that permits synthesis and crystal growth of InP to be performed without explosions at accurately controlled P pressures up to 35 atm. The synthesis ampoule is placed in a steel pressure vessel that can either be evacuated or pressurized with Ar gas, and the P pressure inside the ampoule is automatically matched to the Ar pressure. Pressure balancing is made possible by the use of an ampoule (Fig. 1) that incorporates a flexible diaphragm whose position is determined by the difference between the pressures inside and outside the ampoule. A voltage that depends on the flexure of the diaphragm is generated by means of linear differential transformers that are mounted at the ends of two long quartz arms attached to the diaphragm. The diaphragm voltage is used as the input signal to a controller that adjusts the power to the P reservoir heater, thereby fixing the P pressure.

At the beginning of a synthesis run, the evacuated and sealed ampoule is placed in the pressure vessel (Fig. 2). The vessel is then evacuated, and the controller is adjusted to maintain the diaphragm voltage at the value obtained at this time, when there is no pressure difference between the inside and outside of the ampoule. The pressure vessel is then filled with Ar to any desired pressure, and the controller automatically adjusts the temperature of the solid P reservoir to establish the same P pressure inside the ampoule. In recent runs, P pressures ranging from 200 to 540 psig have been controlled to ±0.5 psig for periods up to 3 days.

In this paper we describe the design and fabrication of the fused quartz diaphragm gauge. Satisfactory operation of this gauge is essential to the success of the pressure-balancing technique.

Gauge Design

The diaphragm gauge is in the same family as the spoon gauge,[1,2] and is related to gauges of the Bourdon or bellows type.[3,4] However, the high pressures involved in InP growth require a much heavier wall thickness than is typical for such gauges.

* This work was sponsored by the Department of the Air Force.
† G.B. Finkenbeiner, Inc., Waltham, MA.

Figure 3 is a cut-away view showing the basic design adopted. The nominal wall thickness is 1 mm, which allows enough flexure for sensitive pressure control while providing adequate strength to permit evacuating the growth ampoule in air and to withstand even higher differential pressures if necessary. The total length of the two arms attached to the diaphragm (see Fig. 1) is 32 in. Such long arms are used in order to locate the differential transformers in a cool enough region for long-term operation and also to amplify the motion of the diaphragm for increased gauge sensitivity. The ends of the arms are slotted to receive the movable transformer rods. With the transformers currently used, voltages in the range of 2.5 to 10 V are generated by a difference of 1 atm between the P pressure inside the ampoule and the Ar pressure outside it, corresponding to a linear displacement of 0.5 to 2 mm/atm between the ends of the arms.

Fabrication

The fabrication process begins with the selection of tubing with wall thickness as uniform as possible. We start with a 28-mm o.d. tube, the middle of which is blown out to 38-mm o.d. in order to obtain the desired amount of wall thinning (Fig. 4). The target wall thickness for the face and shoulder of the gauge is 1 mm. After the shoulder is blown to 38-mm o.d., one side is collapsed and tabulated with 12-mm o.d. x 8-mm i.d. tubing (Fig. 5). The wall thickness for the transition to the tubulation is approximately 1.5 mm. (This tubulation is later sealed to the P end of the synthesis ampoule.) The opposite side of the gauge is shaped by drawing, thinning, and collapsing to form a flat face. A wide, forceful flame, perpendicular to the face, is then used to push the thinned quartz into the desired concave shape with a 20- to 25-mm radius (Fig. 6).

Next, the piece is held vertically in a ring stand or bench clamp and tubulated with 8-mm o.d. tubing in two places at opposite edges of the diaphragm face. Each tubulation is buttoned off at a length of about 3/4 in (Fig. 7), a 6-in length of 8-mm tubing is added to each one (Fig. 8), and the assembly is annealed. To avoid unnecessary handling of a long and fragile piece of quartz, the additional 8-mm tubes needed to extend the arms to a total length of 32 in are not added until afte the synthesis ampoule is loaded, evacuated, and sealed. Before the extension tubes are added, the far end of each one is slotted to accept the movable transformer rod. In sealing these tubes to the gauge assembly, the slots are oriented horizontally (Fig. 9), so that the motion of the rods will produce a transformer voltage that is accurately proportional to the displacement of the arms. The advantage to having two arms on the gauge is that in the event the entire systhesis tube were to move (a possibility with this apparatus), the motion effect on the transformer of one arm would be compensated for by the opposite motion effect on the transformer of the other arm.

In addition to the heaters for the In boat and P reservoir, a separate heater is required for the diaphragm gauge in order to prevent the condensation of P vapor, which would render the gauge inoperable. This heater is contained within an open-ended quartz tube 36 in long that slides over the end of the synthesis ampoule and is held in place by means of two double wrappings of 0.015-in-thick x 1-in-wide quartz cloth tape (Fig. 10). The tape provides a very firm fit and holds up well at 700°C. The tube also shields the diaphragm gauge arms from contact with heater wires, thermocouples and any other obstructions, so that the arm movement is not hindered. Because the quartz tape provides such a firm fit, the outer tube also serves as a handle for loading the synthesis ampoule into the pressure vessel. Attaching the tube to the ampoule by means of a ring seal, rather than the tape, would significantly complicate the fabrication procedure.

Conclusion

A fused quartz diaphragm gauge has been developed for the purpose of controlling the P vapor pressure during the synthesis and crystal growth of InP by the horizontal gradient-freeze method. The synthesis ampoule containing the diaphragm is heated inside a pressure vessel that is filled with Ar gas. Flexure of the diaphragm generates a voltage that depends on the difference between the P pressure inside the ampoule and the Ar pressure outside. By using this voltage as the input to a controller that adjusts the temperature of the P reservior, the P pressure is automatically matched to the Ar pressure. The diaphragm is strong enough to withstand the pressure differentials encountered in the synthesis process but flexible enough to allow accurate pressure control. We have found that the precise shape and wall thickness of the diaphragm are not critical for successful operation of the gauge. As well as permitting the P pressure to be set at any desired value simply by adjusting the Ar pressure, the pressure balancing technique greatly increases the maximum P pressure that can be employed during synthesis. Pressures up to 35 atm have been controlled to ±0.1 percent over extended periods.

Acknowledgment

I would like to thank John McMillan for assistance in the fabrication of synthesis ampoules, and Alan Strauss for a critical review of the manuscript.

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Figure 1: InP synthesis ampoule.

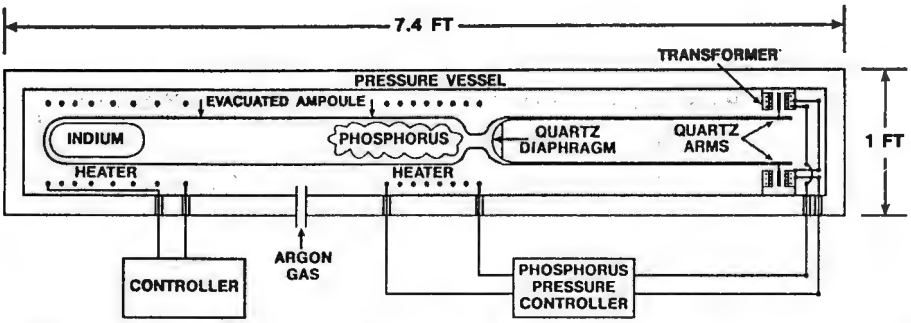

Figure 2: Schematic cross-sectional diagram of InP synthesis system.

Figure 3: Cut-away photograph of fused quartz diaphragm gauge.

Figure 4: Blowing out to thin the wall.

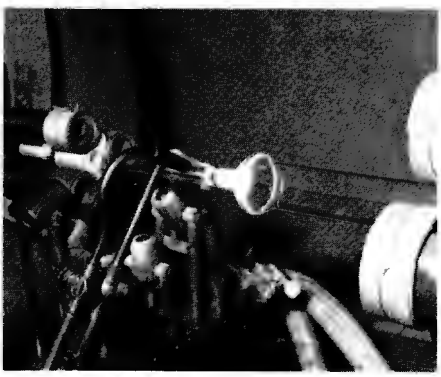

Figure 5: Transition to 12-mm o.d. quartz.

Figure 6: Forming concave face.

Figure 7: Tubulating and closing arms.

Figure 8: Adding extensions to arms.

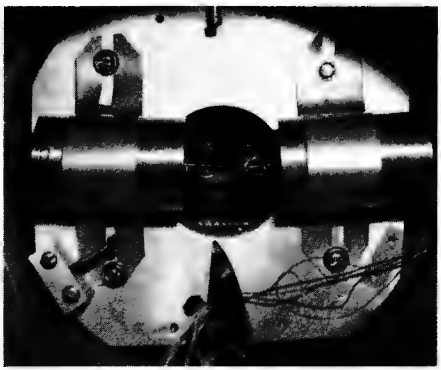

Figure 9: End view of gauge arms and transformers.

Figure 10: Assembly of outer shield tube and synthesis ampoule.

A GUIDE TO SAFE HANDLING AND DESIGN OF LABORATORY GLASSWARE

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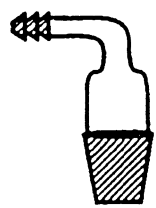
Safety records at Ethyl's research and development facilities show that injuries have frequently happened from the improper handling of laboratory glassware. These data also show that new and less experienced employees are at greater risk of having an accident.

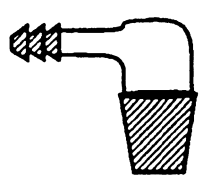
I prepared a set of guidelines aimed particularly at inexperienced Ethyl employees, in the hope of greatly reducing injuries caused by glassware. My objective in publishing this guide is that others might find it helpful in teaching the safe use of scientific glassware in their own organizations.

Why is a glass used to make laboratory apparatus? Glass is transparent, you can see what is taking place inside the apparatus. Glass does not react with most laboratory chemicals. The exceptions are hydrofluoric acid and strong bases. Glass can be fabricated into almost any shape by a glassblower. It is extremely strong under compression, making glass well suited for vacuum applications. The exception to this is flat glass or flat areas in apparatus. Glass has a low tensile strength making it unsuitable, for the most part, for pressure applications.

The contents of the guidelines are:

1. Always provide hand protection, either leather gloves or heavy cloth, when applying any force to glassware.
2. Connecting rubber or plastic tubing to glass connectors.
   a. Avoid using curved-type connector. These will snap off easily if too much pressure is applied.
   b. Use a side-sealed connector for a 90° application. This style is less likely to break.
   c. Straight sealed adapters are best.
   d. Use leather gloves when pushing tubing on to connector.
   e. Warm plastic tubing with a heat gun to soften before pushing it on to the glass connector.
   f. Wet the glass connector with water before pushing the tubing on.
   g. Secure with a hose clamp or wire.
   h. Cut rubber and plastic hoses off—don't pull them off.
3. Glassware under vacuum
   a. Never apply vacuum to a creased (Morton) flask or any apparatus with flat areas.
   b. Wrap Dewars with glass cloth tape.
   c. Consider using a fiberglass caddie to cover large storage type Dewars.
   d. Metal and plastic Dewars are also available.


(a)


(b)


(c)

e. Use a heavy-wall suction flask for filtering. Never use an Erlenmeyer for this purpose.

f. All Desiccator flasks must be taped.

4. Grease stopcocks and ground glass joints.

   a. Use a minimum amount of grease.

   b. Too much grease may cause the bore of a stopcock to become plugged, or cause contamination of product.

   c. Too much grease on a stopcock or gound glass joint may cause a vacuum leak due to channeling.

   d. Teflon® TFE sleeves for glass joints are available in the stockroom. They are superior to grease in applications where solvents may dissolve normal grease applications.[1]

5. Removal of frozen (i.e., stuck) joints and stopcocks.

   a. Apply heat with a heat gun, then tap joint with a small block of wood. Never use anything but wood to tap the glass.

   b. Use tweezers as a wedge. Come to the glass shop for a demonstration of this technique.

   c. Bring the apparatus to the glass shop for the glassblower to separate it, if you have any problems separating the glassware.

   d. Teflon® TFE stopcocks that are stuck should be packed in ice for ten minutes. This will cause the teflon to contract, then the stopcock plug can easily be removed.

6. Heating of glass vessels.

   a. Flat bottom beakers should only be used for gentle heating. The bottom will expand much faster than the sides when heated and cause stress to form in the corners. The larger the beaker is, the more stress will be created.

   b. Erlenmeyer flasks are good for moderate heating, such as boiling water.

   c. Round bottom flasks are the best for strong heating. Their round shape distributes the heat evenly over the surface causing the least amount of stress.

   d. Never heat a heavy-wall filter flask.

   e. Standard (borosilicate) laboratory glassware with brand names - Pyrex®, Kimax®, Duran®, can be heated to 513°C; this is the strain point. A practical maximum service temperature will always be below this point.[2]

   f. Quartz glass can be heated to 1000°C for continuous use, 1200°C for a short period.

7. Drilling rubber stoppers and inserting a glass tube.

   a. Carefully select a stopper borer that just fits over the glass tube that you want to insert through the stopper.

   b. Use a soap solution to lubricate the borer stopper.

   c. Bore the hole in the stopper and leave the borer in the stopper. Remove the rubber from inside the borer.

d. Insert the glass inside the borer. Remove the borer from the stopper. The glass tube will be inside the rubber stopper without having applied any pressure to the glass.

e. See the glassblower if you need any assistance or a demonstration of this technique.

8. Rotary evaporator maintenance
   a. Clean and regrease seals frequently.
   b. Only the glassblower should remove the center tube for cleaning and regreasing.
   c. Keep the condenser covered with polyethylene mesh.
9. Sample jars, storage jars, and vials.
   a. These types of containers are made from soda lime glass. They cannot be heated.
   b. Jars and vials are not meant to hold pressure or vacuum.
   c. If jar lids become stuck, a rubber disk or rubber tubing and leather gloves will help with removal.
   d. Never exert more than moderate pressure when removing or tightening a jar or vial lid.
10. Glassware under pressure.
   a. Glass has low tensile strength. Pressure puts glass under tensile stress.
   b. Due to undectable flaws that may be present in the glassware, it is not advisable to use glass in a pressure situation.
   c. If you want to use glass under pressure the glassblower must be consulted and a safety audit will be performed.
   d. Never pressurize a glass vessel to transfer a liquid.
11. Care and disposal
   a. When storing glassware in drawers, line the drawers with rubber matting and place paper between the different items to keep them from abrading each other.
   b. Store round bottom flasks on cork rings.
   c. Use rubber matting in sinks when washing glassware. Don't fill a sink full of glassware; wash one piece at a time.
   d. Never pick up a flask by a side neck. Always hold the center neck with one hand and support the flask from the bottom with the other.
   e. Inspect glassware before you use it. When in doubt, consult the glassblower. Look for chips, cracks, and scratches. Bring to the glass shop if it needs repair. Never use chipped, cracked, or broken glassware.
   f. Use new glassware if the job requirements are anything above normal.
   g. Avoid the urge to catch falling glass. You could be seriously injured if the item breaks in your hand.
   h. Periodic annealing in the glass shop will relieve stress in the glass, but it will not eliminate flaws.
   i. Consult the glassblower to determine whether the glassware should be discarded or repaired.

j. Dispose of glassware in the proper container. Every lab should have a "glass only" waste container. Don't drop or throw the glassware into the waste container. This may cause glass pieces to fly out. Don't pick up broken pieces by hand. Use tweezers or sweep them up. Pick up small shards with a damp cloth or damp paper towel.

k. When carrying glassware, always have one hand free. Don't overload yourself. If you need to move a lot of glassware, place the glassware in a box, bag or bucket.

12. Clamping glass apparatus
    a. Wrap the glassware with glass tape at the point where it is clamped.
    b. Adjust the clamps tight enough to hold the apparatus securely, but not so tight that you stress the glass.
13. Protective equipment
    a. Always use leather gloves if you apply physical force to glassware.
    b. Under vacuum or pressure, tapes, protective mesh, shields, hood sash, face shield, and goggles should be used in the proper manner.
14. Keep glassware clean
    a. Dirty glassware could hide serious flaws.
    b. Glassware sent to the glass washing room must be free from hazardous chemicals.
    c. All glassware sent to the glassblower must be free of all chemicals.
    d. If you have any special cleaning problems, consult the glassblower.
15. Glassware containing mercury
    a. Broken thermometers should be brought to the glassblower in a suitable container. The glassblower will remove the mercury and discard the thermometer.
    b. Any glassware containing mercury should always have a catch pan beneath it to contain the mercury in the event of a spill.
    c. If a mercury spill occurs, consult MSD sheet.
    d. Glassware which has contained mercury must be thoroughly cleaned with nitric acid before it can be repaired or returned to stock.

These guidelines are intended to help reduce glassware related injuries. They may not be all inclusive. Prudent safety practices and good judgement should always be followed.

A Guide to Glassware Safety & Design

1. Please fill out a Glass Shop Request Form. Fill in the appropriate information; Work Requested by, Room Number, Phone Number, Date, When Required, and Project Name. In the space for "when required" please be honest. A.S.A.P. is meant for emergency. If you need the apparatus for the next day put the next day's date. The "Project Name" is just a general name for what you are working on.
2. Draw a sketch. It isn't necessary to be an artist, it doesn't have to be pretty, just the general shape will do. Draw as large as you can, this will make it easier to add dimensions and detail to the sketch. Use a top view for flasks. If you have any questions ask.
3. Provide necessary information.

When preparing to make glassware there is certain information about the apparatus the glassblower needs to know before starting work.

a. List the dimensions that are critical, length, width, bore, capacity, etc.

b. List the type of joint - tapered, ball socket, etc.
List the size of joint - 24/40, 14/20, 35/25, etc.

c. List the kind of stopcocks - teflon, all glass, high vacuum.
List the size bore for stopcocks - 2mm, 9mm, 6mm, etc.

d. Will the apparatus be used under vacuum or pressure?

e. Give the purpose of the apparatus - solvent still, fractionating column, air sensitive compounds.

f. Discuss the plans with the glassblower.

4. Example of the form filled out. See page 38.

5. Keep in mind the safety items that were discussed earlier; No flat areas on vacuum apparatus, straight or butt seals, hose connections, proper stopcocks and joints that suit the application, easily accessible stopcocks and valves.

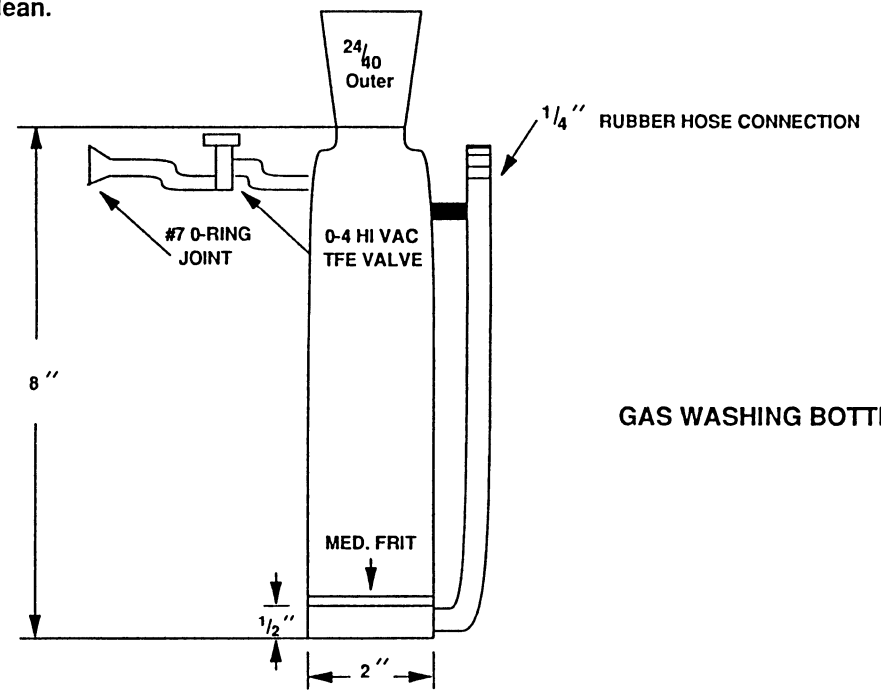
6. Review the job with the glassblower. This is the best way to insure the glassware will be made to fulfill your needs.

Footnotes:

1. Teflon REG TM E.I. du Pont de Nemours & Co.
2. Corning Glass Works, The Lab Book, 1983

WORK REQUESTED BY	ROOM NO.	PHONE NO.	DATED	WHEN REQUIRED	PROJECT NAME
A-1 CHEMIST	404-A	EXT. 5998	1-25-90	2-2-90	XYZ

Make sketch in sufficient detail so that the job can be completed without instructions. If space is insufficient, use reverse side or attach another sheet. All glassware for repair must be clean.



GAS WASHING BOTTLE

LEAK DETECTION WITH A TESLA COIL

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Introduction

Using a Tesla coil to leak-detect glass vacuum systems has been a standard reliable technique used by both glassblowers and lab technicians for many years. However, there is a standard problem of using the Tesla coil near or around metal parts. This paper will discuss the problems with past techniques used to by-pass the "metal barrier" as well as explain why the Tesla coil works. The mechanism of how the Tesla coil works turns out to be fundamental in explaining why metal is a problem in Tesla coil leak detection in the first place. I will also offer a technique for Tesla coil leak detection which does not require a vacuum.

Background

The standard practice is to hold the tip of a hand-held Tesla coil a few centimeters away from the glass and pass it over likely regions where a leak is suspected.

A leak is detected when the spark from the Tesla coil enters the glass wall at the point of the leak (see Fig. 1).[1] The leak is easier to see by turning down, or off, the lights (see Fig. 2). Once located, the leak is easy to repair by removing the glass in the specific spot that is leaking, add glass if necessary, work till smooth, and flame anneal.

Figure 1: Spark Testing in Light

Figure 2: Spark Testing in Dark

As we all know, this technique works well except in locations near, or around, metal parts. If the Tesla coil is passed near metal parts such as glass to meal seals or supporting clamps, the spark from the Tesla coil will go directly to the metal. This will happen even in the vicinity of a known leak.

One technique, often suggested when encountering this problem, is to spray a probe gas around the apparatus in question while running a Tesla coil against its side. The presence of a leak can be identified by the change of discharge color within the glass apparatus when the probe gas invades the discharge. This technique is one of the variations on the standard *outside-in technique* that is often used with thermocouple gauges, pirani gauges, and helium leak detectors. The criterion for selection of the probe gas is made from a variety of reasons including its cost, safety, and its uniqueness to the system.

However, the user is directed to choose a probe gas from a table of discharge gases. Table I is one example of gas discharge colors. The colors listed are those observed for the various gas discharge tubes. [2, 3, 4, 5, 6.]

However, the colors initiated from discharge tubes are not a good reference for leak detection. This is because of a variety of reasons, among which are:

1) the applied voltage and pressure can vary greatly.
2) the Tesla coil discharge, in leak testing, is a through-glass discharge whereas a discharge tube is a metal-electrode discharge.
3) the positive and negative glow depends upon the pressure and/or the distance between metal electrodes, and regardless, the distance between electrodes in a discharge tube is fixed as opposed to the distances within an apparatus being tested which can vary from as little as several centimeters to many meters during leak detection, and
4) the colors indicated in the tables are for pure gases. In a working vacuum system there will always be some air and moisture mixed with the probe gas. There will also be a trace amount of hydrocarbon vapors (from the mechanical pump) and/or other gases and vapors that may incidentally be in the system.

Table I

Appearance of Discharges in a Gas Discharge Tube at Low Pressures (7)

Gas	Negative Glow	Positive Column
Air	Blue	(Reddish)
Nitrogen	Blue	Yellow (red gold)
Oxygen	Yellowish white	Lemon
Hydrogen	Bluish pink (bright blue)	Pink (rose)
Helium	Pale green	Violet-red
Argon	Bluish	Deep red (violet)
Neon	Red-orange	Red-orange (Blood red)
Krypton	Green	—
Xenon	Bluish white	—
Carbon monoxide	Greenish white	(White)
Carbon dioxide	Blue	(White)
Methane	Reddish violet	—
Ammonia	Yellow-green	—
Chlorine	Greenish	Light green
Bromine	Yellowish green	Reddish
Iodine	Orange-yellow	Peach blossom
Sodium	Yellowish green (whitish)	Yellow
Potassium	Green	Green
Mercury	Green (goldish white)	Greenish blue (greenish)

— indicates no distinctive color

( ) indicates a different observers' opinion of the color

To understand why a discharge is created in a vacuum system or a discharge tube, we must first understand what the Tesla coil does. The Tesla coil generates voltages as high as 50 kV at the spark electrode. This creates a plasma of ions, metastable atoms and higher-excited-state, rare gas atoms within its proximity.[7] If a Tesla coil is brought close to a glass system where the pressure is between 20 and 10^{-3} Torr,[8] a glowing discharge will be created within the evacuated chamber.[9] If the pressure is greater than ' 20 Torr, the discharge will be quenched. In other words, the molecules will collide with each other and lose their excited-state energy by non-radiative pathways such as bumping into each other. If the pressure is less than ' 10^{-3} Torr, there will not be enough molecules present within the system to sustain the discharge. The color and character of this discharge depends on the gas, or gases, present in the system as well as the pressure. [10, 11]

The specific colors seen in a discharge are caused when the electricity from the Tesla coil raises the energy level of the electrons in the gas. Since this higher excited activity level cannot be sustained, the electrons fall back to lower energy levels. During this energy-level fall, they give off energy in the form of radiant light. Each

atom has a different atomic structure and therefore emits different visible and invisible light characteristics during this energy fall.

Among the properties of a discharge is the ability to conduct electricity. Thus, the spark from the Tesla coil enters the leak as the shortest, easiest route to obtain ground. This can be demonstrated by a simple experiment. If a Tesla coil is held up to a pinhole in a non-evacuated glass tube, no spark will be generated toward the glass or the pinhole (see Fig. 3).

However, if a short wire is inserted into the glass, with one end near the pinhole, the Tesla coil will generate a weak spark to, and through, the pinhole. If a longer wire (which is a better ground) is substituted ($\approx$ 1 meter), a spark to the pinhole will be initiated. This spark is identical to the type of spark seen going to a pinhole in an evacuated system. Finally, as a test, if an insulated wire is inserted into the piece in question, no spark will be initiated from the Tesla coil to the pinhole. This is regardless of the length of the wire (see Fig. 4).

Figure 3: Non-Vacuum Spark Testing

Figure 4: Insulated Non-Vacuum Testing

The source of the ground within the system can be the discharge within the vacuum line and/or the mechanical pump. If the size of the discharge is sufficient, it can itself supply an adequate ground. However, if the size of the discharge is insufficient to supply an adequate ground (such as the test case with a short wire), the discharge can still provide electrical conduction to the mechanical pump which can easily supply the ground.

This demonstrates why it is difficult to use a Tesla coil near metal parts. Metal is a much better conductor than the discharge within the system, and the spark is simply taking the path of least resistance. Figure 5 shows the discharge going from point of excitation directly to the pump.

There are also times when a number of very small holes will have the cumulative effect of a large leak. Thus you are unable to achieve a vacuum better than 10^{-3} Torr, but the Tesla coil is unable to pinpoint the specific spot of the leak. This is sometimes called a "colander effect". Sometimes, if the Tesla coil is allowed to sit on such an area, a spark may be able to 'punch' through a weak wall and thereby indicate its presence. However, it is possible to enclose the area suspected to be leaking in a plastic bag filled with a probe gas. Any observed change in the discharge indicates the existence of a leak.

Figure 5: The Full Discharge

However, there is still the problem of how to use probe gases successfully with

a Tesla coil. Since the success of the probe-gas method depends on being able to detect a color change in the discharge, it is important to know what colors and/or variations in intensity to expect. I had already ascertained that the colors from either the negative or positive glow of a gas discharge tube cited in the tables[12, 13, 14, 15] could not be used for comparison of an ordinary vacuum apparatus. The study presented here was carried out to determine (experimentally) the appearance of the discharge formed by various probe gases under actual leak-testing conditions.

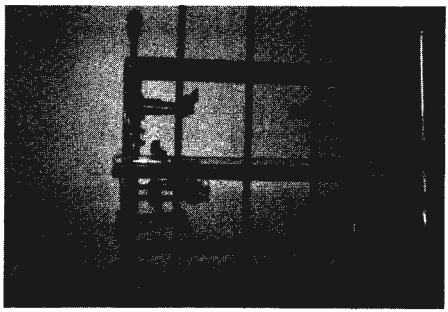

Figure 6: Testing Manifold

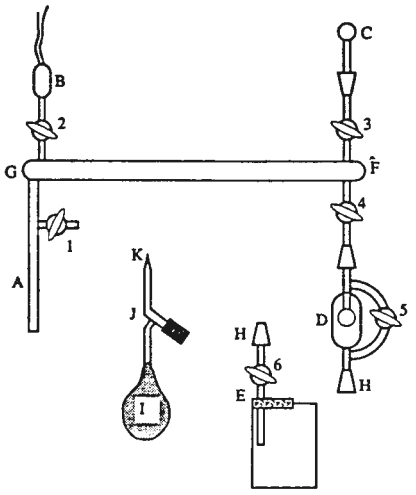

Illustration 1

Experimental

An apparatus was constructed to simulate a vacuum system with two leaks. It is shown in Fig. 6 and Illustration 1.

The leaks were made by fusing a piece of ceramic fiber into the end of a glass tube. This closed end was then blown into a small, very thin-walled bulb. Passing a Tesla coil over this bulb, while the tube was under vacuum, "punched" a hole through the weakened glass, providing the leak.

The probe gases were introduced by two methods. In one method, a bag containing the probe gas (E in Illustration 1) was attached to the system via the tapered joint at point H. The section between the leak (D) and the bag was pumped through stopcock 5. The Tesla coil tip was placed about 5 mm from point F in illustration 1 and turned on. Stopcock 6 was then opened, exposing the leak to the probe gas from the bag, and any color change could then be observed.

In the second method, the gases were placed in a balloon (I) and, through a drawn out tip (K), sprayed over the leak on top (C in Illustration 1). A rotary valve (J) was used to control the gas flow rate.

The system was given two leaks so that is could resemble a multiple leak situation. The leak on the bottom (D in Illustration 1) could be used to introduce the probe gas while the second leak on top (C in Illustration 1) pulled in air. This was used to determine if air could effect the color of the probe gas.

The vacuum was measured by a thermocouple gauge (B). The pressure with the stopcocks to both leaks turned off was $\approx 10^{-3}$ Torr. When the top leak was opened, the vacuum was $\approx 7.0 \times 10^{-2}$ Torr. When the bottom leak was opened, the vacuum was $\approx 11.5 \times 10^{-2}$ Torr. When both were opened, the vacuum was $\approx 18.5 \times 10^{-2}$ Torr.

The gases were selected because of their availability in a typical laboratory. The various volatile liquids were selected for their relative safety for both the equipment and the user. The gases and liquids are listed, with their results, in Table II. Figure 7 is a sample of the probe gas discharge testing.

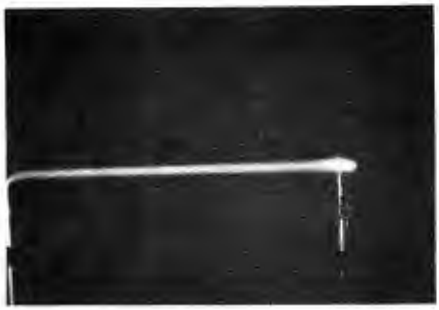

Figure 7: Testing a Discharge

Figure 8: Tesla Coils Normal & Heavy Duty

The manufacturer of the common, light duty Tesla coil recommended that they should not be run continuously for longer than ten minutes at a time. Therefore, if long continuous use is planned, one should use three Tesla coils in a five-minute-at-a-time rotation. Heavy duty Tesla coils are available that can be run continuously for hours and therefore do not require rotation for cooling (see Fig. 8).

Table II

Appearance of Discharges in Gases and Vapors of Volatile Liquids at Low Pressures When Excited by a Tesla Coil

Gas	Color Observed in Discharge	Gas Mixed w/ Air
Air (room)	soft violet	(same)
Argon	pale-magenta	pale-magenta
CO_2	greenish white	washed out magenta
Helium	soft-pink	purplish-magenta
Nitrogen	(too pale for magenta, not quite purple)	purple
Oxygen	white	pale-magenta
Volatile Liquid	**Color Observed in Discharge**	**Gas Mixed w/ Air**
Acetone	turquoise (then quickly to purple)	void* (then quickly to purple)
Dichloro-methane	bluish-white	void* (then quickly to bluish white)
Methanol	void* (then slowly to soft violet)	void* (then slowly to soft violet)

*void: a lack of discharge due to a loss of vacuum.

Results

The results of this study are given in Table II.

It is clear from Table II that the colors observed under actual leak-testing conditions differ from the colors reported to be observed in a discharge tube. The sprayed probe-gas method was tested with helium and oxygen only. These were chosen because they displayed the colors most easily distinguishable from air. It was necessary to spray the gas directly over the leak before any significant color change could be noted. The characteristic discharge was observable only in the vertical section (of leak C) on the testing apparatus (refer to Illustration 1). Further away from the vertical tube of leak C, within the main horizontal tube, the color had changed to the typical soft violet discharge typical of air. This depended on the distance from the Tesla coil, not on time.

Conclusion

Despite several limitations, the Tesla coil can be used effectively to detect leaks that are in the vicinity of metal parts which would normally preclude its use. However, there is a poor correlation between the colors observed in Table II and those that are reported for standard discharge tubes as in Table I. Therefore, it is inadvisable to use tables of discharge tubes as a guide to the color expected during leak detection with a Tesla coil.

There was limited, or no, change in the observed color of the discharge when the various gases were use as a spray from a balloon. It may be possible to go around this limitation by spraying directly from a compressed tank which would supply a greater quantity of gas at greater pressures. However, this could lead to filling the testing area with the probe gas and decrease its sensitivity. Therefore, the recommendation here is that the various probe gases be used in sealed environments, such as a bag around the suspected area. This would allow the user to at least verify whether or not there a leak is present. However, when it is necessary to find the specific location of a leak, it seems best to use either acetone or dichloromethane on a cotton applicator. Then slowly wipe the area until a color change is evident. The liquid should not be squirted or applied with a soaking cotton applicator around greased joints and/or stopcocks, o-rings, or other membranes that could be dissolved, or otherwise effected, by the solvent.

It should also be pointed out that the easiest observations will be obtained in a *very* dark room.

It is interesting to add that the Tesla coil is capable of finding leaks only in the typical range of mechanical pumps. If a leak is sufficiently small that the system is capable of a vacuum greater than 10-3 Torr, a discharge cannot be created and the Tesla coil cannot be used for leak detection. For leaks of such small size, a helium leak detector is recommended.

Acknowledgment

I would like to thank Dr. Cathy Cobb for her assistance and guidance in helping me understnd the physics involved in the Tesla coil, plasmas, and discharge tubes.

I would also like to thank the Journal of Chemical Educations for allowing me to present this paper to the ASGS as they will be publishing a form of it in the future.

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FABRICATION OF A SPECIALIZED RIGHT-ANGLE OPTICAL DEWAR

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Introduction

This paper describes in detail a method for fabricating an experimental right-angle dewar of clear fused quartz for use in low temperature studies of luminescence under intense magnetic fields (Figure 1). Although many of the approaches and techniques used are not necessarily novel, the fabrication of a liquid nitrogen-free optical path between the light source and the chemical specimen points out the utility and versatility of the quartz molecular bond or diffusion seal. These characteristics of the seal permit the glassblower to assemble quartzware possessing unusual aspects of dimension and design.

Fabrication

Essential highlights of the dewar's fabrication were as follows:

(1) Parts for assembly of the dewar were made with careful attention paid to the dimensions specified in Figure 1. [Figure 2(a)] The diameter of the flare on the inner member corresponded approximately to the inner diameter of the jacket. An extension was sealed on the inner member and subsequently trimmed so that as much of the extension as possible would fit into a thin bubble blown out on the side of the jacket.

[Figure 2(b)] The center sample tube and its associated extension as well as the outer member extension were then made. The optical windows were ground to dimension and sealed to their respective extensions by means of molecular bonds in a manner described by David Blessing[1,2].

(2) [Figure 3] With care in preparing the setup, the dewar seal was then completed. The inner member was held internally with a graphite rod into which a groove had been cut in order to vent the expanding gases in the lower section of the inner member. Alternatively, a stainless steel tube could have been used as an internal holder. Note that the packing between the holder and the inner member was situated so that the opening in the inner member was effectively sealed off.

(3) [Figure 4] With the inner member extension orifice closed off with a plug, the vacuum tip-off tubulation and a section of rod were sealed on the dewar.

(4) [Figure 5] The thin bubble on the outer member was then blown out and peeled away to expose as much of the tubulation on the inner member as possible and to leave a short shoulder on the jacket.

(5) [Figure 6] The sample tube was now passed down through the inner member and out through the port. With the tube held snugly against the inner wall of the dewar with a roll of packing material, the sample tube extension (trimmed to dimension) was sealed to it.

(6) [Figure 7] Using a mache packing and an annular winding of paper to centrally position the sample tube assembly within the dewar and through the inner member tubulation, respectively, the brace between the sample tube and dewar body was fused. A thin capillary was inserted through the mache and fused to the vacuum tip-off tubulation with a piece of rod.

(7) [Figure 8] A piece of 9 X 11 mm tubing was cut so that its length just equalled the portion of exposed sample tube and window. Using a short test tube of quartz and some annular packing as a closure, the 9 X 11 mm tube was fused to the inner member tubulation.

[Figure 9] In turn, the end of the 9 x11 mm tube was sealed to the edge of the sample tube window. Upon completion, the piece was annealed.

(8) [Figure 10] Next, the outer member extension was trimmed to length such that the distance between the two windows was 1-2 mm. This was then sealed to the jacket. Finally, the dewar was annealed a second time, silvered, closed at the base, evacuated, baked, and tipped off.

Conclusion

The fabrication of this custom dewar is but another example of the usefulness of the quartz molecular bond. Because of the relatively small amount of heat required to produce the seal and its inherent structural integrity, the glassblower can render quartzware to fulfill the stringent needs of the researcher.

Acknowledgements

I thank Dr. Guillermo J. Ferraudi of the Notre Dame Radiation Laboratory for whom this work was done, and Mr. Michael Pecina for his expertise in preparing the final drawings. I would also like to express my appreciation to Mr. David Blessing, my former supervisor at Notre Dame, who taught me the general method for making the quartz molecular seal.

The research described herein was supported by the Office of Basic Energy Sciences of the Department of Energy. This is Contribution No. NDRL-3296 from the Notre Dame Radiation Laboratory.

Footnotes:

1. Blessing, David, "A Vessel to Perform Pulse Radiolysis of Liquid Ammonia in a High Pressure Vessel," *Proceedings of the Nineteenth Symposium on the Art of Glassblowing*, American Scientific Glassblowers Society, Toledo, Ohio 1974, pp. 79-81.
2. Blessing, David, "Question and Answer Report," *FUSION - Journal of The America Scientific Glassblowers Society*, Toledo, Ohio, November 1985, pp. 20-21.

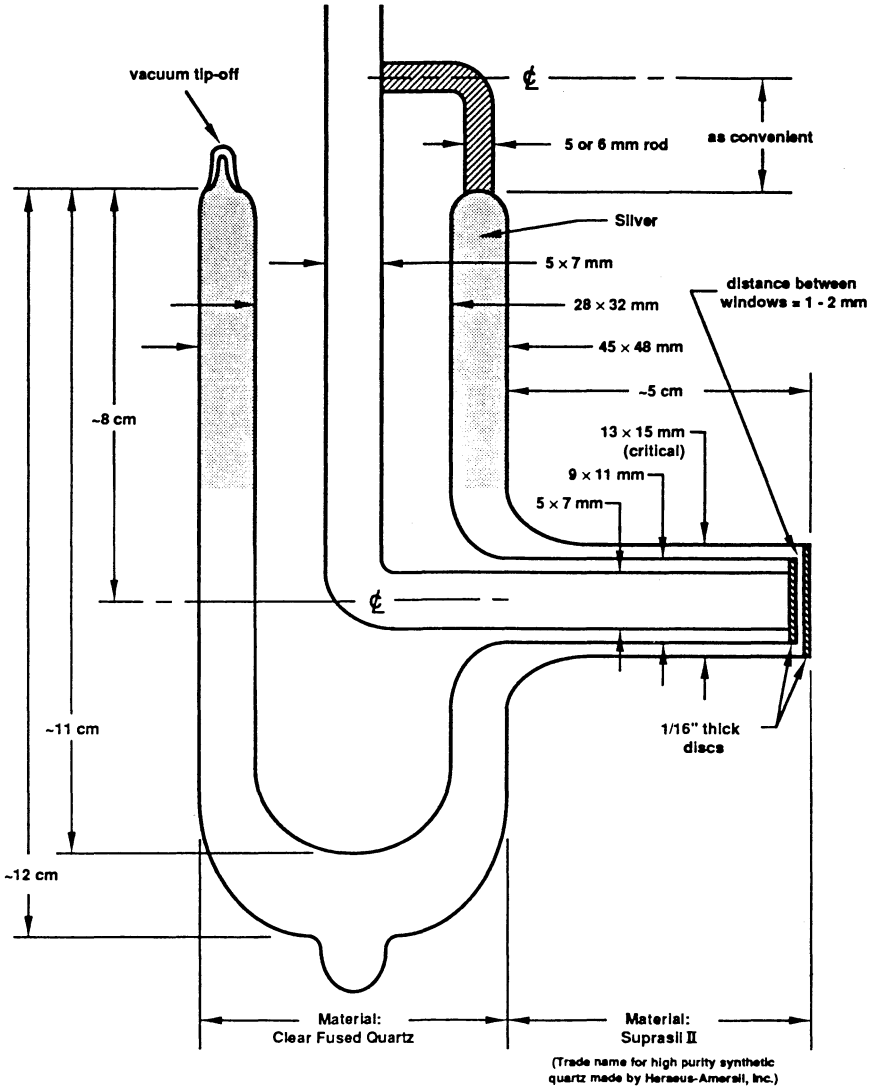

vacuum tip-off
℄
5 or 6 mm rod
as convenient
Silver
5 x 7 mm
distance between
windows = 1 - 2 mm
28 x 32 mm
45 x 48 mm
~5 cm
~8 cm
13 x 15 mm
(critical)
9 x 11 mm
5 x 7 mm
℄
~11 cm
1/16" thick
discs
~12 cm
Material:
Clear Fused Quartz
Material:
Suprasil II
(Trade name for high purity synthetic
quartz made by Heraeus-Amersil, Inc.)

Figure 1

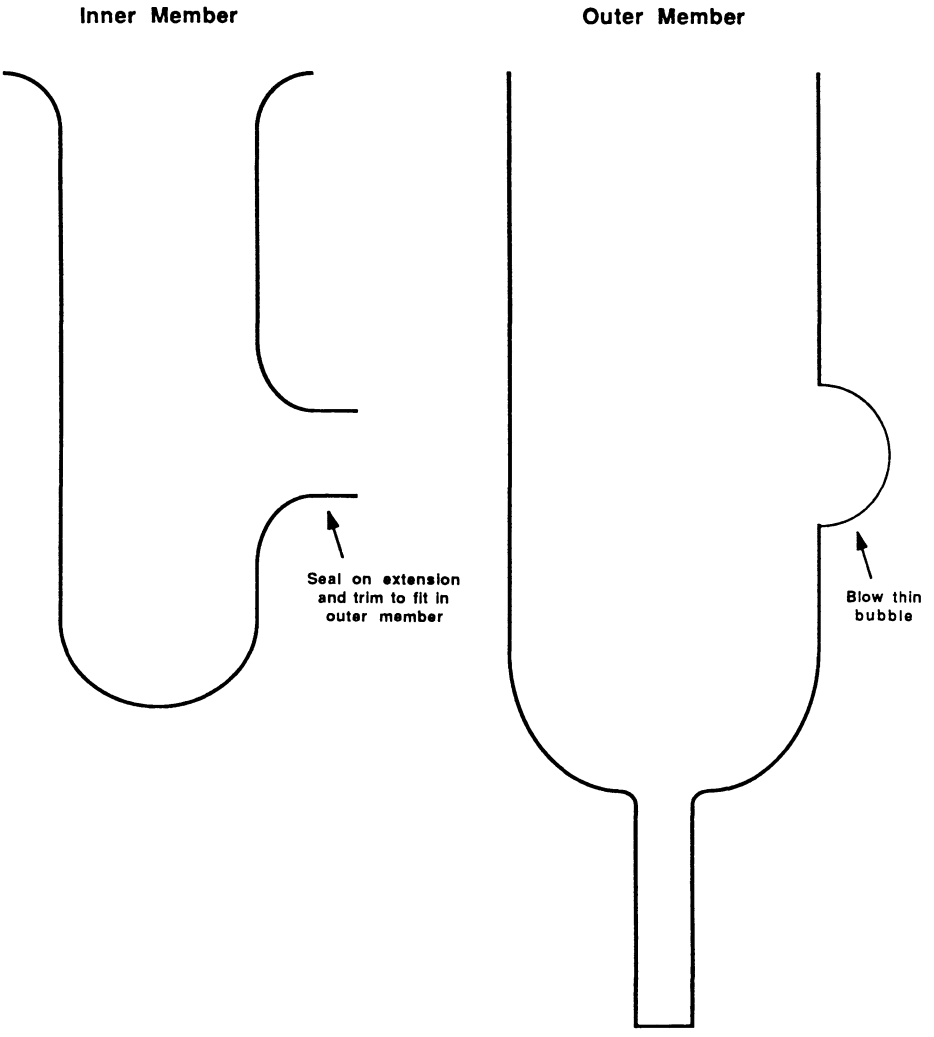
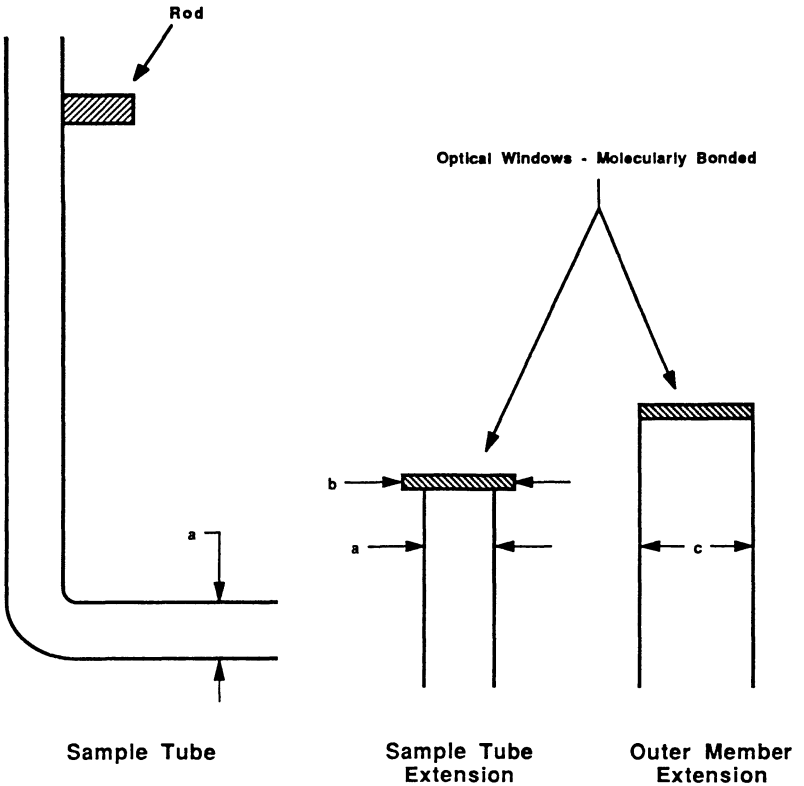


Figure 2(a) - Parts



a = 7 mm
b = 9 mm
c = 13 mm

Figure 2(b) - Parts

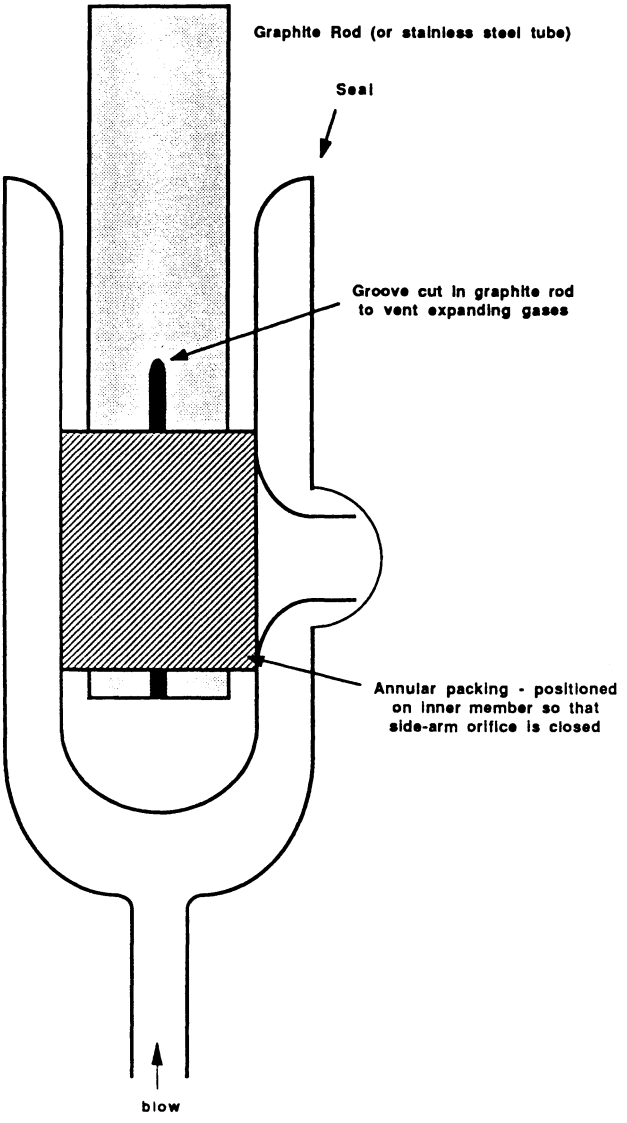

Graphite Rod (or stainless steel tube)
Seal
Groove cut in graphite rod to vent expanding gases
Annular packing - positioned on inner member so that side-arm orifice is closed
blow

Figure 3

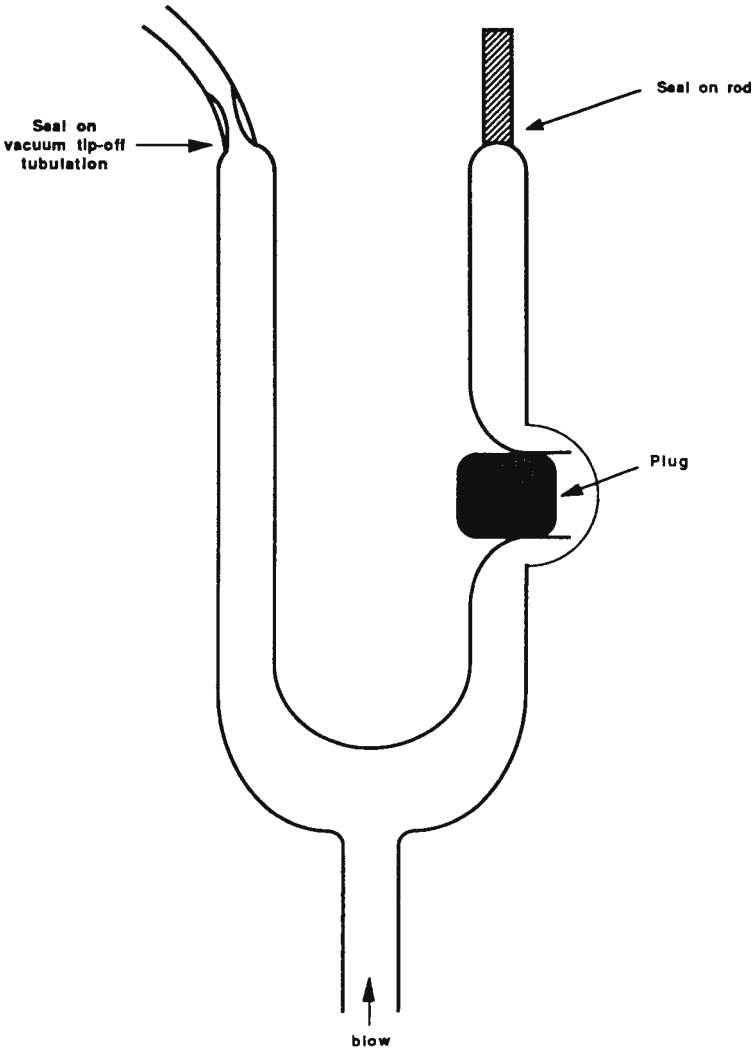


Figure 4

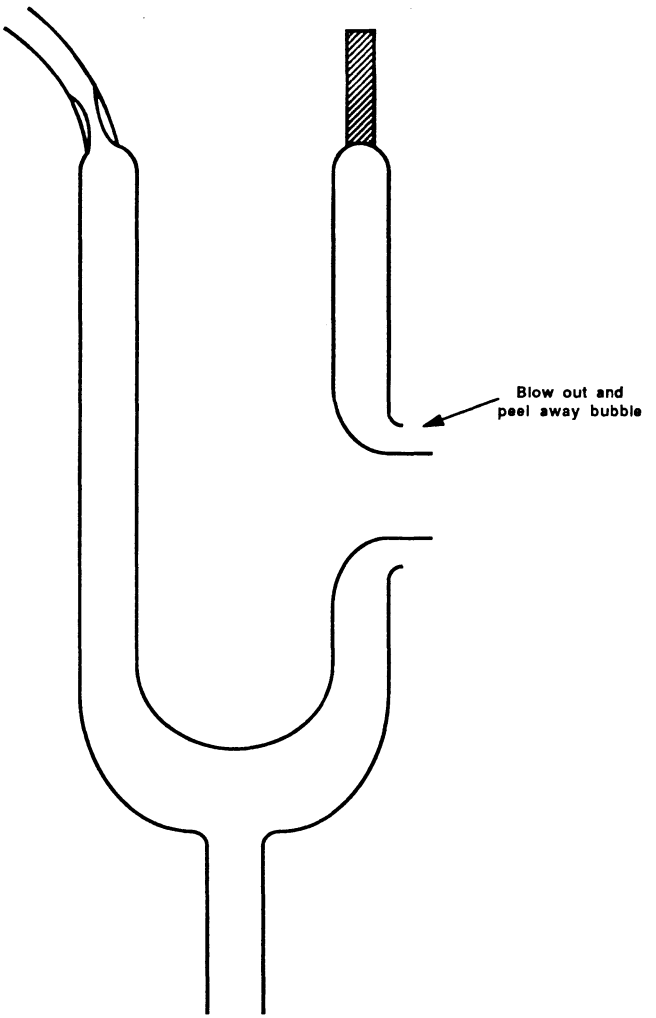


Figure 5

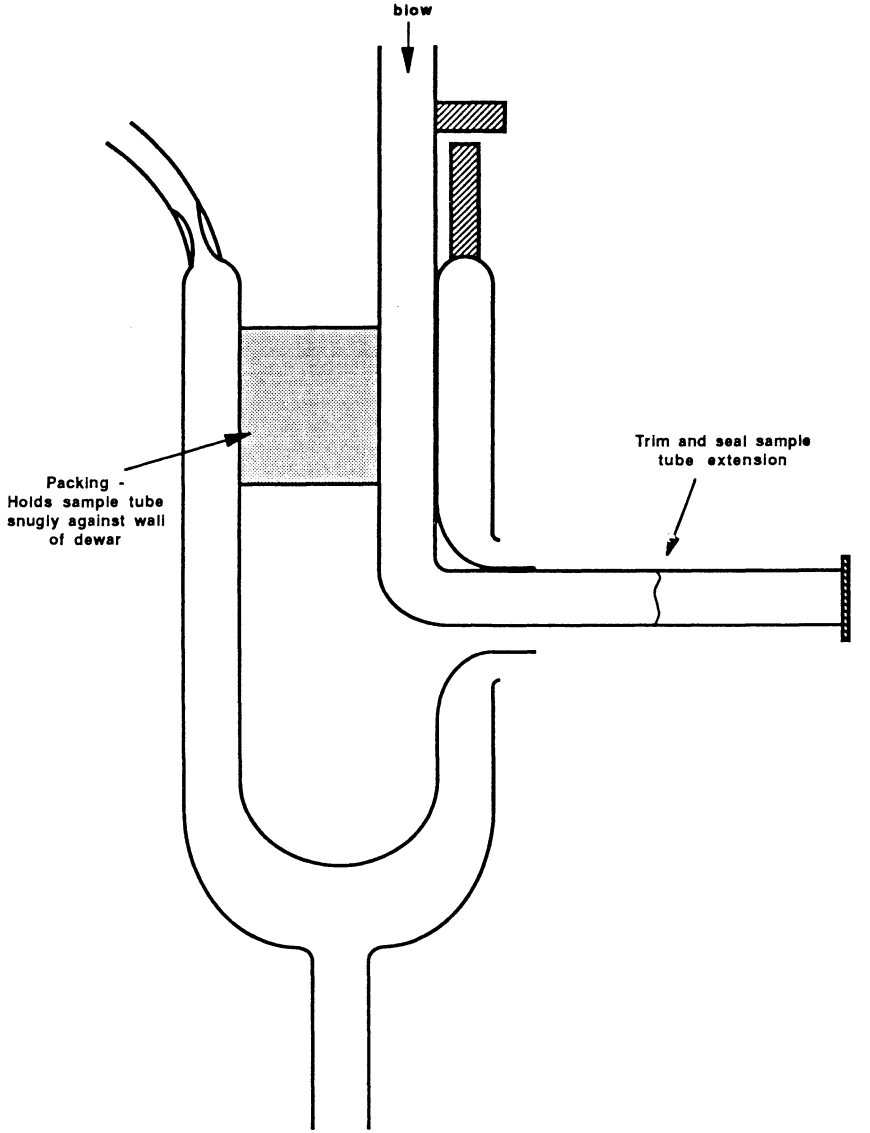

blow
Packing -
Holds sample tube
snugly against wall
of dewar
Trim and seal sample
tube extension

Figure 6

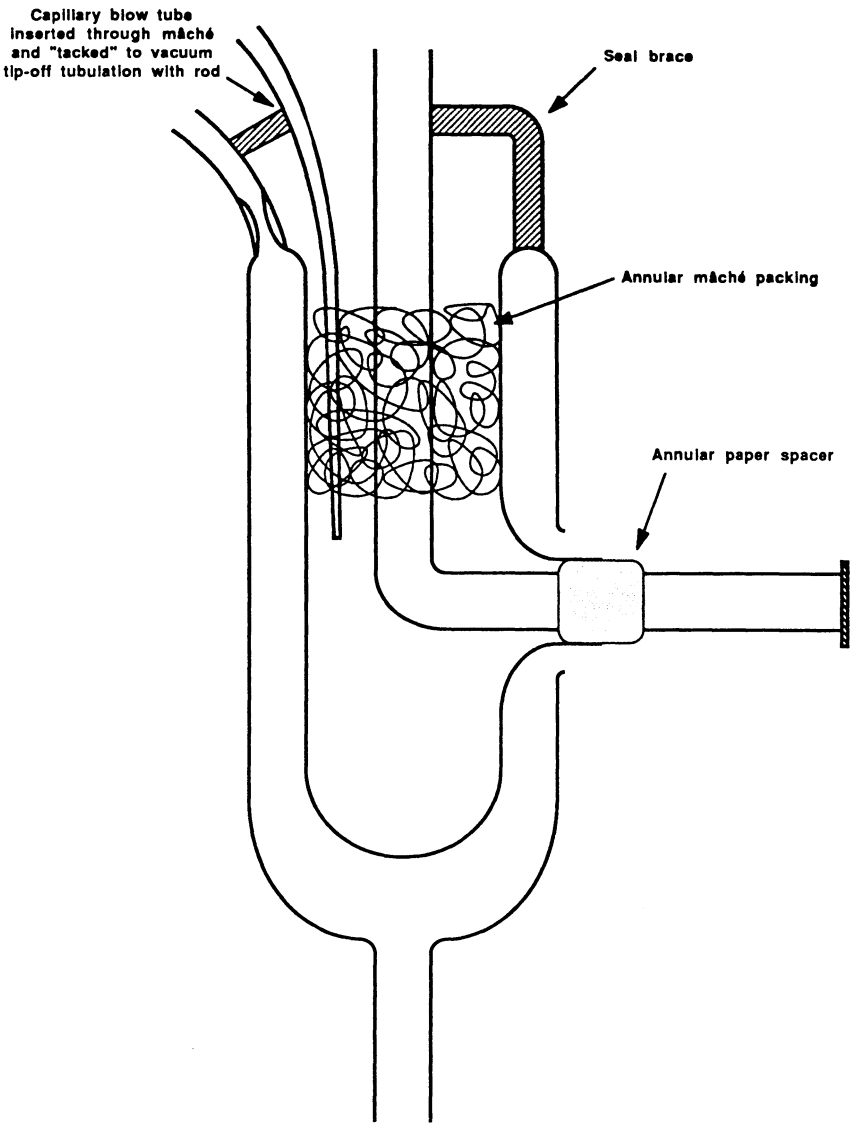

Capillary blow tube inserted through mâché and "tacked" to vacuum tip-off tubulation with rod
Seal brace
Annular mâché packing
Annular paper spacer

Figure 7

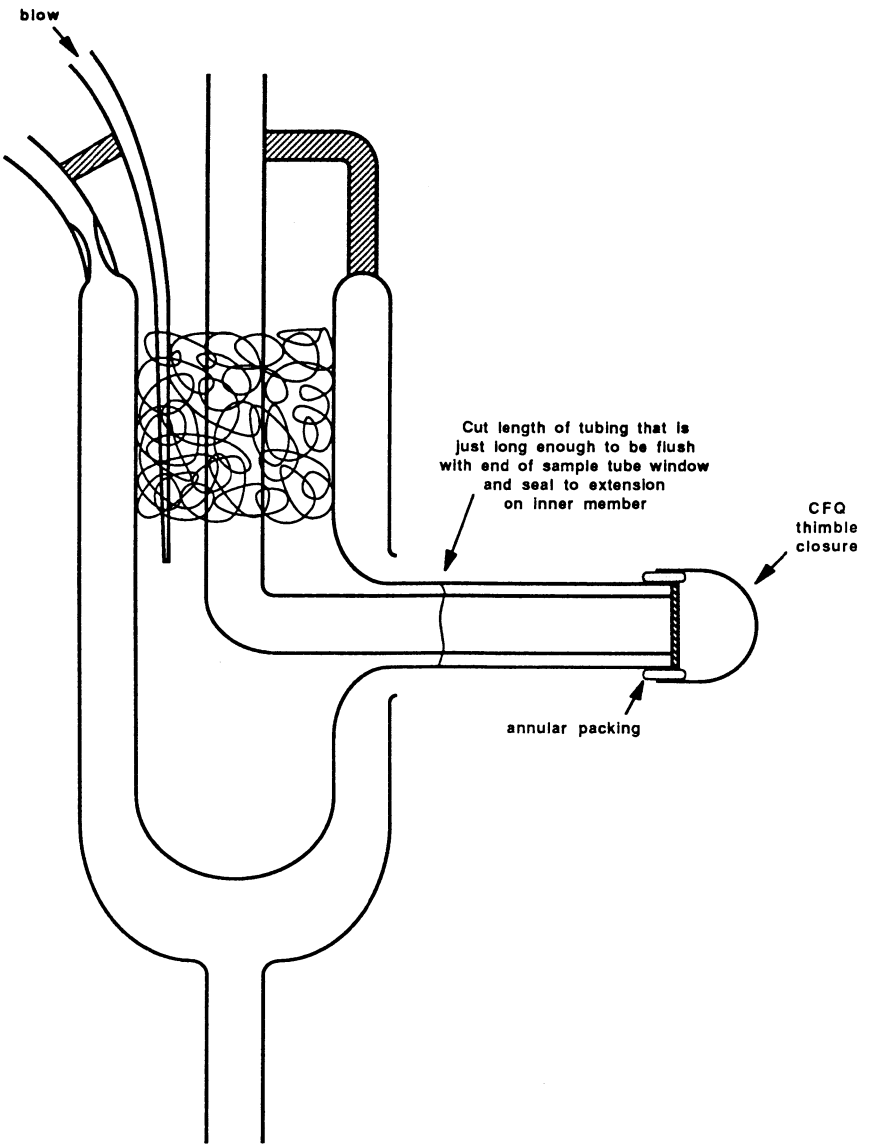

blow
Cut length of tubing that is just long enough to be flush with end of sample tube window and seal to extension on inner member
CFQ thimble closure
annular packing

Figure 8

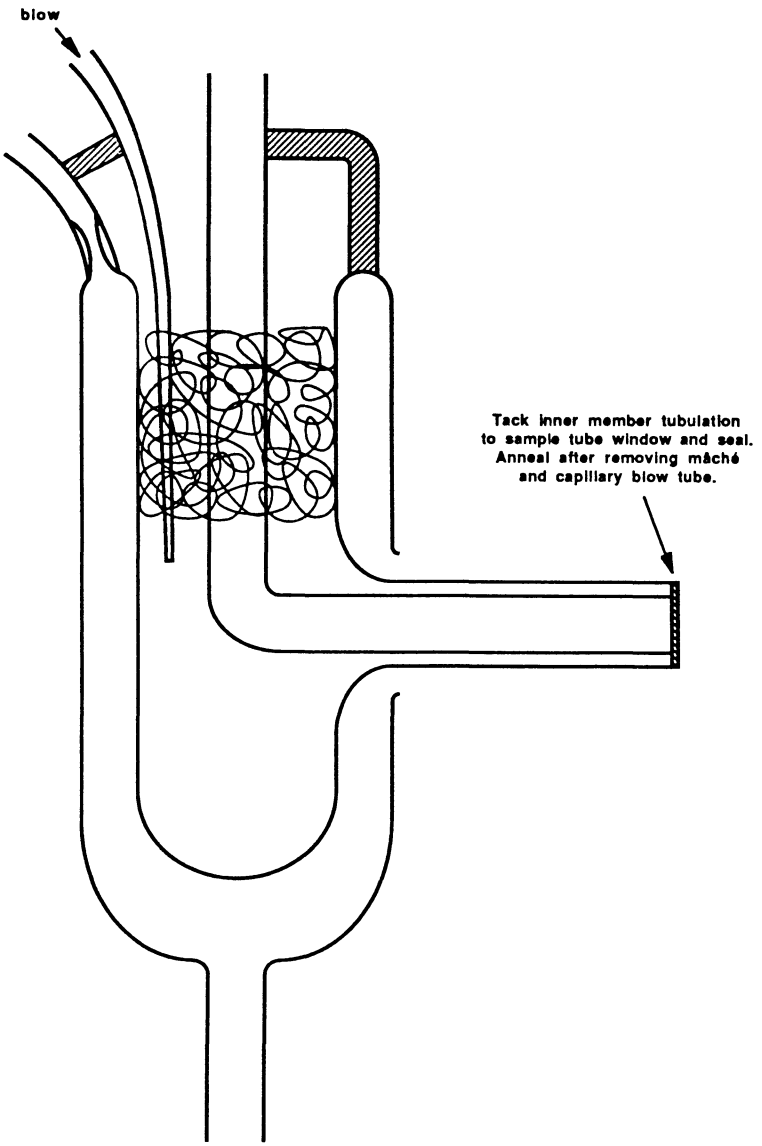

blow
Tack inner member tubulation
to sample tube window and seal.
Anneal after removing mâché
and capillary blow tube.

Figure 9

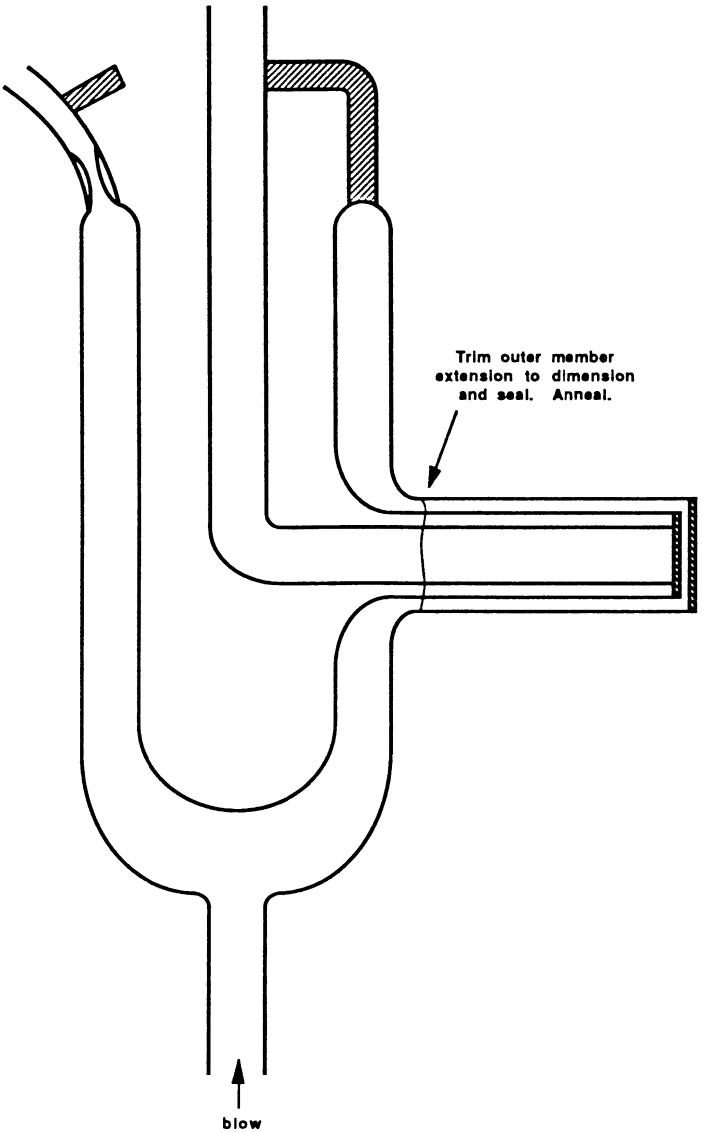

Trim outer member
extension to dimension
and seal. Anneal.
blow

Figure 10

LIABILITY AND INSURANCE ISSUES FOR THE MOONLIGHTING GLASSBLOWER

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Berg, Brick, and Weber
La Cañada, California

This paper will discuss two topics of importance to "moonlighting" glassblowers. It begins by discussing the three major theories of law under which a court could impose liability on a glassblower. It then proceeds to address the issue of whether the glassblower who moonlights at his/her place of business or out of his/her home should carry liability insurance. Although this paper focuses on the moonlighting glassblower, it must be emphasized that this discussion will actually be relevant, in varying degrees, to almost any glassblower.

This paper also requires a few important caveats. First, while the theories of liability discussed exist in all 50 states, their application to any particular set of facts differs from state to state. California law forms the basis of this article. Your own state may follow the same rules; however, it may follow different rules in certain respects, most notably in the area known as "product liability." Second, it is not possible, in a short article, to discuss all of the variables which create or limit liability; therefore, you should not consider this paper as the final authority on any issue. You should not rely upon it as a legal opinion regarding your own potential liabilities and risks. This paper is intended only to alert members to issues which may be of importance to them. If you have particular questions about potential liability risks, you should consult an attorney in your own state. If you have particular questions about insurance, you should consult an insurance agent or broker experienced in commercial insurance.

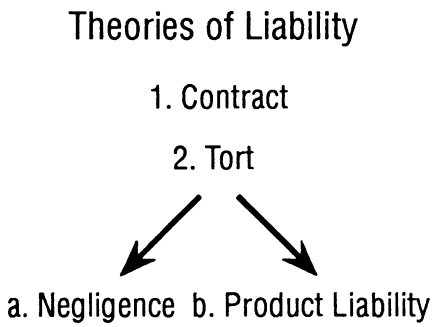

Figure 1

1. Theories of Liability

The concept of liability in the Anglo-American legal systems first arose in "assumpsit," the ancestor of our law of contract. Over the centuries, these contract notions failed to provide adequate remedies for a variety of injuries occurring outside of contractual relationships, and the theory of "tort" liability, in particular negligence liability, evolved to fill the void. Again, over the years, contract and negligence theories proved inadequate to remedy a variety of injuries, and a third theory, also a "tort," — strict product liability theory — has evolved. These form the three primary theories of liability which should be of concern to glassblowers. (Figure 1)

A contract is an oral or written agreement between two or more people.[1] The agreement consists of an offer, acceptance of the offer, and "consideration."[2] Consideration is what one party does to get the other party to enter the contract.[3] For example, a lab may offer to pay you to make glass apparatus. If you accept the offer, the lab's "consideration" is the payment to you. If the agreement does not require something of each party, there is no contract; the law considers these agreements gratuitous and unenforceable.[4] A promise to give a gift is not a contract, and not enforceable, for this reason.

If you fail to carry out your obligations under a contract, you may be liable for breach of contract.[5] This may occur if, for example, you do an unsatisfactory job, use the wrong materials, or fail to deliver glass apparatus on time. If you breach the contract, the other party to the contract can sue for damages.[6] Damages include the value of those benefits due under the contract;[7] any "consequential damages" directly arising from the breach, provided that both parties contemplated the potential of those damages at the time they entered the contract (for example, if it is known that delay in performance of the contract by one party will cause the other party to lose the opportunity to sell his products at a special sale, damages may include loss of profits)[8]; and interest accruing on these damages. You can only be liable, however, to the particular persons or businesses having enough of a relationship to the contract that they have rights under it. These would include those with whom you made the contract, anyone to whom they "assigned" (gave or sold) their rights under the contract,[9] and any others whom the contract intended to benefit.[10]

A tort is a little more difficult to define. A "tort" is any one of a variety of acts which wrongfully causes injury to another. The historic tort of importance to this discussion is that of negligence. You may be negligent if you unreasonably fail to protect others from risks of injury[11]. In order to be found liable under a negligence theory one must have violated (breached) a duty of "due care" owed to a person, resulting in an injury.[12] The "duty of due care" means that you should conduct your affairs in such a way that they do not injure others. Thus, if you sell glass apparatus, you have a duty to protect people from injury arising from the apparatus. The duty extends not only to users of the apparatus, but also to other "foreseeable" persons, for example, bystanders who may be injured as a result of glass failure.

The concept of "duty" is not unlimited. One has this duty of due care only for reasonably foreseeable risks of injury to foreseeable classes of people.[13] For example, it is "foreseeable" that improperly annealed glass may fail, causing injury to the user and bystanders. Thus, one has a duty of due care in annealing glassware. If the risk is not one which can fairly be anticipated, it is not "reasonably foreseeable" and negligence theory does not impose liability. For example, suppose poorly annealed glass explodes, startling a person in the next lab, who, in turn, drops a box of supplies onto a cat which, in panic, bites the foot of yet another person. It is unlikely that the glassblower would be found to have owed a duty to the injured person, because it is not foreseeable that improperly annealed glass would lead to cat-bite injuries.[14]

Negligence theory also requires that the injury be "proximately caused" by the negligence. This means not only that the negligence must have caused the injury, but also that there were no other events which intervened to break that cause-and-effect relationship.[15] Thus, if you negligently anneal glassware which is then misused in such a manner that even properly annealed glassware would have failed, the misuse may end the relationship between your negligence and an ensuing injury (unless, of course, the misuse was sufficiently foreseeable that you had a duty to protect against the misuse).

Negligence damages can include not only the economic value of the damage to person or property, but medical bills, loss of earnings, and pain and suffering.[16] This same measure of damages applies to the final theory of liability to be discussed in this paper, *strict product liability theory.*

A form of tort which has evolved over the last forty or so years, *product liability theory* or *strict liability theory* is the courts' solution to some problems of proof at trial which often prevented people from successfully suing under contract and negligence theories. In this context, the word "strict" means absolute and without regard to one's awareness of any potential problem. The words "product liability" limit the theory to

goods and other things which are manufactured or marketed.[17] If you help someone set up the parts of a vacuum system but do not supply the parts, you are providing a service which falls outside the doctrine.[18] By contrast, if you make any part of the system, you are providing a product and potentially subject to the doctrine.

Product liability theory is based on the idea that those who place products into the stream of commerce should ensure that those products are as safe as is reasonably possible. Thus, a major limitation on the theory: it applies only to products regularly placed into the stream of commerce, that is, made and held for sale to others. The glassblower making animals as Christmas gifts for family and friends is probably not at risk under this theory.[19] By comparison, if those animals are sold, the theory may apply. If the glassblower is selling his goods on any recurring basis, he is probably at risk that the theory would apply.

As already indicated, the doctrine applies to products placed in the stream of commerce. "Commerce" is interpreted broadly; thus, whether you provide your products to schools, industry, or individuals, or to other glassblowers or manufacturers wishing to integrate your products into their own products, you are "in the stream of commerce."[20]

Product liability theory applies when one of three circumstances creates a dangerous condition: a defect in design[21] (an example being a car designed such that the gas tank explodes on minor impact), in manufacturing[22] (for example, unannealed glass), or in the warnings provided to the consumer[23] (for example, absence of instructions on storing certain acids). Not all dangerous conditions result in product liability. The courts generally look for dangerous conditions which the consumer would not expect to exist, and which therefore make an item unfit for its expected uses as well as for predictable misuses or abuses.[24] Obvious and unavoidable dangers may fall outside the doctrine: knives have to be sharp, and glass always has a certain risk of breakage. However, if there is a safer and economically viable alternative, liability may arise.[25] For example, it is obviously dangerous to stick a finger into the working motor of a lathe. Since it is economically viable to put a cover over the motor, protecting against finger injuries, a manufacturer has a duty to provide an adequate cover.

Having determined that manufacturers and marketers alike should protect the product-buying public, the courts have put everyone in the chain of commerce at risk for injuries from defective products.[26] (Figure 2) Where a glassblower subcontracts to make dewars for a glass company, which incorporates the dewars into systems provided to wholesalers, who market them to retailers who, in turn, sell them to ultimate consumers, everyone in the chain of manufacturing and marketing of the product is potentially liable when a defective dewar breaks and causes injury. Since this is a "strict" doctrine, their being unaware of the defect is no

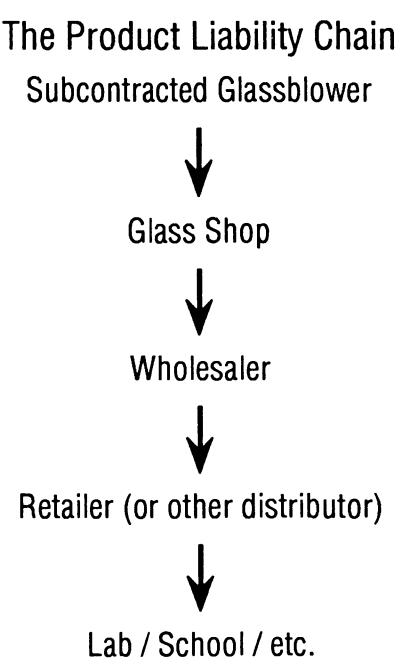

Figure 2

defense. There are limitations to this rule. One who makes a non-defective product is generally not liable when that product is put into a defective product, unless there was reason to suspect the defect would occur. Also, in some cases a supplier's duty to warn consumers may be satisfied by warnings to the manufacturer who incorporates the supplier's part into a product.[27]

There are defenses to a claim of product liability. If the value of the product is significantly greater than the risk and there is no safer alternative available,[28] if the risk is unavoidable and known to the consumer or is the subject of adequate warnings,[29] or if the consumer makes an unforeseeable alteration or unreasonably abuses or misuses the product,[30] product liability may not be imposed. The defenses turn on the facts of the individual case and the laws of the state.

These, then, are the three major theories of liability important to glassblowers. Which theory — or theories — an injured person will assert depends on the particular facts of the accident. Knowing some of the kinds of issues raised under these theories can assist you in taking the precautions to avoid liability.

2. Insurance Issues

The liability theories discussed above apply equally to the employee glassblower, self-employed glassblower, and moonlighting glassblower. However, unlike the employee who is insured through his employer, and unlike the self-employed glassblower who is likely to carry liability insurance on his business, the moonlighting glassblower may face a rather significant risk of liability for which he may have no insurance. Thus, the moonlighter should consider what kinds of liability risks are posed by the moonlighting, how great those risks may be, whether they warrant obtaining a liability insurance policy, and, if insurance is too expensive, whether the risk of liability suggests that the glassblower should cease moonlighting.

These are issues which are highly personal in nature. Without doubt, there will be those of you whose moonlighting income is not sufficient to equal or exceed the premiums on liability insurance. For you, the question is whether to cease moonlighting, reduce the risk of liability by refusing certain types of projects, or simply assume the risk as it now exists. For some of you, the moonlighting income is more than enough to pay insurance premiums, but the remaining profit is not worth the time or expense. For still others, although the income left after buying insurance is substantial, the cost of insurance is daunting. Each of you must make a decision whether to insure, "go bare" (i.e., go without insurance), or close up shop. Regardless of your ultimate decision, it is far better to make that decision based on some knowledge of the risks.

It is strongly recommended that any moonlighting glassblower seek the counsel of an experience commercial risks insurance agent or broker. Whereas the insurance on your home is relatively standardized in its terms and premiums, commercial insurance varies considerably. A basic liability policy likely does not insure against breach of contract, but such coverage may be added. Premiums are often calculated based partly on the size of your business. By narrowly defining which risks you want insured, you may be able to eliminate unnecessary coverage and reduce premiums. For example, if you do not use your car to transport your products, you might be able to delete any automobile coverage from a policy insuring your glassblowing activities. An agent or broker could help explore ways to make insurance viable.

Finally, even if you believe that your moonlighting business does not need insurance, I would encourage you to contact your insurance agent if you are doing glassblowing out of your home or garage. Homeowner's policies are not intended to insure commercial enterprises, and the presence of a shop at the house may affect

your coverage. That is, if there were a fire and the insurance company discovered that you had compressed gasses and open flames at the house, it might well conclude that your claim should be denied or that the policy should be cancelled or rescinded (the contract equivalent of a marriage annulment, rescission declares that policy never existed). This might be true even though the glass workshop did not lead to the loss. Simply put, in matters of insurance and liability, it is important to know all of the facts, and not to proceed on the assumption that nothing can go wrong.

Footnotes:

1. California Civil Code §1549; Restatement 2d, Contracts §1.
2. California Civil Code §1550.
3. Restatement 2d, Contracts §71 (1) (2).
4. Restatement 2d, Contracts §71 et. seq.; Fritz v. Thompson, 125 Cal. App.2d 858, 863, 271 P.2d 205 (1954).
5. Restatement 2d, Contracts §235(2); Sterling v. Gregory, 149 Cal. 117, 121, 85 P. 305 (1906).
6. California Civil Code §3300.
7. Christensen v. Slawter, 173 Cal. App.2d 325, 330, 343, P.2d 341 (1959).
8. Hadley v. Baxendale, 9 Ex. 341, 156 Eng.Rep.R. 145 (1854); California Civil Code §3300.
9. La Rue v. Groezinger, 84 Cal. 281, 24 P. 42 (1890).
10. Lawrence v. Fox, 20 N.Y. 268 (1859); California Civil Code §1559.
11. Restatement 2d, Torts §282; California Civil Code §1714.
12. Toomey v. Southern California Railway Company, 86 Cal. 374, 381 24 P. 1074 (1890).
13. Palsgraf v. Long Island Railroad Company, 248 N.Y. 339, 162 N.E. 99 (1928).
14. Mosley v. Arden Farms Company, 26 Cal. 213, 220, 157 P.2d 372 (1945).
15. Merrill v. Los Angeles Gas & Electric Company, 158 Cal. 499, 503, 111 P. 534 (1910).
16. California Civil Code §3333.
17. Greenman v. Yuba Power Products, 59 Cal.2d 57, 27 Cal. Rptr. 697 (1963); Restatement 2d, Torts §402A.
18. Silverhart v. Mount Zion Hospital, 20 Cal.App.3d 1022, 1026, 98 Cal.Rptr. 187 (1971).
19. Shook v. Jacuzzi, 59 Cal.App.3d 978, 981, 129 Cal.Rptr. 496 (1976).
20. Restatement 2d, Torts, §402A, comment 1.
21. Pike v. Hough Company, 2 Cal.3d 465, 475, 85 Cal. Rptr. 629 (1970).
22. Midgley v. S.S. Kresge Company, 55 Cal.App.3d 67, 71, 127 Cal.Rptr. 217 (1976).
23. Greenman, *ibid.*
24. Thomas v. General Motors Corporation, 13 Cal.App.3d 81, 89, 91 Cal.Rptr. 301 (1970).
25. Baker v. Chrysler Corporation, 55 Cal.App.3d 710, 127 Cal.Rptr. 745 (1976).
26. Vandermark v. Ford Motor Corporation, 61 Cal.2d 256, 37 Cal.Rptr. 896 (1964); Restatement 2d, Torts, §402A, comment f.

27. Groll v. Shell Oil Company, 148 Cal.App.3d 444, 196 Cal Rptr. 52 (1983).
28. Restatement 2d, Torts, §402A, comment k.
29. Restatement 2d, Torts, §402A, comment j.
30. Restatement 2d, Torts, §402A(1)(b).

AN ECONOMICAL AND EASY TO BUILD THIN-LAYER SPECTROELECTROCHEMICAL CELL

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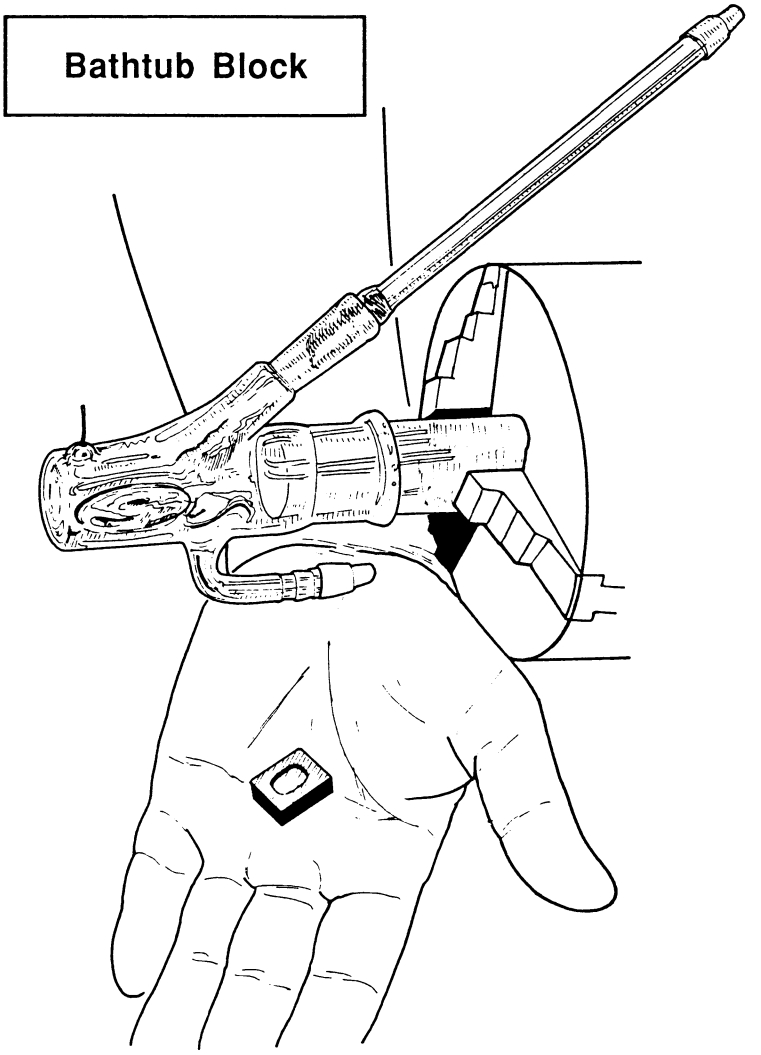
A vacuum-tight all glass thin-layer cell with a platinum working electrode was constructed for spectroelectrochemical studies. The cell body can be made from borosilicate glass for studies in the UV-visible region or from quartz for studies in the ultraviolet and visible regions. The rectangular thin-layer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

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

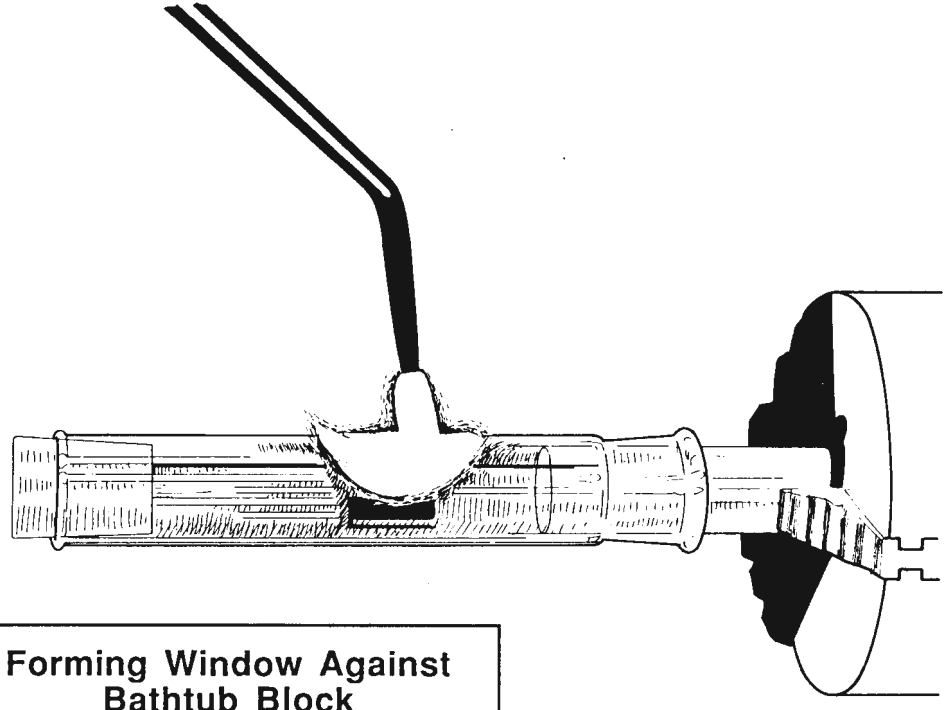
This design of a spectroelectrochemical cell has the advantages of minimum solution resistance (iR drops), durable mechanical strength, freedom from contamination and good experimental reproducibility.

Acknowledgement.

I would like to acknowledge Dr. Karl M. Kadish, Dr. X. Q. Lin and B. C. Han for assistance and helpful discussions with respect to construction of the cell.



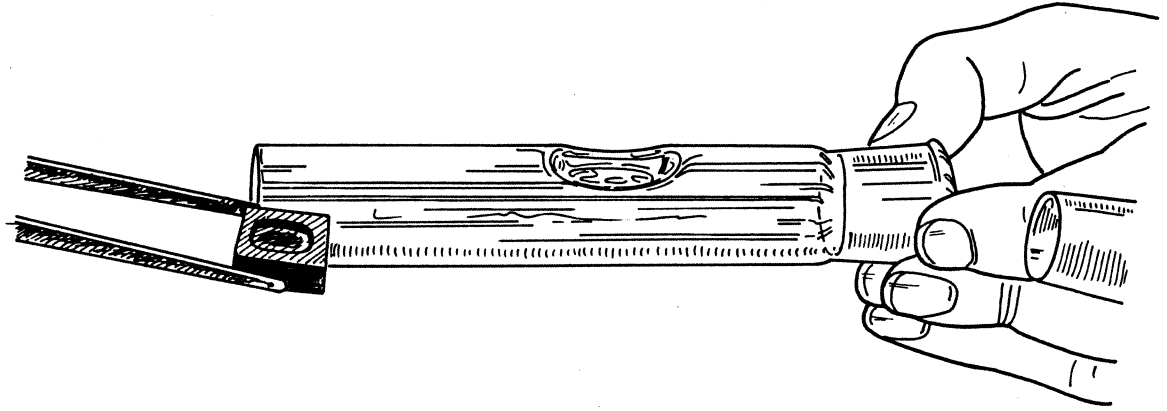
2. THIS IS THE HEART OF THIS CELL — BUILDING THE "BATHTUB BLOCK". IT IS GRAPHITE WITH A SMALL INDENTION MACHINED INTO IT TO FORM THE BATHTUB WHERE THE WINDOW WILL BE FORMED. OUR SPECTROMETER HAS A 2 MM LIGHT BEAM SO IF THE WINDOW IS NOT QUITE FLAT THE CELL CAN BE POSITIONED FOR OPTIMUM SIGNAL.



Forming Window Against Bathtub Block

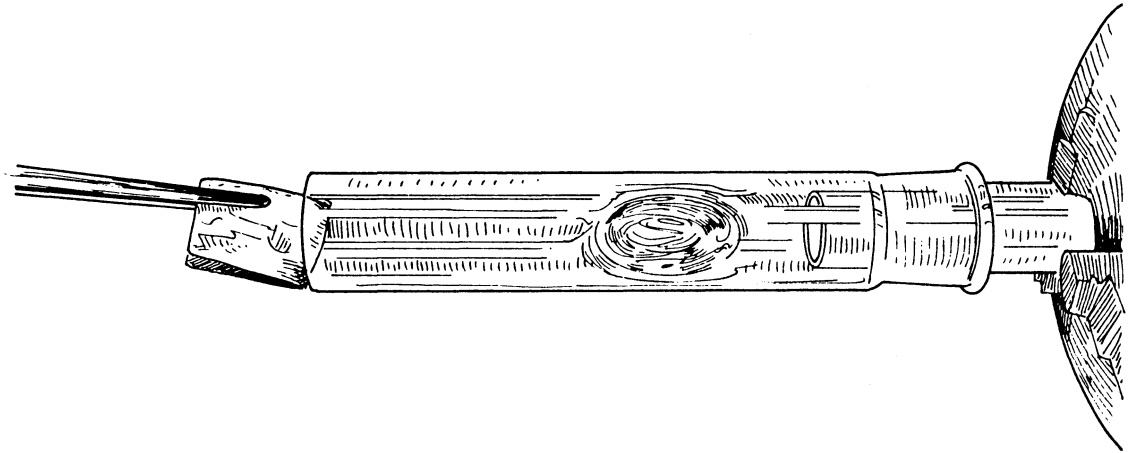
3. THE CELLS WE MAKE ALL START OUT AS 24/25 OUTER JOINTS. THE BATHTUB BLOCK IS PLACED IN THE JOINT WITH A SPACER UNDERNEATH. CORK AND ATTACH TO HOUSE VACUUM. LIGHTLY HEAT OVER THE TOP OF THE BATHTUB BLOCK — THE GLASS WILL BE SUCKED INTO THE FORM. REMOVE THE VACUUM AND LIGHTLY FLAME ANNEAL.

Removal of Bathtub Block



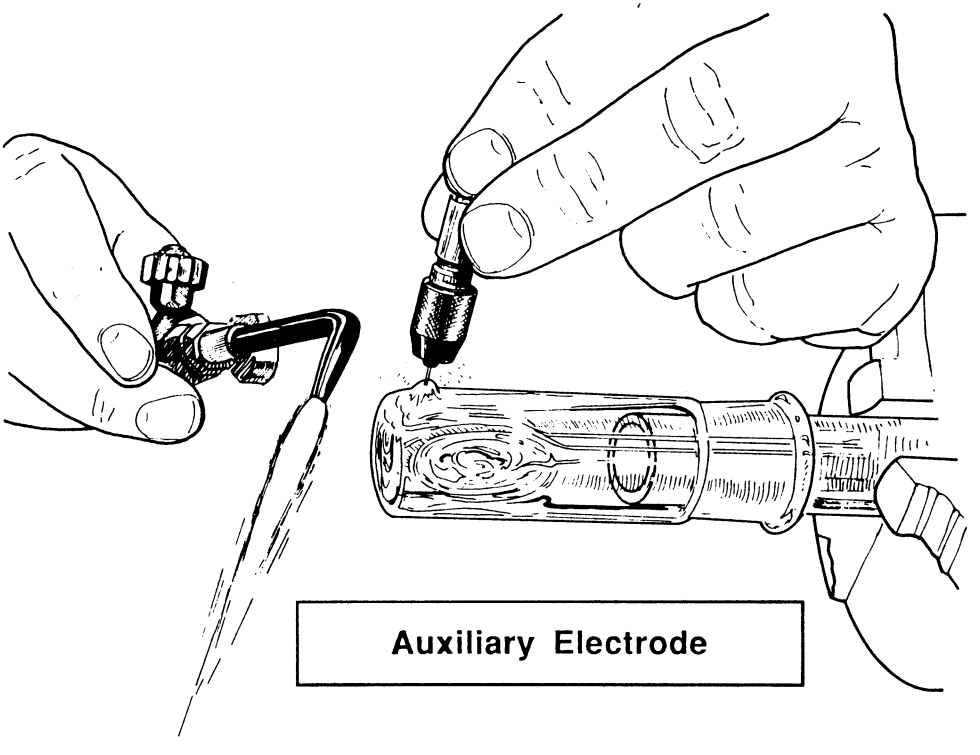
4. REMOVE THE SPACER AND THE BATHTUB BLOCK WILL COME OUT NEXT.

Removal of Graphite Tape Used as .2mm Spacer

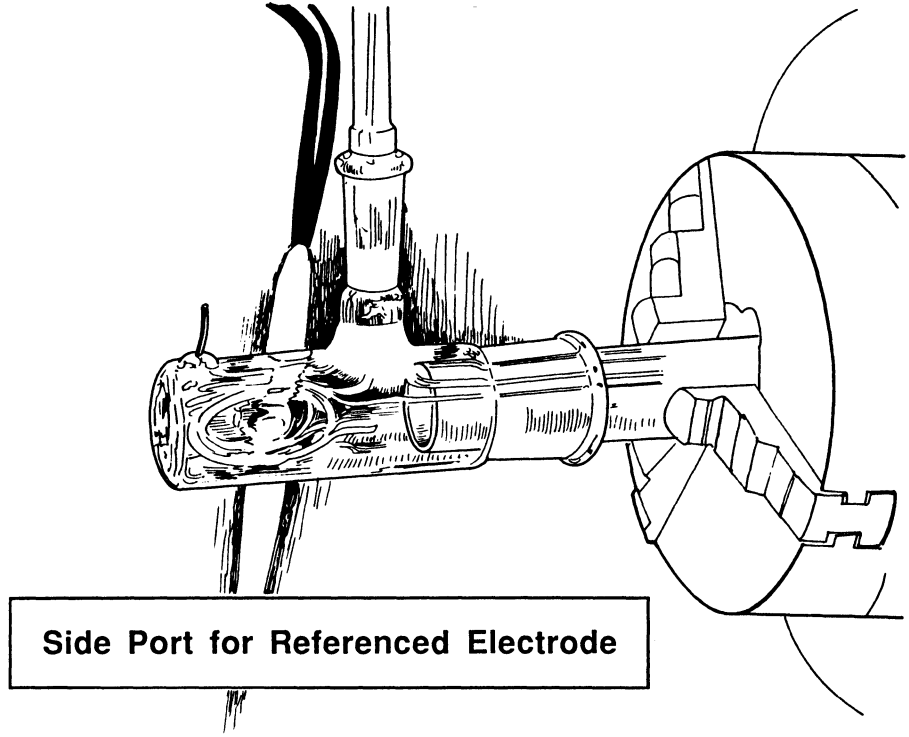


5. THE PATH-LENGTH OF THE CELL BEING PREDETERMINED, GRAPHITE TAPE IS USED AS A SPACER AND THE OPPOSITE WINDOW IS FORMED JUST AS THE 1ST BY EVACUATION AND GENTLE HEATING. LET COOL AND THE TAPE WILL SLIDE OUT.

   CAUTION: EXCESSIVE HEATING WILL CAUSE THE GRAPHITE TAPE TO BECOME A PERMANENT PART OF THE CELL.

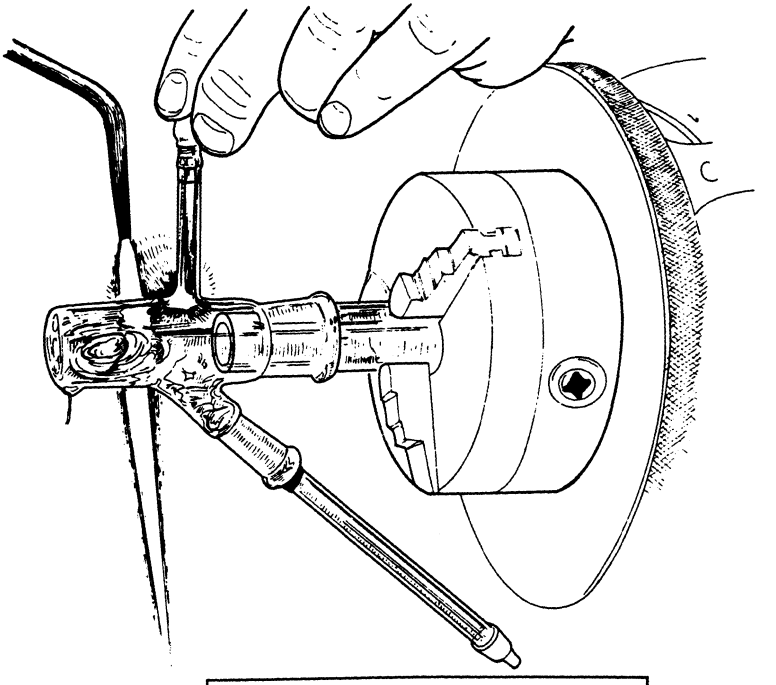


6. NEXT — FLAT BOTTOM THE JOINT JUST BELOW THE WINDOW, AND USING COBALT, SEAL IN A SHORT PIECE OF PLATINUM AS THE AUXILIARY ELECTRODE.



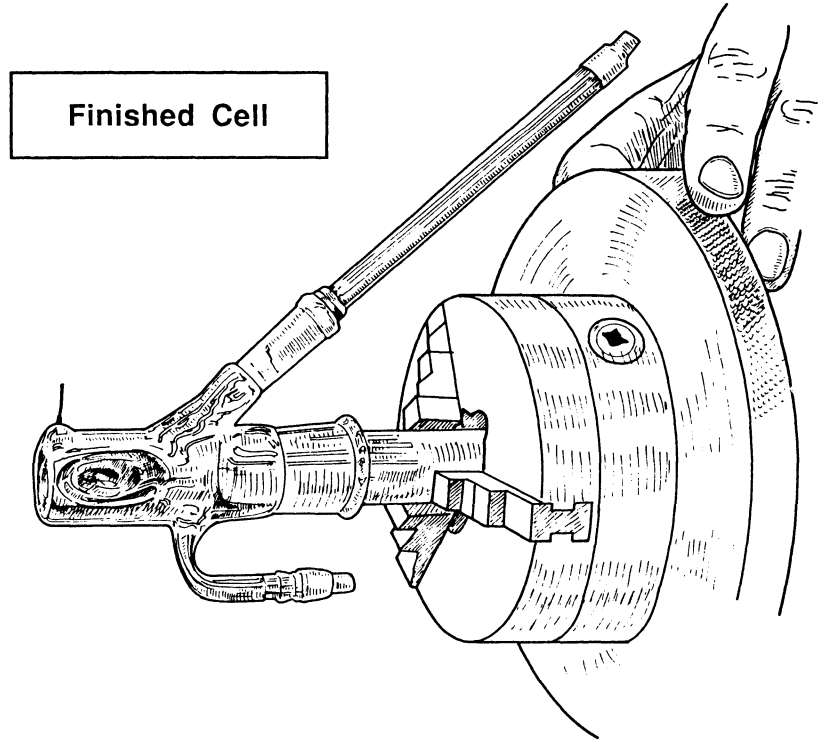
Side Port for Referenced Electrode

7. A 10/30 OUTER JOINT IS THEN ATTACHED FOR A REFERENCED ELECTRODE.

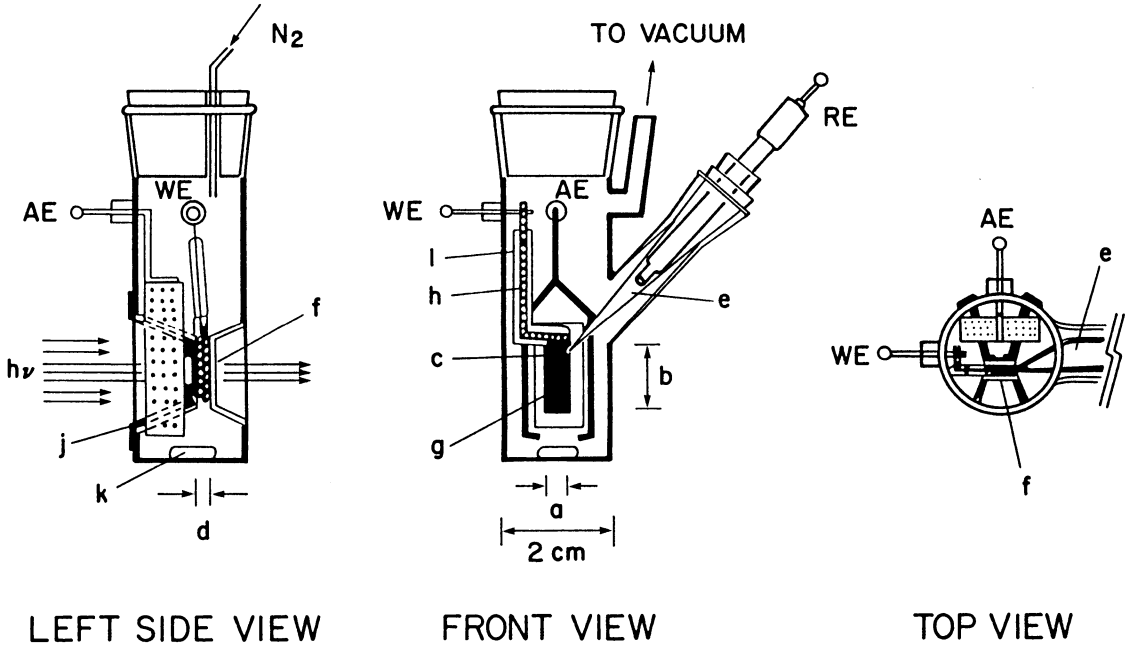


Vacuum Connection

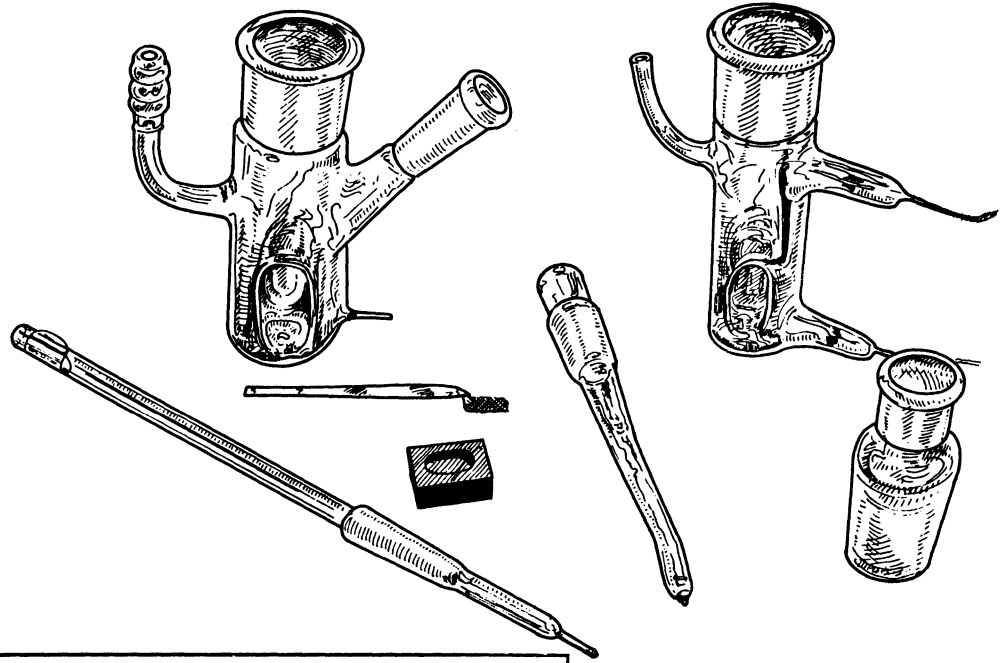
SOME COMPOUNDS ARE AIR SENSITIVE, SO A VACUUM CONNECTION IS ATTACHED.



9. A FINISHED VERY SIMPLE CELL.



10. A CELL WITH ALL ELECTRODES AND HARDWARE.



2 Types of Cells with Electrodes

11. TWO DIFFERENT CELLS WITH SPECIFIC APPLICATIONS.

CARBONFIBER MICROELECTRODE

Allan B. Brown
University of Connecticut

A few years ago teaching labs at the university level started using Mini-Lab glassware in 19/22 standard taper sizes to save on solvents and organic compounds. The teaching labs are now using Micro-Mini Lab kits with 7/10 standard taper sizes for even more savings. Research projects on the graduate school level are now beginning to use the carbonfiber microelectrode in a single droplet of compound. The scientific glassblower is currently being asked to produce more and more smaller glassware for research. I hope this presentation will be of help to some of you.

Carbonfiber microelectrodes are used in analytical electrochemical and biomedical applications. This paper will describe the construction of a microelectrode 4 microns in diameter. This is an unusually intricate procedure due to the minute size of the carbonfiber.

What is a microelectrode? According to Webster's Dictionary, "micro" is defined as "exceptionally little, abnormally small"; and a "micron" is "one millionth of a meter; one thousandth of a millimeter". That is extremely small. An "electrode" is defined as "either of the two terminals of an electric source", thus anode or cathode. Microelectrodes have been made with a variety of materials and in various geometric forms. Microelectrodes are made in various assortments of shapes such as disk, line, cylinder, ring, microsphere and hemisphere and tunneling tips. Sizes may vary according to demands. In the manufacture of microelectrodes the most commonly used materials are silver, platinum, gold and carbonfiber, in this order.

The applications described herein are applicable for all of the above-mentioned materials and shapes except microsphere and hemisphere. Remembering exactly how small a micron is, it is easy to understand how painstaking it is to seal only one carbonfiber into a piece of glass without burning it up! Equally difficult is the fact that in most cases you need a microscope to even see the single carbonfiber. When the size of the wire or fiber is less than 10 microns, the sealing becomes difficult and the quality of the microelectrode tip becomes very critical. Typically, a 100% success rate for microelectrode fabrication is neither achieved nor expected. The following procedure should produce carbonfiber microelectrodes better than 80% of the time.

The materials used to produce carbonfiber microelectrodes as discussed in this paper are P55-s grade VSB-32 carbonfibers, donated by Union Carbide Corp., and Corning's 7740 glass tubes. The step by step procedure will be supported by Figures 1 through 11. A 6 mm O.D. Pyrex tube approximately 12 cm long is first heated (Step 1) and pulled from one end, forming the tube into a pipette shape (Step 2). After cooling, the pipette tip is scored and snapped off exposing the tip with the inside diameter of approximately 1 mm (Step 3). Attach a blow hose and a swivel to the 6 mm O.D. end of the pipette and seal off the pipette tip; then gently blow the end open forming a funnel shape. Carefully select one carbonfiber and insert it approximately 4 cm into the pipette through the funnel end just completed (Step 4). Remove the blow hose and replace it with a cork. Next, seal the pipette tip closed making sure that the carbonfiber is firmly sealed into the end (Steps 5 & 6). The cork prevents the carbonfiber from being sucked out of the pipette tip by the velocity of the fire during the sealing of the tip. Before proceeding further, take the electrode and examine the tip under a microscope to insure that there is only one carbonfiber sealed into the end. Using a mechanical vacuum pump, place the pump hose over the 6 mm end of the carbonfiber microelectrode (Step 7). Slowly rotate the carbonfiber microelectrode tip in a small pin fire (oxygen-propane) while evacuating it (Step 8). You will learn that

you must work out at the very very end of the fire and sometimes you will wonder if the carbonfiber microelectrode is even being heated. Looking closely at the area that is being heated you can watch the glass seal down onto the carbonfiber. Care should be taken not to allow the carbonfiber to glow red; if it does, throw it away and start again from Step 1. Start heating approximately 1 cm from the tip. While slowly rotating the electrode, you can watch the glass seal onto the carbonfiber. Next, carefully work your way back towards the 6 mm diameter end. The sealed area when completed will be approximately 15 mm in length (Step 9). When the carbonfiber microelectrode is cold, examine the tip under a magnifying lass or microscope to determine if it contains any trapped air bubbles. If you find trapped air bubbles, you have not heated the tip evenly enough. Uneven heating causes cold spots which prevent the glass from shrinking down uniformly onto the carbonfiber, thus causing trapped air. If there is a uniform seal on the tip, score and snap the tip of the electrode off exposing the end of the carbonfiber (Step 10). Grind the polish the tip with a Buehler Carbinet Silicon paper of grit number 600 using lots of flowing cold water (Step 11). The carbonfiber microelectrode is now polished flat. The next step is to add a contact wire of choice through the 6mm. end using a drop of mercury as a conductive material between the carbonfiber and the contact wire. The procedure is now complete.

Looking at the very bottom end of the microelectrode tip towards the carbonfiber, the latter appears in the shape of a disk, hence the name carbonfiber disk microelectrode.

You can only make a few carbonfiber microelectrodes at a time. Trying to separate individual carbonfibers and insert them into pipette tips is exceedingly tedious. I believe, however, that you will find this technique extremely useful in microelectrode production.

DIFFERENT APPROACHES TO REACTOR DESIGNS

John W. Squeo
Amoco Research Center
Naperville, Illinois

The Chemists Cecilia Radlowski and Lisa Green were getting poor circulation in the bottom of their "fixed bed reactor." (SLIDE 1) In this approach, a tube comes down the center of the reactor, injecting a liquid or liquids, and gas or gases, to fluidize their bed of catalyst. They wanted to change from this system to what is called a "fluidized bed reactor" where the catalyst bed is supported by a frit and the liquids and gases (hydrocarbons) would be introduced into the catalyst bed from under the frit. By switching they felt they would get different product selectivity and a more uniform temperature. They also desired good gas and catalyst mixing, without blowing the catalyst out of the unit. You see a bubble could form under the catalyst and raise it up and out of the reactor.

(SLIDE 2) You can see in slide 2 the different fluidization regimes that can happen.

(A) bubbling - best fluidization where the bubbles are quite small. This gives maximum contact with the catlyst.

(B) slugging

(C) channeling

(D) jetting, and

(E) spouting; all have minimal fluidization

They wanted glass so they could:

(1) measure bed heights

(2) watch what was happening as they varied the gas flow

(3) allow them to calculate gas flows necessary to achieve different fluidization regimes

Ideally they wanted a stainless steel frit for the following reasons:

(1) a known pressure drop across the frit for proper fluidization

(2) it won't degrade under process conditions

(3) they knew the particular frit they had in mind would hold up catalyst particles, 11 microns or greater.

(4) it would be the same frit that would end up in the all stainless steel reactor they were planning in the future; and would be able to see exactly how it will be working in the future reactor now; equalling more accurate results.

They wanted the new reactor:

(1) to be made of quartz - because of furnace temperature

(2) screw into a unit that they presently had - without moving anything. (The top of the reactor was to screw into a unit and fit the furance position.)

(3) to have inlets for liquids, and gases (hydrocarbons), to come in from the bottom of the reactor to mix; travel up the reactor in the furnace, turning the liquids into vapors, thru the stainless steel frit to fluidize their catalyst bed.

(4) flexibility to change their frit if desired

Starting from the top (SLIDE 3). I had the stainless steel screw part welded to a S.S. to pyrex seal. Then sealed that to a 28mm o.d. pyrex to quartz graded seal. I had only 4" to work with from the knurled part of the screw, to the top of the existing

furnace. Being unsure of how the graded seal would react, I wanted a 3/4" space between the bottom grade and the top of the furnace; and yet the welder needed some space between the glass and his weld.

Moving down to the S.S. frit, which they didn't want to leak...Basically I weighed up 28mm o.d. quartz; but keeping the o.d.; 28mm Then ground a seat using a grinding tool, (SLIDE 4) designed to give an apx. 1/8" ground ledge for the S.S. frit to rest on, and a hair larger in o.d. than the S.S. frit which was to be inserted; because the S.S. will expand in the heat and the quartz won't. Bob Bentanear, an engineer, reminded me. He also suggested we slightly champer the inside corner of the seat. Please note on slide 4 the dimension .015 x 45° circled, with an arrow pointing at the champer on the tool. Above the print of the tool, is the print of the frit. Please note the dimention; "break" .020 x 45°, with an arrow pointing to a corner of the frit. Bob didn't think the grinding tool would be able to duplicate the preciseness the S.S. frit could be cut to (almost perfectly square); So these slightly angles were built into the tool and frit.

I purposely ground the seat from the bottom of the reactor, because the gas flow would be coming from this direction, and this would help push the frit into the ground seat. To keep the grinding tool straight and aligned during the grinding process - so a nice flat seat would result; I rolled masking tape onto the shaft of the grinding tool in one spot, just large enough to fit into the 25 x 28 quartz tube which was weighed up. The placement of the tape was apx. 1 1/2" under the knurled handle of the grinding tool.

(SLIDE 5) The S.S. frit was held in place with one long quartz - what I'll call "pin" - that would hold the frit snuggly in place; but at the same time, flex, to allow the expansion of the S.S. frit. Notice it is curved to allow some what of a spring action. Where I secured the pin to the 25 x 28 quartz, needed to be apx. 1 1/2" away from the frit; because when I sealed it in place, 1" away from the frit; the frit picked up the heat and expanded. (SLIDE 6) Even though it was tight when the operation was completed; when the frit cooled, it was loose; and needed to be tighter. Even though it would be operated at high temperature, and would snuggly fit then, I just wasn't sure; and I wanted a tight fit. The pin pictured in this slide could use a bit more curve for spring action.

Now moving to the bottom part of the reactor. The chemist wanted only two connections into the reactor from the bottom; one inlet for gases and one for different liquids to be introduced into the system. They also wanted everything to be mixed thoroughly and in a vapor state by the time it got up to the S.S. frit. I felt the way to go was to inject the gases and liquids into the system through 2 jets, with i.d.'s of less than lm.m; aimed at each other (SLIDE 7) so the gas injector and liquid injector paths crossed apx. 1/4" away from their outlets. When I tested this, the gas turned the liquid into a fine mist, and I felt the heat from the furnace would turn that mist into a vapor by the time it reached the frit. I also felt if I put these jets on an outside edge of the tube the jets are mounted on; I'd get an swirling effect up the tube, causing more mixing and turbulence inside the reactor. An added benefit of this is that the jet stream could be aimed to hug the inside wall of the reactor where the heat is more intense. There then was a consideration in my mind of how close to the frit to place the injectors, to get the best results for the chemists. An adjustable height seemed to be ideal in my mind; so by extending the reactor beyond the bottom of the furnace; grading from quartz back to pyrex, then sealing to a #25 ACE thred (SLIDE 8) the quartz, adjustable height injection system was possible. The liquid or liquids were introduced under pressure through needle valves connected to the 5mm i.d., 7mm o.d. center tube, which turns into a helix; which slows down the liquid so as to preheat it, before exiting out the injector. The gases were introduced thru the outer jacket of the

system. A "stop" was attached to the outer jacket of the injector system, so the jets would never accidently touch the pin which holds the S.S. frit in place during adjustments.

The chemists said that the liquids actually turned into a vapor before they got out the end of the jet. The helix worked out better than I hoped for.

(SLIDE 9) This system was designed and built in 1988. The chemists now have three, fluidized bed reactors; completely made out of stainless steel. They are copys of the reactor design, with the adjustable height injection system; and they are running now.

The next case, is a reactor which was all quartz (SLIDE 10) with the exception of the two liquid inlets at the top of the reactor which were graded to pyrex, to be able to seal a special pyrex connector - that connects to 1/8" o.d. teflon tubing.

A gas was entering the reactor, thru the 18/9 ball joint at the top of the reactor. One of the problems was the liquid pumps didn't have a perfectly steady flow, and this caused "pulsing". Droplets of liquids would end up running down the inside wall of the reactor into the catalyst bed, where vapor was desired. Also condensation formed in the part of the reactor not inside the furnace; which would also after accumulating, drip down the inside wall of the reactor causing "pulsing". This "pulsing" caused the data to vary. The chemist, Jin Yoo, and technician, Rafi Sohail, wanted a steady flow, no condensation, and no liquids to touch the catalyst bed. Besides this, Rafi said the quartz, extra course frit was clogging up very often.

The first thing I changed was moving the two graded seals from the side arms. I used one graded seal on the reactor body - just before it exited the furnace on top, where 2" of the heat brick insulation is, and put another on the thermowell. I also put another graded seal on the reactor exiting the furnace at the bottom, in the heat brick area (not where the heating elements are).

Next I remembered some interesting material called Celcor, made by Corning. It is a honeycomb ceramic support, made of magnesium, alumina, and silica oxides. It cost $48.00, in June of 1989, (when this reactor design evolved), for a piece 1" high x 3" wide x 6" long. In the piece we ordered, there are 400 holes per sq. in.; which was bigger than the catalyst it was to support, also available is Celcor with 300 holes, 200 holes, and 100 holes per sq. in.. There was a purer honeycomb material available from Corning with 900 holes per sq. in., called "Lithum, Alumina, Silica"; but its cost was $275.00 for a piece about 2 1/4" in diameter and 1" high. It's now being made with 1300 holes per sq. in.. After talking things over with Jin Yoo and Rafi Sohail, the following things were noted:

(1) The holes in both materials were larger than the catalyst. (900 holes per sq. in. was still almost twice as big as the catalyst)

(2) Cost difference between the Celcor and Lithium, Alumina, and Silica

(3) It was a new idea and I wasn't even sure if either would seal to the quartz - which I had in mind.

(4) A thin layer of quartz wool could sit on top of the Celcor - if I could get it to seal to the quartz reactor, (to hold up the catalyst bed).

Charles Camilo, a Senior Sales Engineer, was very helpful in faxing data and literature to me; and in verbal communication he also got the Celcor for me to work with extremely fast. His phone # is (607) 974-4085.

Somehow I got the idea to crisscross two sliced sections of Celcor, to try to hold up the catalyst, instead of using the quartz wool on top of one layer of Celcor. Rafi liked

this idea, however, his critera to me was for the overall thickness of the frit to remain 1/8". My goal then was to make two frits apx. 20mm o.d. and apx. 1/16" thick each. I discovered trying to get the Celcor that thin wasn't the easiest thing to do; you see the edges of the honeycomb crumble quite easily trying to slice it 1/16" thick.

(SLIDE 11) The best approach that worked for me, was first core drilling, with a diamond core drill (and water). This went almost as easy as a hot knife through butter. I then sliced sections out of the core piece, with a diamond wet saw, almost 1/8" thick. Then using a fine grit diamond sander (attached to a foam block) with water; I proceeded to sand the excess material off the frit.

I'd like to note here, there are chemists at Amoco Research Center that use similar Celcor materials. I've seen up to 3" tall sections, apx. 1/2" to 1" in diameter; and held in place in their reactors, with just indents.

(SLIDE 12) Here is a reactor the chemists wanted to assemble and diassemble quickly, and liked the idea of the Ace Threds and adjustable thermowells, which will hold various lengths of Celcors in place. They can, whenever they desire, put a shorter or longer piece of Celcor in the reactor without visiting the glass shop for an adjustment.

(SLIDE 13) Here is a 2 foot tall drying tower. The technician wanted a glass grid sealed inside of a 140mm o.d. tube, with a 9mm thick wall. He wanted the grid to support 36 lbs. of materials. By just dropping a 1" or 1 1/2" thick piece of Celcor into the bottom of the drying tower was not only cost effective but easier, as you could imagine. Besides these benefits I didn't have to worry about the possibility of the pyrex grid failing during a run. I felt very confident. I did however, round the edge of the Celcor frit where it rests at the bottom. I did this with a diamond belt sander.

I will now describe how I sealed the Celcor inside the quartz reactor.

(SLIDE 14) I first tried sealing one Celcor frit in on the lathe with crossfires. As you can see here, this approach proved to be just a tad too much heat for it. The best way I can describe it is that it seemed to bubble, disolve, or vaporize. It melted right into the inside wall of the quartz, leaving a gap.

In my second approach, I started by making a slight knockdown in a 25 x 28 quartz tube on the lathe. Then holding the tube vertically on a ring stand; positioned two 1/16" thick Celcor frits in a crisscross fashion.

(SLIDE 15) If you look closely at the slide, you'll be able to see the bottom frit under the top one. Then with a hand torch, I heated a spot, apx. 1/8" diameter, on the quartz; and with a rounded narrow carbon rod, gently pushed the molten quartz in to tack the delicately positioned crisscrossed Celcor frit. I then went 180° to the other side, and did the same thing. (SLIDE 16) Then continued in a similar fashion until the entire double frit was sealed in. I did hear something like cracking when I was finished, and the quartz cooled off. It turned out to be only a couple of chips of glass that popped out from the inside wall. The seal was airtight and acceptable to my clients. I ran it thru the oven a couple of times as a test; it was still fine.

By the way, the catalyst with an o.d. of apx. .425 of an inch didn't pass thru the crisscrossed frit, without the use of quartz wool, as I hoped for.

The next problems that had to be addressed were the pulsing of the liquids; and the condensation in the system. (SLIDE 17) In the original designs (pictured right), the gas and liquids come into the reactor thru separate side arms. The i.d. of the gas inlet was 9mm, thru the 18/9 ball joint. The only place the condensation was occuring was above the top of the furnace.

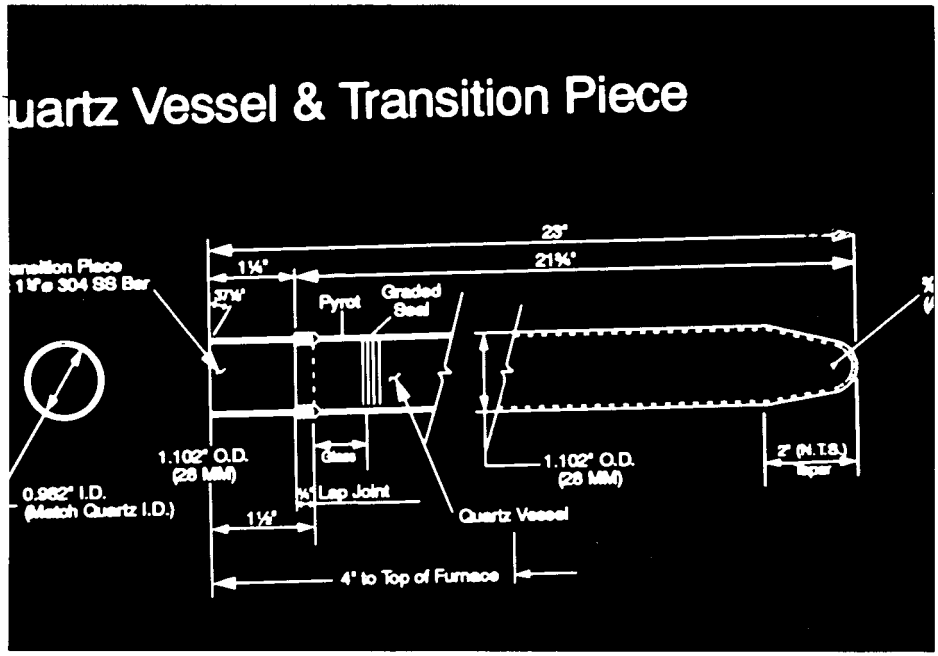
Out of a few designs I came up with; Jin Yoo ok'd the fabrication of the design pictured on the left (of Slide 17).

I felt one way to cut down on the condensation was to cut down on the space it was in. The original design the top of the furnace came up to 1" below the 18/9 side arm. In the new design the top of the furnace came up to the bottom of the 24/40 joint. I then moved the 18/9 gas inlet to the highest spot in the reactor. Then put the two liquid inlets into the space where the gas was traveling. I also put in that space some quartz wool, so the liquids would hold up in that space; giving the gas some time to turn them into a vapor. I put 3 indents in the gas flow route after the liquid inlets, to make sure the quartz wool didn't go any further and clog the new injector. I knew by decreasing the 9mm i.d. gas inlet (as on the old design), to a small hole; and by curving the end of the injector (SLIDE 18) around the 6mm o.d. thermowell; would cause a swirling of the gas mixture going down the reactor; thus eliminating more of the condensation. By adding the flexability of a #15 Ace thred; I allowed Rafi to play around with the angle the injector was going to "shoot" the gas stream, so he could find the best angle possible for eliminating the condensation. The 6.m.m o.d. thermowell needed to be quartz in the reactor; and also I wanted the pyrex Ace thred to be sealed to a pyrex 24/40 male because it would be easier than going to quartz in this area; so the graded seal was put on the thermowell, above the top of the furnace; which is located at the bottom of the 24/40 joints.

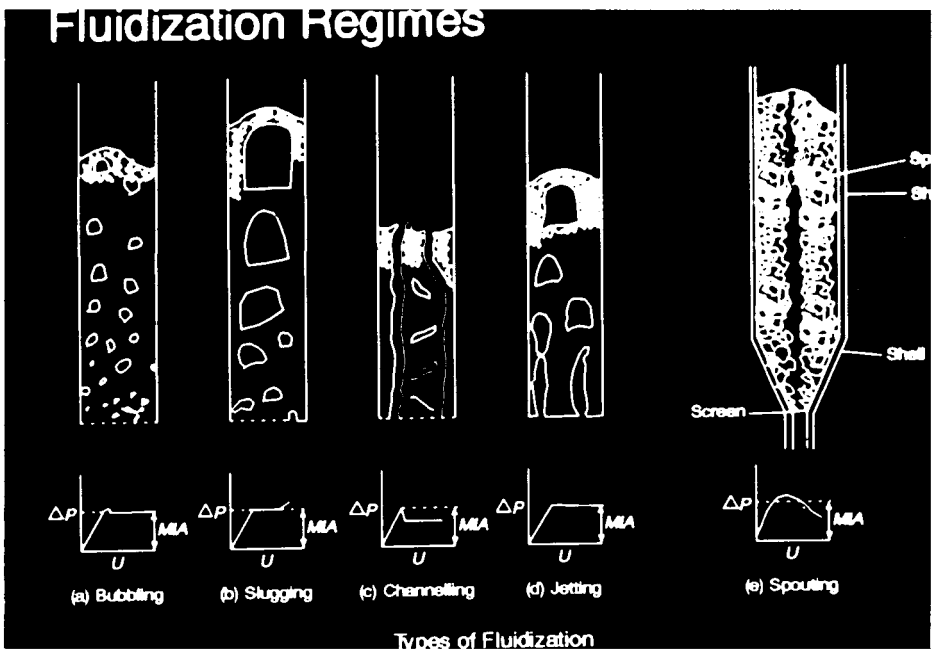
I felt if this didn't take care of the condensation and pulsing problem, this would:

Starting where the condensation made it's home just above the furnace, using 1/2" wide heat tape, begin wrapping the 24/40 joints. Continue wrapping the heat tape around the bend, the Ace Thred and where the quartz wool is holding up (SEE SLIDE 17) the two liquids to be vaporized. The two liquid inlets are separated wide enough for the heat tape to pass between them, at an angle. Continue wrapping over the 18/9 joints and up the gas feed line to preheat the gas, even before it enters the glassware; thus making an ideal environment for the liquids to vaporize in.

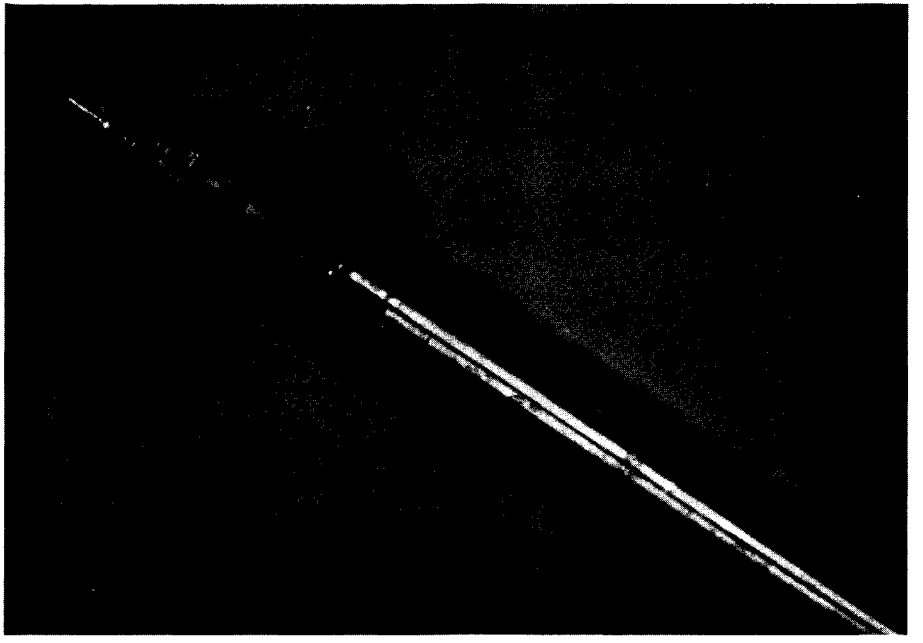
Rafi, the technician, could control the heat tape temperature to whatever was necessary to do the job, for maximum performance. Eventually Rafi found the ideal angle for the injector to be. (SLIDE 19) The next reactor Jin Yoo wanted the #15 Ace Thred eliminated; as pictured in this slide on the right.



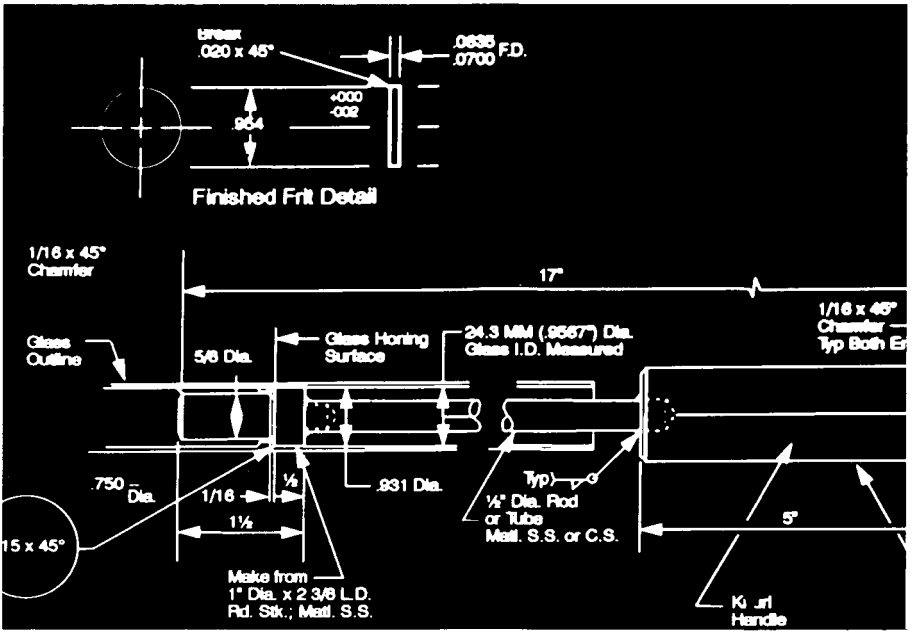
Slide 1



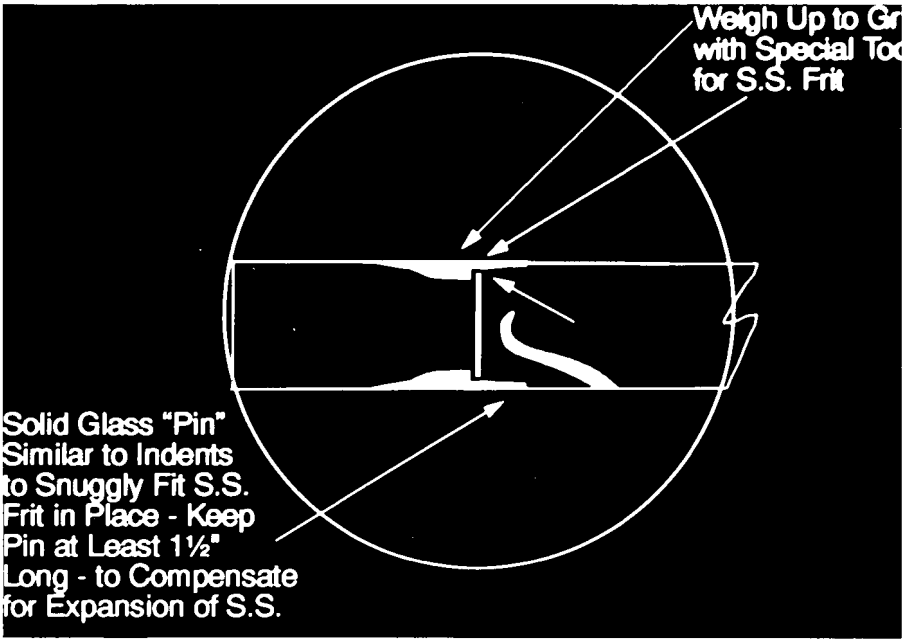
Slide 2



Slide 3



Slide 4



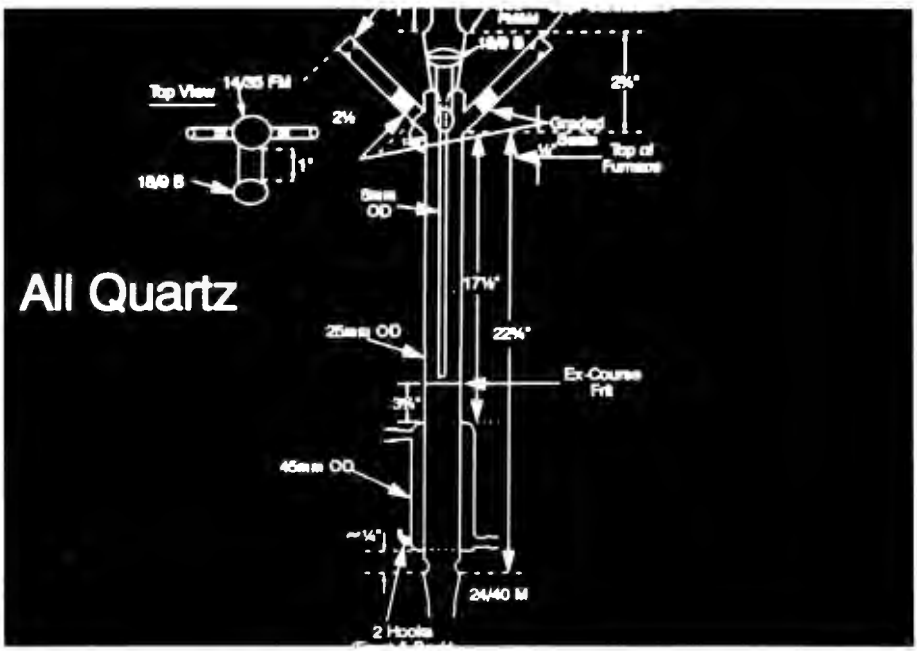
Slide 5

Slide 6

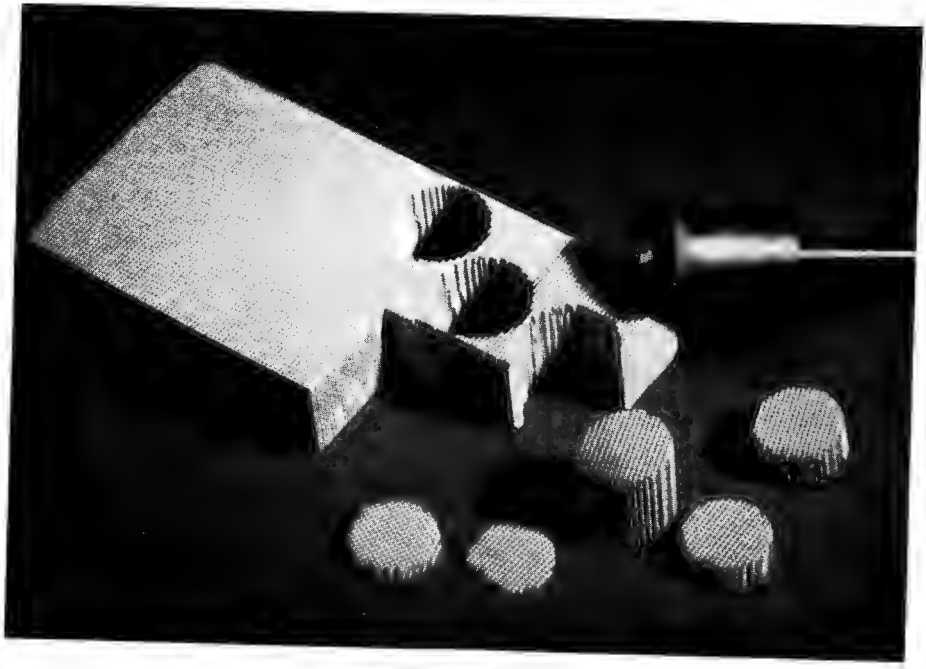
Slide 7

Slide 8

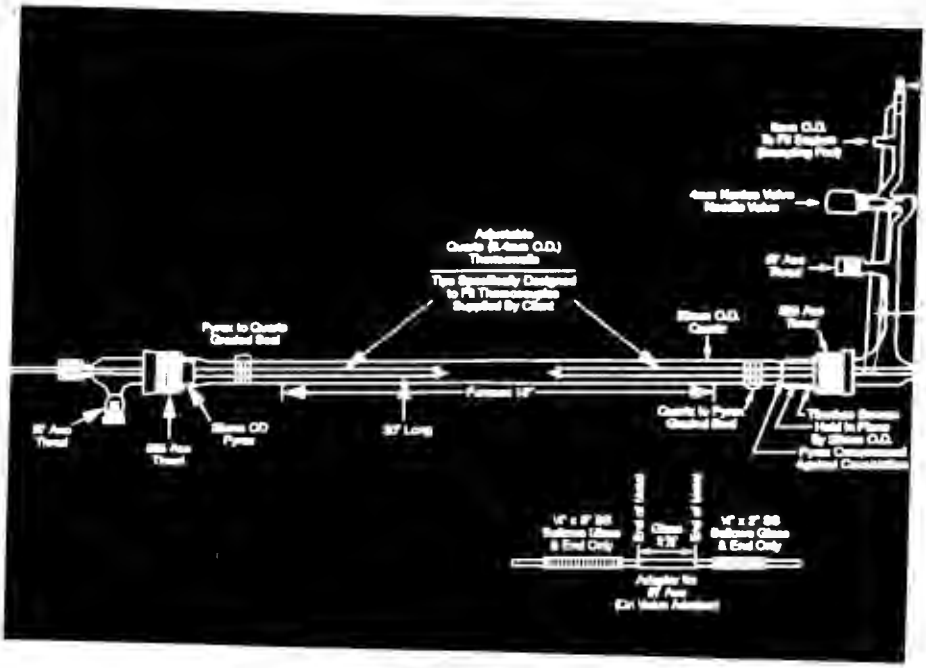
Slide 9



Slide 10



Slide 11



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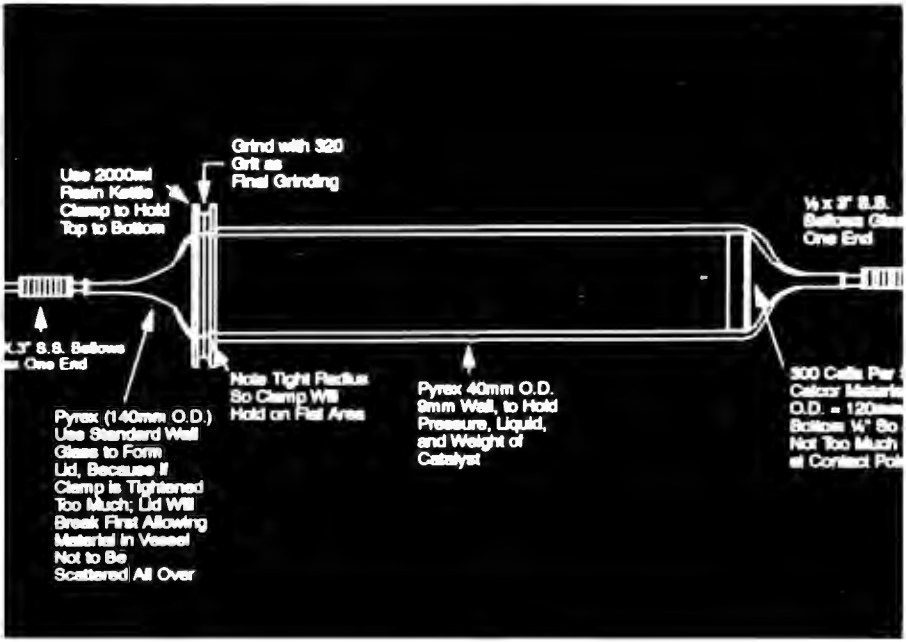

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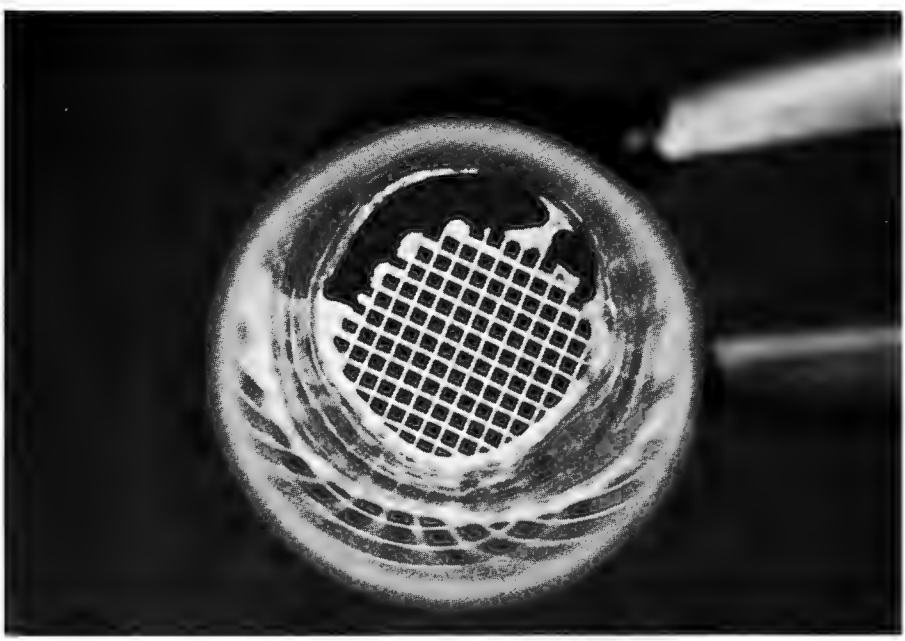

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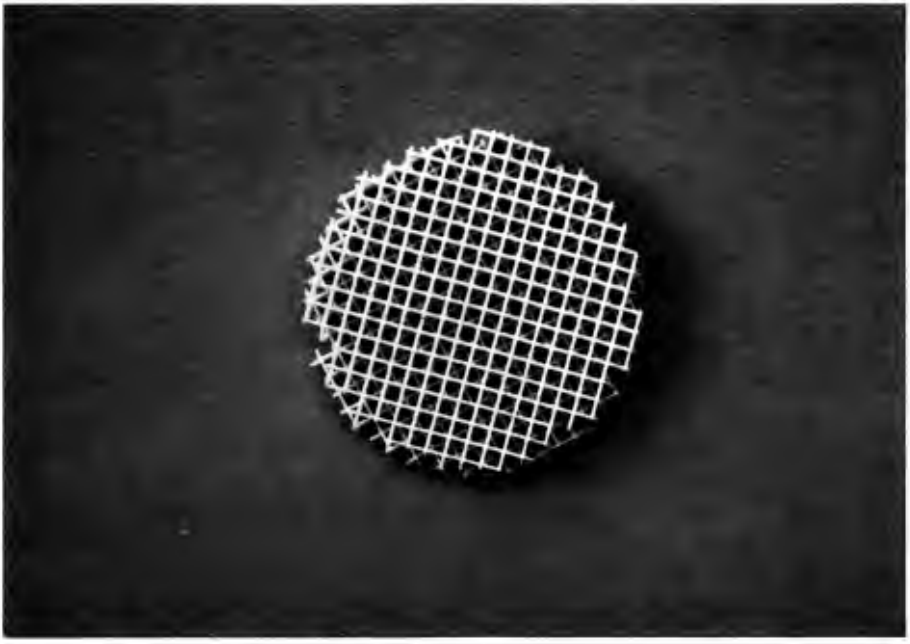

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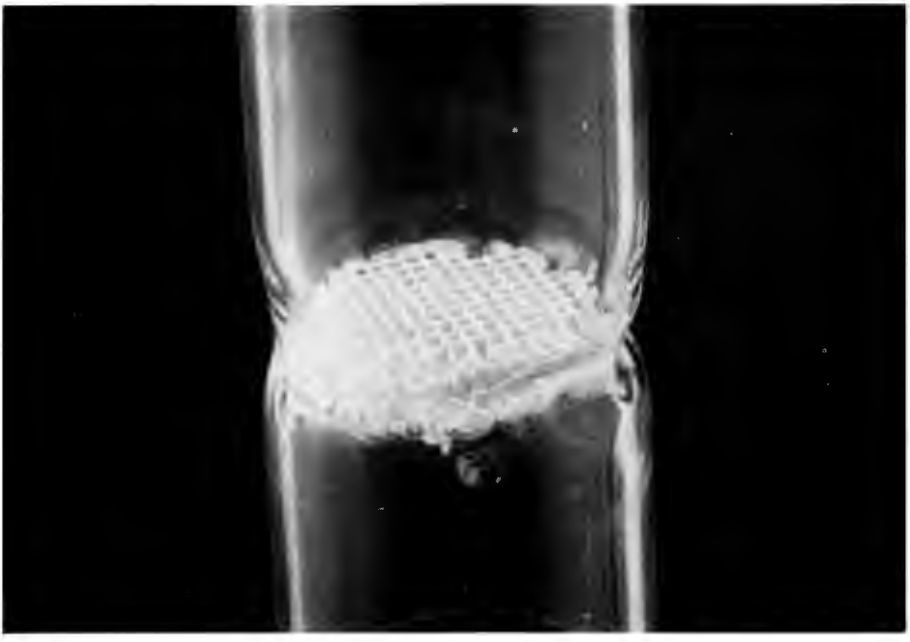

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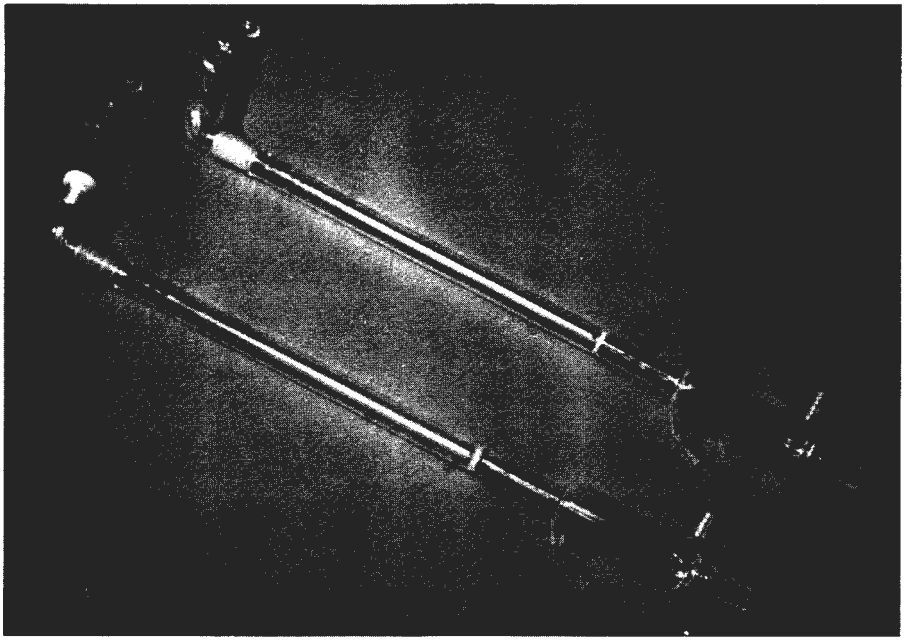

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