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THE THIRTY-FIRST SYMPOSIUM
ON THE
ART OF GLASSBLOWING

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THE AMERICAN SCIENTIFIC GLASSBLOWERS SOCIETY
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"INVOLVING FAMILY IN OUR DAILY BREAD"

Shirley Fake Platt
DYNA-CUT INC.
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Involving family in our daily bread is a pure and simple matter of communication. We all do it whether we intend to or not. It is, at its best, the kind of communication that affords relief from stress, releases our creativity, and increases our productivity. It can be the kind of supportive pulling-together such as has effected the success of The American Scientific Glassblowers Society, not merely man-on-man but family-by-family.

Family involvement in its livelihood has its roots in antiquity; since the dawn of time and Eve's rendezvous with the snake by the old apple tree, keeping food on the table, clothes on the back, and wolf away from the door has required the united effort of the entire family. Every culture has its traditional familial transmission of survival skills to subsequent generations. Many family genealogies and our names themselves reveal to the present generations the predominant craft of our ancestors. Miller, Hostler, Cooper, Farmer, Smith, Glass, etc. . . Many of these were closed trades and one learned them only having been born, married or indentured into them. Where several like-skilled families banded together into a guild, their trade secrets were preserved and perpetuated within its confines, its membership held within its bounds as tightly as its technology. Few comprehend this all encompassing view of family involvement better than the glassblower who for centuries throughout the Old World and then in the New World protected his family, his livelihood and his technology within a guild system.

Times have changed and so have the circumstances and attitudes of society in general toward family involvement in the marketplace. It would appear that sharing our work with our family, let alone family involvement in the workplace, nowadays is the exception rather than the rule. More accurately, though, that involvement has assumed new and divergent definition, a more sophisticated commitment. I believe there is no better profile of this than that reflected among the modern society of scientific glassblowers.

It was a statement on this very matter which had hit home with quite a few of us in a recent Section discussion, set my brain spinning overtime, and provided the genesis tor the study upon which this paper has developed; i.e. –

"I'd like to know just how many of you take work home to your family!"

First hand, I have experienced not only bringing work home to family, but living with it twenty-four hours a day. There has been quite a mixed bag of familial, economic, psychological, and educational blessings for us and our three children because of having Bob's workplace a "roll out of bed" away and the "whole catastrophe" being needed on-hand in or near the exhibit hall during the symposia. It's having a definite impact upon the career choices of our children, just as Uncle Allen Alexander's "taking work home" has had upon the direction Bob's life has taken and the blessing we all have come to know because of it. I could list endlessly benefits of traditional family inclusion in the family livelihood, as well as the handful of stresses and headaches they render bearable. Many others of you can do the same and better.

Among our close friends and acquaintances about the exhibit hall and in the Society in general, I am aware of an impressive representation reflecting a tradition of family togetherness in the workplace, in some instances beyond the fourth generations. Many of you live with it day and night; your children cut their teeth

on the tools of our trade, and spend in a day's time as many hours underfoot in the workplace as most youngsters spend plugged in to the TV nowadays. But even more of us work in relatively large companies and universities and manage to involve their families thoroughly without that ease of access afforded by either the Family Business or the small college lab. Some of us, however, prefer to maintain a complete separation of work and family life, yet even this is bringing "work" home to family. We all do it somehow or other, regardless.

The inquiry needed more than my own experience or observations alone would afford. It had raised additional questions in my own mind as well. Hence, throughout the spring of 1986 a survey was circulated among the membership of the Society in an attempt to ascertain the extent and nature of family involvement among us, its impact on our families and our profession, and our viewpoints in that regard. By hand and mail at least 225 of you received copies of a questionnaire which went out pretty much at random into every Section; 13% returned them, commenting at length. Numerous others made direct comment both in-passing and at length in very informative discussion right up to the day of the oral presentation of this paper. And as many have extended comment subsequently.

Your response to the survey indeed has exceeded my anticipations. During this busiest time of your year, many of you have given most generously and thoughtfully, expressing your opinions, your enthusiasm, your concerns and disappointments, but mostly your joys. You've presented a potpourri of diverse opinions and practices while agreeing on one thing most noticeably, your attitude toward your work. The long renowned singer Peggy Lee once described success as "loving your work". At that rate my survey reflects the opinions of the most successful group of people in the land.

Many of you who have responded to my survey appear to indicate the critical importance of scientific glassblowing as you speak of the great pride and satisfaction you feel over having participated directly and indirectly " . . . in many scientific accomplishments that have (had major impact) on world history, i.e.: lasers, solid state devices, transistors, micro wave tubes, magnetions, Klystron Traveling Wave Tubes, Tel Star Communications Satellites, medical research, biology, geology, chemistry, physics, etc., etc. . . ." While some of you are discouraged over "how some clients put (your) work to use, eg. Starwars and animal experimentation," you all communicated feeling strongly enthusiastic about your providing valuable service to research, to industry, to mankind.

Not a one of you would dispute the value of scientific glassblowing to the welfare of our world. But would you likely be as astonished as I had been learning that research into this paper has been only the second reference to scientific glassblowing our upper-county librarian has known of in more than thirty years? She expressed concern over what she sees as diminished opportunity for young people today to develop special skills and be inspired and guided into challenging occupations such as her high school friend received from an elderly Delaware neighbor who had taught him scientific glassblowing "back in the days when glassblowing as an occupation had been unheard of." Our discussion suggested, because of our modern wide-open but compartmentalizing and insulating social situation with its defensive attitude over strangers and neighbors, risks and liability, our youngsters are left with the media, school, and their families from whence to discern the array, skills, attitudes, value and appreciation of work. Will scientific glassblowing as an occupation continue to be unheard of?

All A.S.G.S. participants in my survey have kept themselves current in attendance in the Society and its publications. Seventy-five percent of them are

regular members, the balance associate members; and of all these only 1/6 are exhibitors. Fifty percent have had between 20 and 45 years in their field, operated their own shop either in association with employ or as self-employed entrepreneurs. 93% are fully active in the workplace with half of this group being under 50 years of age. They are predominately men, only 50% are schooled beyond the high school level with roughly a third having earned college degrees.

Most of you have had a mentor who introduced and encouraged either your developing the glassblowing skill or led you into a craft that supports the glassblower. For more than a third of you this had been your parent, a grandparent, uncle or aunt, in one case a father-in-law; in another the influence came during visits to the father's job site. While more than half of us received considerable on-the-job training, it appears fewer than a third of us had our first contact with the material of our trade at work or in school. A solid fifth of us attribute our inclination because of a good neighbor or a friend, and a handful more of us to having had lots of opportunity to tinker at home when ma wasn't looking or while completing college lab assignments. A few recount being impressed by public demonstration of glassblowing or other crafts, but not a one mentioned either literature or the media having acquainted or influenced them.

One tenth of the corresponding sample have not yet married, one of whom is female; the remaining respondents are family men, inclusive of those who did not declare their identity. Their children are clustered in the late teens and older with fewer than one third having children younger than 18. Half of those responding to the survey are the only member of their family who have "been in glass", and a third have spouse, children and parents working it. In their workplace one third have family working with them on premises either in production or in other support capacities.

100% report extending the job to the family in some manner, most sharing particularly their joys and interesting incidents, but only a third expressing their job frustrations to spouse alone. Fifty percent manage to have family visit the job site during working hours and as many their children's active interest about the workplace; a strong third are able to accommodate their children's self-motivated participation in appropriate tasks. While most of these youngsters began to show interest and participate during their teen years, a noticeable genesis of interest appeared in a fifth of them before starting to school. The spouses of one fifth showed interest before marriage and as many early in the marriage as later; perhaps it is because their active participation is very situation oriented, there is an even distribution through out the stages of family development.

It would appear also that where the spouse shows interest the children do too. Statistically that family interest appears highest where the husband particularly loves his work and enjoys sharing it, where he has consciously attempted to enable the family to accept the demands of his job, and where he encouraged work skills and healthy attitudes toward work. There also appears to be a high correlation between this interest and the inclusion of spouse and children in appropriate activities of the professional organization.

It seems to me the most valuable portion of this survey has been provided in your responses to the open questions which allowed free expression of opinion and experience. Indeed it has the most flavor. In many instances as I read I could also "hear" the individual's manner of speaking in their statements, recognizing personalities even before noting their names. Your expressed viewpoints are collated as follows.

TAKE THE JOB HOME?

In answer to whether or not you "take the job home" to family or friends, while one replied "Not anymore", we received an enthusiastic "yes" from most. Our single members discuss their work with parents, siblings and friends, explaining the nature of their work and occasionally its problems. Writes one young man, "Since glasswork was so important to my personal growth, they heard about the highpoints (accomplishments, ideas, promotions) and the low points (health hazards, wages, pressures). Long hours have been spent explaining the esoteric applications of glassware I constructed."

HOW "TUNE-IN" FAMILY TO WORK? . . . PROFESSIONAL SOCIETY? . . . CONTINUITY OF YOUR TECHNOLOGY OR BUSINESS?

It appears most families use the opportunity of Section activities and the symposium to "tune in" their families and involve them in their work. Because of security reasons many families never get to see the work place, yet as a result of awards bestowed both through work and the A.S.G.S., they become aware of their parent's valuable contributions to the advancement of technology. Through symposia many of our children, and spouses too, have had their eyes opened and filled with pride over the quality and value of the work their breadwinner engages in; they become aware of some of its problems as well as the fascination he feels for it. For those of us working in the lower security college lab or in the family business, easier access to the workplace encourages our showing our family the latest development or even involving them actively in the project. And let's not forget "employing the family in the business" as a means to preserving the business or teaching an appreciation for it while at the same time teaching saleable skills.

ADVANTAGES AND DISADVANTAGES OF FAMILY AWARENESS AND INVOLVEMENT?

What are the immediate and long-range advantages and disadvantages you experience in the home and on the job as a result of your family's awareness or involvement? Many of us see its being an educational opportunity apart and beyond formal schooling; it affords an opportunity for all to grow as personalities and as workers. For some it has provided the energy that led to advancement in job status. Because of our familiarizing our families with our work "they are more interested and therefore become more interesting persons". "We share a common interest; we have something we can talk about together. Otherwise I would be isolated from my family and my family from me." Joe Barker profoundly explained in a conversation that "If my family expected to see me we had to work together, especially while we were getting started in the business."

Still others stated: "Everyone shares in the time taken to work . . . and we are together." "We realize our goals together." "The family is aware of the major problems of my job; they understand why I must work late. If everything goes well they know how hard I worked to make it (succeed)." "They understand what I do which means they can support me when needed," . . . offering compliments and encouragement where appropriate," ". . . the wife making good suggestions from a female perspective;" "everyone knows his way around the shop and in an emergency is able to carry on." Repeatedly, family involvement in the workplace not only has held the family together through a major crisis, but it enabled the business to survive it as well.

"It effects better husband-wife relationship" and affords a bridge of acceptance and respect across the generations. "The kids and wife are impressed." "The children know how their father earns our living and what it means to him." "My

wife and I can be proud of our accomplishments and careers. In work they realize that family involvement is necessary for a productive and happy employee."

Among the disadvantages you mentioned that ". . . talking about work is a lot like actually working," especially if the day has gone badly. Particularly in a family operated business, there can be difficulty separating business problems from family life; this can be cause for a big headache and general frustration. It can be difficult to "change caps" from family concerns to business (and visa versa) as the phone rings, etc. especially when there are children about . . . kids will be kids, often inopportunely. You can dismiss your employees, but it takes a real grouch to fire his own mother! . . . or discipline his son or daughter in a business-like manner. "Where there are employees there may be a competitive resentment between the hired personnel and family members, and knowledge of employee wages, restrictions, benefits and amenities may occasionally cause stress and friction."

PSYCHOLOGICAL IMPACT?

Family involvement's psychological impact on you and your family is ". . . in a word – TOGETHERNESS. You tend to know that you have someone there that really cares what you've been doing while you're not at home." "If you've had a hard day you can get problems off your chest through discussion." "For me it has been very positive; for my wife . . . mostly good. However, she has to endure the sleepless nights and pre-occupation I experience during times of stress (It is not easy to grow)." (The parentheses are the correspondent's). Imagine what this would be like for a family who did not communicate about their job-related experiences! Devastating? "For my children, I think it is good for them to share in their parents' experiences, enabling them to better understand and cope with life."

It affords ". . . a greater sense of security and confidence" "peace of mind and a better understanding of each other" and, in turn, ". . . a sense of flexibility, versatility, and adaptability." Being included eases and prevents the build up of stresses within the family over competition with the job for the bread-winner's time, especially when he loses himself in a project. One spouse who shares in the responsibilities of a family business expressed her "need to get away from it from time to time" to something that is particularly her own, maintaining her own unique sense of identity, to provide freshness and variety to the family conversation, as well.

AFFECT ON CREATIVITY? . . . PRODUCTION? . . . IMPROVEMENT OF SKILLS?

How has it affected your own creativity and production? . . . the improvement of your skills? I found particularly stirring such responses as: – "It has forced me to take creative chances!" "Our company's creativity has increased due to the competitive nature of family members." "The encouraging input of the 'outside views' from family has been supportive of creativity on the job." "I keep up my skills because of pride in my work." "Both creativity and production are up because my morale is better." "Having the shop in my home makes it more convenient to work long or unusual hours," "Most important it gave me confidence in myself in the workplace, and from this confidence, a feeling that anything is possible." "Serves as a stimulation . . . and as an additional source of ideas." "Son's interest in computers led to mine and improved business methods."

INFLUENCE ON CHILDREN'S EDUCATION, TRAINING, CAREER CHOICES, VALUES?

In some instances the children when grown stepped right into the family business and in time are assuming the reins; in others they have returned to the family business after numerous years of experience employed elsewhere in the field;

and in others, the careers elected have been other than that of the parents but in somewhat related fields. In other situations it has influenced "awareness of educational benefits and the benefits of education." In our situation it has exposed the children to what we consider a healthy work ethic as well as practical and saleable skills, some of which they are learning and using now. "It has afforded them a diversity of experience: – travel, people, ideas, work, etc." A few others felt it has had little if any influence on their children.

AFFECT ON SPOUSE'S GROWTH?

How has it affected your spouse's growth at work and at home? "About three pounds" declares one husband! "It gave her more confidence to make decisions at home as well as at work." "It effected a positive attitude." "I think the affect is just beginning to show; she is learning new skills (typing) to help her at work. She assumes leadership . . . and is a great help and influence on my leadership responsibilities." One wife who attends section meetings and symposia with her husband, because of her contacts is able to make good suggestions that have been useful to her husband both on the job and in his professional society responsibility. "She grew in ability to organize activities and help in many other ways; she discerned what was required. This helped things run more smoothly both at home and for me on the job."

"We all become more interesting people" as we become involved with being interested in our mate's work as well as our own. Whether or not we pick up the actual skills, we do learn the vocabulary of the trade and come to fathom a bit about the tools, techniques, problems, joys, etc. This can only serve to help our home situation as well as our husbands and ourselves in our respective careers. In my case it effected greater confidence and versatility so that I could work effectively as a substitute teacher wherever they placed me, kindergarten through senior high school, teaching everything in the curriculum including wood shop!

AFFECT ON FAMILY ATTITUDES?

How has it affected your family's attitudes toward your work and the time you give it? The "family has always accepted (my) occupation and involvement since it is an integral part of our existence. Vacations were always attendance at Sym & Sec Meetings." "The family has more appreciation of why so much time has to be devoted to the business." "They are more co-operative and supportive, but they still think it is a lot of time." With genuine interest building, it allows the breadwinner more time and energy to devote to the career that provides for the family. And some feel it has made no difference in their attitudes, if not having had a slightly negative affect.

METHODS UTILIZED? . . . RESULTS?

What methods have you used to encourage and maintain your family's interest a/o paticipation in your work and with what results? In addition to using Sectional Meetings and Symposia as encouraging exposure for the entire family, others have used shop talk, talking about the good things that happen on the job, no deliberate encouragement, and one teasingly would have us believe he uses ". . . everything short of chains, manacles, and torture!" Others, ". . . having to travel a lot, . . . find it difficult to include the family", particularly those with very young families. And one is using patience and tenacity tho' his family's interest seems to have fizzled for a while.

AFFECT ON PROFESSION . . . TECHNOLOGY . . . INDUSTRY . . . LIFE QUALITY?

How does family involvement affect the well-being and growth of our profession and its technology? . . . general industry and productivity? . . . quality of life now

and in the future? "I think family involvement has a very positive and beneficial affect in all three areas. It COULD replace the 'family farm' aspect of our culture, which has all but disappeared. Family involvement has the potential to be one of the strongest positive forces in society today." It allows for continuity of the business. "Freer of organized labor restrictions, it can assure a more creative and productive workplace." While some feel it has marginal impact, others feel family involvement, awareness, interest "can do nothing but improve the society and its members", and "it should improve things generally."

Several reminded that in the "mom and pop" businesses, glass shops too, the employees sense little opportunity for advancement, often to the disadvantage of the company. There is a high turnover of valuable personnel who "see the writing on the wall" and go elsewhere, often on their own. This contributes to stress or instability in the workplace for all. At times the clientele follows the departing employee. Often the controlling family or its interested members wane. In either case both the business and its employees suffer. Incentive opportunities in our family controlled member enterprises appears to be suggested of value to strengthen our backbone industry. It would be interesting to note the statistics on growth and longevity of glass enterprises which have been family founded!

"I suppose family involvement will encourage children to adopt a parent's trade, and guarantee a continued supply of skilled glassblowers, yet I think necessity will remain the ultimate indicator of the state and well-being of the glassblowing industry. As to our quality of life, I have found that an integration of all aspects of one's existence (e.g., work with family life) is beneficial to one's mental well-being and quality of life."

EMPLOYER VIEWS?

What are th employer's viewpoints on "taking the job home to family" . . .its advantages and disadvantages to you and your employees? . . . your viewpoints on A.S.G.S., its affects on the skills and well-being of employees, you and the company? One employer of a sizeable company said that he himself brings his children into the shop, as young as they are, so they'll get a feel for it. Many realize that family involvement is necessary to the productivity and happy frame of mind of their employees; however the nature and extent of family involvement about the employees' work " . . . depends on the interest and personality of each individual worker and family." "It's the sort of thing that takes careful planning." "It is a good thing", but it is also "not good to take the job home on a regular basis," "In most instances it is a productive practice."

"A.S.G.S. is impressive to employees." This sort of "education is the greatest gift you can continue." "Any person coming into Lillie Labs soon becomes aware that we will demand involvement in the A.S.G.S., both at the section and national levels. This attitude has produced an implied obligation and in our small group it's understood that it is the only professional affiliation we have and everyone supports it."

CO-WORKER VIEWS?

As a co-worker or fellow professional, what is your viewpoint on family involvements about you in the workplace? . . . professional organization? Some of you feel "it should be kept separate." Some feel it is no place particularly for children, others that it can be a distraction and an imposition at work or in the professional organization when it is handled thoughtlessly, inconsiderately. For safety and security reasons many establishments and professional groups exercise age restrictions. This greatly reduces educational opportunity. So, whenever young

people are allowed in attendance, it is only prudent and considerate that they be guided by their supervising adults to conduct themselves with the responsibility and good manners such a privilege in adult surroundings merits.

Most of you, however, feel that "family involvement is usually very good, that it depends upon individual family members and their interests and how it is handled." "In many cases it makes participation in the professional organization easier and more affordable." In fact, "those most active in the Society are those who include their families in their work interest and A.S.G.S. activities." "I have yet to witness a situation where family involvement made a situation worse. Spouses spend more time together or with their children; shared experiences appear to strengthen communication and relationships." "With the maturing of the Society I see more family involvement." Much of this is credited to very careful planning by both the spouse committees and the families themselves.

CAREER MOTIVATORS?

What factors, people, and circumstances have encouraged your skill development and choice of occupation? The fear of starvation and the need for a job, any job, afforded a number of us strong motivation. A government assignment during World War II, a training program run by a major company, an orientation with glass being part of the course requirements in college, one's artistic nature coupled with his own fascination for glassblowing, an instructor noticing aptitude in a young lab technician and encouraging continued pursuit of the craft: – "I liked the work very much. I am always competing with myself to improve plus I had the opportunity to work full time as glassblower". "As a laborer in my teens, I realized the opportunity in glassblowing and have extended my abilities and talents to excel in the profession." The family business and A.S.G.S. afforded others encouragement.

A.S.G.S. and its activities has been paramount in importance to many, encouraging skill and technique improvement. Visits to various glass working facilities and the calibre of persons dealt with on a professional level has encouraged many into the field as well as in their improving their skills. Some, while their own formal schooling had not gone beyond high school, continued to educate themselves, strongly motivated to develop to a professional level in the nature of their contributions to science-related fields. Another considerable number of us attribute both their training and their continued progress to the support and encouragement of their wives, friends and family, in-laws included.

MEANING OF OUR WORK?

The question about the importance of our work to ourselves and the modern world, our feelings, attitudes about it and the materials of the trade brought forth the most exciting of responses. One friend shared, "When I am working glass I lose all track of time. When I am troubled I tend to seek refuge in it; at the burner I am in control."

"It gives me a feeling of accomplishment to follow thru on projects from start to finished products." "I get a great satisfaction in producing the best of something." ". . . the uniqueness of glass makes US all unique by virtue of the fact that we work with it. I feel that my desires to be creative are largely satisfied by my work with glass. It also offers the opportunity to contribute significantly to the success of certain scientific endeavors by virtue of my design contributions as well as fabrication skills."

I like the "challenge of designing technical glassware," "When doing my work I am always mindful that I am using my God given talent and I do the best I can (no second best). My work provides me with a creative outlet and is extremely satisfying. I have no need to create artistic glass for satisfaction . . . There is no

other material like glass. No other material has the hot and cold working properties as does glass. While there are some plastics that are commonplace now, that have made big inroads into glassware usage . . . no other material has the combined physical properties (mechanical, chemical resistance, electrical) that glass has."

"Beats working for a living! I love it and they pay me besides! Glass has class! Its unique properties will continue to serve mankind."

"I always knew that my occupation as a glassblower meant a great deal to me. I am more aware of this now that I am no longer employed in this area . . . Glassblowing has served me with a basis in materials, design, (and) manufacturing technologies which has contributed to my pursuit . . . in Industrial Design and . . . in sculpture . . . I don't think there exists a more beautiful, seductive material with which to create anything. Most glass artists will tell you – they can't leave it alone once it's in their blood. The process of blowing glass is a fantastic vision to experience, also. Although glass has seen its replacement (mostly ceramics) in many industrial applications, glass technology will undoubtedly remain irreplaceable in general."

"It's very rewarding to train students in the skills of glass-blowing." "Quite often I demonstrated to civic and school groups about 'the glamorous, glistening world of glass.' Not just a glassblowing demo but on the properties and history of the medium. I feel exposure will dissipate knowledge and benefit the technology intangibly."

A.S.G.S. BENEFIT?

How does A.S.G.S. benfit you . . . and your family . . . ? The symposia, section meetings and literature of the Society have kept all interested persons abreast of current movements and developments in the field of glass. "Professionally, it has exposed me to the latest in equipment and knowledge from others through papers and conversations."

We've gained new skills and techniques and strengthened existing ones, those of the trade and many the founding fathers may not have anticipated: – "The A.S.G.S. has offered me the opportunity to share ideas. It also has offered me the opportunity to listen and to speak at professional meetings . . . to write a few articles and have them published. These accomplishments would not likely . . . have come about was it not for our society. I have learned a great deal from our meetings and symposiums, and had opportunity for leadership. These have all greatly enhanced my professional standing at work, and given me the growth of character that benefits me greatly . . ." Our spouses and our children can also claim having gained confidence, new skills and irreplaceable experiences, many of which have already been described.

The glassblowers' improved status in the workplace and the community is an important benefit derived because of the Society. "I remember . . . when being a glassblower (as an individual) was nothing, but being a MEMBER of the American Scientific GLASSBLOWERS SOCIETY was very important."

"The A.S.G.S. offers the chance to educate oneself beyond the scope of the limited task most glassworkers are assigned at their place of employment. In the practical sense, this may help them further their careers faster than might otherwise be possible."

Through the Society the fabricator is better able to reach his market. The exhibitor and his family, in a face-to-face opportunity, learns what pleases the client about his product (a real boost to the ego). Here he often gains input that is

of value in making improvements that better serve the clients' needs, and the sessions and discussions help him become more knowledgeable of the field he serves.

The Society has had a diverse benefit for everyone, young and old alike. Were it not for the symposia many of us would have missed very unique exposure to educational agents and materials not otherwise available to us or the average person. The travel entailed enroute alone affords all of us valuable opportunities to get to know our land and its people first hand, and keep in touch with friends along the way. The social relationships, the friendships we've made, the unbounding support we've felt, and the good times we've shared through both the sectional and national gatherings are of very special value to all of us.

TREASURED A.S.G.S. EXPERIENCE?

There is some special experience each of us treasures out of our A.S.G.S. memories. More than 30 years ago as he joined the Society one man thought it wouldn't work; he has "been impressed with the way folks have pulled together over the years." Another while chairing a recent symposium, ". . . was overwhelmed by the number of people, particularly exhibitors, who were willing to part with time, money, and materials to help make the symposium a success." During a show in Atlanta "my furnace blew a winding during a talk – glass froze – furnace on wheels traveled across the stage." "When Charlie Litton Sr. told me that he had been considering the building of a cut-off machine and gave up the idea after seeing the first Dyna Cut." Working The Procedures in A.S.G.S. publications is important to another.

The elevators have been good for many a laugh – In a very impressive Toronto hotel, one family's spilling a bag of dirty underwear while waiting; others stranded in another, helplessly riding for an eternity up and down the face of the hotel until the elevator finally stopped by the appropriate levels, their laughter certainly convincing the only alien confined with them that glassblowers are a looped and loony lot.

Then, too, there have been the air-conditioning and booking problems now comfortably reminisced, new foods and waitresses, barbecues and boat rides, stimulating discussions into the wee hours and jaunts with colleagues, getting to know each other and ourselves. The travelling has afforded precious memories of togetherness, . . . and of separation, too. A two year old child on coast-to-coast phone call asking ". . . if she could come over to MY house to see me . . ." One doesn't have to be a parent to feel the love and tender communication shared in this account. Another reflects on his observations of spouses, year in and year out, carrying their babies throughout the symposia, "their small sons growing up completely immersed in glass machining equipment," attendees and exhibitors alike willingly feeding their interest and curiosity.

A.S.G.S. INCREASED EFFECTIVENESS?

How can the A.S.G.S. serve our needs even more effectively, . . . continue to fulfill the objectives of its founding fathers? Our members feel strongly that existing practices and ideas have been working very effectively and should be continued. "Our founding fathers' objectives have been exceeded in almost everything. Who would have imagined all the other Glassblowing Societies that have been established in other countries, modeled on the ideals originally established here. They should feel proud of these accomplishments." "I believe the A.S.G.S. does an excellent job and the question should be, HOW CAN WE SERVE THE A.S.G.S. BETTER?"

"The Society IS its members. The members must be encouraged to participate in every way possible . . . and provided with the opportunity to participate in whatever . . ." Increase opportunities to visit in actual work places during Symposium week as well as in conjunction with Section meetings. "Getting good speakers, good topics and slides and demonstrations on all fields of glass have great value." "By offering meaningful seminars, papers, and workshops. By encouraging participation in all these areas. . . .'Poster sessions' I think have a lot of possibilities for our Society . . . By encouraging membership growth as a priority among the directors. Having the section pick up a new members initiation fee was a good suggestion." Others advocate encouraging new members from associated new fields of technology, with new ideas. Some suggested creating a Supportive Member category, inviting spouses and other interested parties to subscribe to membership. Also recommended is expansion of our scholarship incentive for deserving interested youth. "Help younger members get involved in the Society."

"I feel as though a glassblower has been to a large extent by-passed when it comes to economic compensations commensurate with their relative input to society as a whole," one has voiced knowingly. Others advise that we should ". . . promote the idea of (consumer, society's) dependence on glassblowing for new tech, new products, etc."

We could expand and promote "Fusion" and other A.S.G.S. publications; in addition to serving our membership, this would be of value placed in the libraries of our schools and communities. Some may wish to give earlier editions of our literature and other material on glass to teachers, youth leaders, school and community libraries. Each one of us can "talk-up" our craft with pride wherever the opportunity presents itself. We can promote awareness of glassblowing by our visiting schools and other civic groups where young people are involved, offering to demonstrate or discuss our craft. Each section may take increased advantage of free opportunities to publicize glassblowing and A.S.G.S. activities in the printed and audio/visual media. Likely it may have only a small return in recruitment, but it is bound to create a community awareness, and in time perhaps the hoped for economic compensation added to the rich rewards we already know.

By far the most important investment we can make in the future of the Society, our profession and our work, and particularly in that of our families, however, is that matter we've been discussing, – communication and pulling together. As you have indicated in so many ways, of greatest critical impact is the active communication we share with our families, our involving them about our work. This requires neither conscious planning nor a lot of time. It is simply a matter of taking advantage of whatever time we have with our own families, sharing our interests and encouraging theirs, the curiosity and interest of our own children and their friends. It's a simple matter to include them in what we do while we are not at home with them, but it tells our families most strongly that we care about them, and about their caring too. It's a very special way of saying "Tho' I love my work, I love you best of all."

ACKNOWLEDGEMENTS

My heartfelt thanks to all who have supported me so generously in the development of my paper, my family as well as those of you who supplied the information and viewpoints needed. Without your supportive encouragement my paper would have missed its purpose, that of reflecting the views of The Society Members. The following is but a partial listing of contributors to the content of this paper as it appears today. *Indicates written response.

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STATUS OF THE JAPAN SCIENTIFIC GLASSBLOWER'S ASSOCIATION

Harumichi Shibata
Sibata Scientific Technology Ltd.

First of all, I would like to thank the symposium chairman and the president of the A.S.G.S. for giving me this chance to speak to you about our association called NRGK (Nihon Rikagaku Glass Kogyo Kai, Japan Scientific Glass Blower's Association) and its recent status of activity.

NRGK was founded in 1952 and it is the only nationwide organization of glassware manufacturers for scientific application existing in Japan. In addition to the headquarters office in Tokyo, there are 6 branches: Tokyo, Kyoto, Nagoya, Sendai, Osaka and Kyushu. At present, we have 197 companies as regular members and 16 companies as guest members.

On the other hand, I suppose, there are more than 100 unorganized glass shops. All told, there are about 300 glass shops in Japan. Supposing each shop has 3.5 glassblowers: there are about 1,000 people working. The average age of these glassblowers, now 45.6 years old, is getting higher, and the number of people is declining each year since there have been very few newcomers in this industry in past decades in Japan.

At our annual conference the usual practice is to have a general meeting, presentation of technical papers from our members and lectures by professors of universities and researchers of governmental institutes. We also have survey tours to associated factories and research laboratories, and we always have technical discussions with these people.

Through such events we exchange information with each other, whereby we can improve the quality of products, rationalize production efficiency, learn about new industries and new materials and cultivate mutual friendships.

In the last 34 years, our association has cooperated with MIT (Ministry of International Trade and Industry) and the Ministry of Labor. We have prepared JIS R3503 (Japanese Industrial Standard) for glass apparatus for chemical analysis. In the last 5 years we have been adjusting to the ISO Standard as per MITI's recommendation. I have brought a copy of the book JIS R3503, which I will present to the A.S.G.S. as a memento of my visit.

We have cooperated with the Ministry of Labor in establishing a system to give vocational tests which certify glassblowers skills, and are recognized by the government. So far, 150 people have been entitled to the first grade and 45 people to the second grade, under this system.

The theme of the practical test for the first grade is the manufacture of a so-called Soxhlet extraction unit. The theme for the seond grade is the manufacture of a Dimroth condenser. In addition, we have complied a glassblower's handbook, which is not only helpful to the candidates for this test but for glassblowers in general, to study glassblowing.

The glassblowing part of the tests are given in the glass shops of our members' companies in the presence of the officers of the Labor Ministry and the official approval committee. Written tests are given 2 to 3 days after the glassblowing part.

In recent years we have been exchanging visits with Korea, Taiwan and China in order to determine possibilities of cooperation and to establish long-term business relations. Mr. Kinoshita has visited China several times and has published a book about the glass situation in China.

Today I have mentioned very briefly the history and activity of our association. In the very near future, I hope, we shall be able to send our people to this conference with more technical papers. In exchanging people with you by taking the opportunity to attend your annual meeting of the A.S.G.S., I hope we can certainly contribute to the improvement and progress of our industry and our societies in the future.

In the time I have remaining, I would like to talk some about the current status of typical industries in Japan, such as electronics in general, including semiconductors, consumers electronics, cars, cameras, etc. In such mass-production industries, the production efficiency has been remarkably improved by drastic changes and by the extensive adoption of automation since the oil shock in 1973. I remember when I visited a Sharp calculator factory in the NARA prefecture for the first time in 1970. There were about 600 workers, mainly young girls on the assembly line. When I visited there again, in 1985, I was surprised to find so few people working there. To my surprise there was only one, a manager, and this manager was not working.

In such high technology, mass production industries, I think that the Industrial Revolution is now going on in Japan, Germany and even Korea. In such industries there have been great changes in factory management systems in the last 5 years. When we look at our industry, however, the situation is quite different from those industries and there is little room for change, since we have to resort to human skill that requires many years of experience.

It is sad to say that the membership in our association has decreased from about 240 to 200 since 1971. This means that the demand for our industry has been declining year by year. In order to deal with this change in the business climate, we have been trying to find new markets for our industry in recent years. Unless these efforts are successful in the near future, we are afraid that we will not be able to preserve our precious technology accumulated from our predecessors, and pass it on to the next generation.

We wish to welcome your frank advice, so as to help us find a way for our industry to survive.

Thank you for your attention.

THE MECHANICS OF SCORING AND BREAKING GLASS

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April 3, 1986

The process of separating glass has changed very little for over 100 years. The least expensive and quickest way is to create a fault, or fissure along which a break may be induced.

This technique may be described as a controlled catastrophe. By contrast, sawing glass with diamond media, water jet, or laser may seem less a catastrophe, but involves considerable expense and time.

Irrespective of the alloying ingredients, glass is an amorphous material lacking grain or other directional characteristics. Often called a super cooled liquid, glass might be likened in many ways to a gelatin such as Jello.

In proceeding from a heated liquid to a solid, the surfaces tend to reach a solid state sooner than the interior. As the interior continues to cool, its contraction places the exterior in compression leaving the interior in a condition of tension. The gradient of opposing forces is less severe in well annealed glass, and conversely very extreme in tempered glass where the exterior cooling is accelerated by sudden quenching.

The various methods of making glass result in different compression/tension gradients, but these internal forces tend to be in equilibrium or the glass assumes a warped shape. One can generalize by stating "the greater the force gradient, the harder the surface 'seems' to be."

Another point of relevance is that glass has a lower rupture modulus in tension than in compression.

The above described characteristics of glass are important to an understanding of the process of scoring and breaking.

SCORING:

Several tools are in use today, but the most common is the wheel. Diamond in the shape of a pyramid was once the most prevalent tool and still is widely used in some foreign countries. The 90 degree edge of a block of tungsten carbide is often used in tube cutting. The wheel, however, remains the easiest and most cost effective, and is the tool this presentation will address.

Today's glass cutting wheels are made of hardened tool steel or tungsten carbide. The surface of glass is relatively hard and the scoring action is abrasive, so wheel life is a function of the hardness and wear resistance of the wheel material. Generally, tungsten carbide wheels can be expected to outlast steel by a factor of 8 to 10.

The geometry of the wheel is significant. The smaller the diameter, the less force required to create a fissure. The typical range of diameters is .140 to .250 inches. The angle at which the apex is honed is critical. The most widely used angle is 120 degrees included, but the range for mass produced glass is 114 to 140. Special angles outside this range are used with the following guide: the harder the glass, the sharper the angle should be. For example, quartz tubing may separate easier by using an angle as sharp as 88 degrees. Thick flat glass, in excess of 16 mm, may score best with wheel angles up to 160 degrees.

Angle specification can best be appreciated by examining what happens when a wheel rolls along the surface. Regardless of the angle, the apex must be a well defined edge, not rounded nor variable in its profile. The wheel generates shearing or tensile forces in the glass at right angles to its honed faces. The direction of these forces tends to be more parallel to the surface of the glass with sharp wheels and more oblique as the wheel angle increases.

A fissure will be created which tends to be in line with the wheel and extending into the glass to a depth related to the force applied to the wheel. In flat float glass, this depth would be approximately .012 to .015 inches. If the wheel angle is too sharp and/or the force applied is too great, the glass tends to chip along the score line. This is undesirable not only cosmetically but more importantly, the energy that should be creating the fissure is partially dissipated.

BREAKING OUT THE SCORE:

The "controlled catastrophe" is generally completed by bending the glass in such a direction as to place the score line in tension. A typical practice with flat glass is to place a small cylindrical object under the glass and in line with the score at the edge where the score was completed. Applying a modest downward force on both sides of the score, the glass will be seen to separate in a rapid "run" of the break from edge to edge. One can test the fact that glass breaks more readily in tension than in compression. Try bending the glass with the score on the inner arc of the bend and nothing will happen until much higher forces are attained. The break may become a general fracture unrelated to the score line.

With tubing, the practice is much the same. A bending moment is applied with the score at the outside of the bending arc. It is important that the fulcrum over which the glass is bent be directly beneath the score line, otherwise, an unsquare edge will result. This is more critical with tubing than flat. The break tends to propagate from the score to the fulcrum.

Tapping under the termainal end of the score line is a common technique for starting the break. A shock wave is induced which acting on the notch effect of the fissure, causes a rupture to occur. This often creates a flare in the break because it is difficult to direct the blow exactly under the score line and even more difficult to judge the force of the impact.

Another technique frequently used to propagate the fracture is to introduce an intense and highly local flame to the glass. Rapid expansion creates the added tensile forces which complete the break.

The process of "glass cutting" is not cutting at all. While it is a necessary activity to the production of objects to be made of glass, it is in many ways more art than science and as such, affords an element of creativity and satisfaction when done well.

PROCEDURE FOR LOW VOLUME SILVERING USING LONDON LABORATORY SOLUTIONS

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Submitted for the American Society of Glassblowers Symposium of 1986

I. INTRODUCTION

To fabricate glassware that shows the pride you have in your work, only to discover that the silver plating has a tacky appearance, is most discouraging. My clientele has referred to me, on such occasions, I am sure, as "that old grouch", for the remainder of the day. My silvering quality has ranged from great to awful. I was frequently perplexed. I needed to take the "Black Magic" from silvering, remove the dread of failure, and achieve consistent results that would make silvering a task to anticipate with pleasure.*

We all seek high reflectivity without defects, but with reliability. The London Company silvering kit uses for solutions: NS1 (primarily silver nitrate), NS2 (alkali), NR reducer (primarily invert sugar) and RX sensitizer. Brashear's, as most of you know, is silver nitrate, potassium hydroxide, ammonia and invert sugar. Having had problems with both London and Brashear's solutions, I intend to provide substitutes for two of the London Company's solutions that have proved troublesome; and, in addition, after comparing London (1) and Brashear's (2) reliability and quality, show that the London formula should be the one preferred.

II. PROBLEMS FROM STORING SOLUTIONS

A. The Alkali

Since the London company product gave excellent results at first, I was very pleased to find that after a time quality became the exception rather than the rule. Grasping at straws, I decided the house distilled water was at fault, or possibly, the glass was not clean. I spent an inordinate time removing poor silvering and re-cleaning glass, all to no avail. I actually considered returning to the old Brashear formula. The last remaining possibility was that the London solutions themselves were at fault. Deterioration during storage was worth investigating, but how to identify the culprit?

I received a strong hint upon opening a partially filled bottle of the alkali ammonium hydroxide (NH_4OH)* and not experiencing the immediate attack on my sinuses. In fact, the solution resembled water more than NH_4OH. This indicated that alkali, over time, could become less than puissant.

The chemistry people said that indeed, hydroxide solutions would deteriorate if stored in glass. Even when dilute, the base will attack the glass and become contaminated with silicate. I admit that I had stored my dilute NS2 in a Florence flask. I won't do that anymore.

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*Knowing little of chemistry, I have made the errors of the untutored. Therefore, much of the information in this paper will be obvious to the chemist. However, my intent is to share my experiences with those who may also be having similar difficulties and to allow them to avoid the frustrations of discovering the answers themselves.

*The "OH" is the hydroxide and is the part of the compound which produces the strength as an alkali.

Also, chemists indicated that hyroxide solution deteriorates in the presence of air, reacting with carbon dioxide to form salts and carbonates, (3) hence, weakening the solution, resulting in poor plating action.

In short, I reasoned that the London NS2 could be the culprit, if the NS2 is a hydroxide. I needed to know the molar strength of the dilute NS2 base to control the speed of the reducing reaction. Making use of the talents available at a university and by gentle threat of – no titration; no glassblowing; – a chemist determined the molar strength of the NS2 concentrate, but not the identity of the hydroxide.

Knowing that Brashear uses potassium hydroxide (KOH), I thought it a good bet that London Company did also. I didn't have any KOH at the moment, so I substituted sodium hydroxide. It is cheaper and requires fewer grams per liter. I made four small dewars; two were silvered using fresh NS2 concentrate and two plated using ordinary sodium hydroxide diluted to correct molar strength.

After annealing, the reflectivity and thickness of the coatings was very good and appeared to be identical. Wishing to leave no stone unturned, I substituted potassium hydroxide for the sodium hydroxide with the same good results. Sodium and potassium hydroxide are virtually interchangeable.

The NS2 concentrate turns out to be 5.5 molar. The London Company instruction sheet directs that 500 ml of the dilute solution be prepared. I, therefore, make my hydroxides to the same volume. This is achieved by using 4.4 grams (3) sodium hydroxide or 6.2 grams potassium hydroxide in 20 ml water for the concentrate and adding 480 ml water. Any mention of water, of course, means distilled. The chlorine in tap water would really wreck one's efforts, not to mention the effects of local variations in soluable salts.

The London instructions also direct that after the 500 ml of NS1 (silver nitrate) and 500 ml NS2 (alkali) dilute solutions are made, the two solutions are mixed to make 1 liter of dilute. This forces storage of this solution after silvering, since the NS1 contains valuable silver. Subsequent deterioration of the alkali, however, renders the carefully saved solution useless for good silvering. My method is to carefully measure the required volume for each dewar and mix 1/4 NS1, 1/4 NS2 and 1/2 NR reducer. This leaves my dilute and valuable NS2 unsullied for storage.

With some apprehension, I contacted the London Company. Chemist, Mr. Bahls, kindly confirmed the validity of the 5.5 molar concentration and volunteered that a small amount of ammonium hydroxide was also added (4). The amount of NH_4OH per liter of concentrate is 4 ml of 19% NH_4OH or 2.75 ml of 28%.

Sodium hydroxide grams	Potassium hydroxide grams	ml H_2O concentrate	ml NH_4OH 19%	28%
220	308	1000	4	2.75
110	154	500	2	1.37
55	77	250	1	.68
22	30.8	100	.4	.28
8.8	12.32	40	.16	.11
4.4	6.16	20	.08	.055

* Chemicals required to produce convenient volumes of NS 2 concentrate.

The 4.4 and 6.6 figures in 20 ml water for the alkali added to 480 ml H_2O will fit in conveniently with the volumes NS1 and NR reducer as stated in London's instruction sheet (3). The ammonium hydroxide is added to give chemical stability and also control the reaction rate (4). More ammonia slows the reaction. Therefore, the micro amount of ammonia indicated in Table I is not that critical. I use a 1 ml syringe and add 0.2 ml NH_4OH of 28% to make up the 500 ml dilute. If you wish to store the dilute alkali, the concentrate should be diluted with water that has been boiled to remove dissolved carbon dioxide (7). Store in a virgin polyethylene container as indicated in Figure 1. All this preparation seems bothersome to a busy glassblower. It is much simpler to prepare the NS2 fresh for each use and discard any remainder.

B. The RX Sensitizer

Unfortunately, there is also a problem with the storage of the London RX sensitizer. Upon long storage, an unappetizing mess of yellowish solids appear. This is because old stannous chloride concentrated solutions decompose slowly in water (3). London Company ordinarily supplies to volume users and therefore did not anticipate problems with their NS2 and RX solutions. But it was clear to me that a homemade substitute would be desirable for low volume users. Since I had used stannous chloride (5) to treat glass surfaces when using the Brashear's method, and noting that London instructions called for 0.2 ml RX sensitizer in a liter of water; the similarity of this to the stannous chloride treatment (5) employed by Allen B. Brown using Brashear's, was hint enough. I therefore, prepared my fresh RX sensitizer as Mr. Brown indicated. Namely, 1 gram stannous chloride in 10 ml water for the concentrate and 0.2 ml of this in one liter water for the dilute solution and proceeded as the London instructions indicated.

The results were excellent. Stannous chloride does not deteriorate in the dry state in a reagent bottle. I now had my very own fresh RX sensitizer. Again, feeling insecure about my efforts, I called Mr. Bahls of the London Company and received confirmation that the substitution would work and that their RX did deteriorate over time. Actually, their RX is stannous chloride.

I did not inquire as to the detailed content of the NS1 silver nitrate or the sugar NR reducer. This is proprietary knowledge that I do not need because the keeping qualities of these chemicals are excellent, convenient and reliable.

III. COMPARISON OF LONDON AND BRASHEAR'S METHODS

The literature seems to treat Brashear's and London methods as equal; other than that London is more convenient to use. This in my view is not the entire case. The London formula deposits virtually no bloom from spent solutions, and as important, has no explosive hazard while mixing solutions (4). Also, when adding ammonium hydroxide to silver nitrate and potassium hydroxide when using Brashear's method, it is easy to go beyond the end point with unfortunate results. London will give the same quality every time with no hassle results.

I now wish to examine this problem of bloom (6) deposited during silvering, more closely. My chemistry department recently dumped on the glass shop old dewars and columns, that had been collecting dust in the basement. Upon cutting them up for disposal, I was startled to find the first surface (as defined in Figure 2) plating of the inner vacuum space bore no resemblance to the classic grey bloom the literature so often described. The coating appeared to range from cream colored to an off-white and to be quite thick, as is evident from the photo shown in Figure 3. The reflectivity was low. Scott (6) states that the first surface evacuated space reflectivity is the most effective side in reducing heat transfer.

It can be argued that in some special conditions reflectivity is not that important, but I am sure that generally, it is important. For example, holding time for liquid nitrogen, or keeping liquid helium long enough to complete a study. Further, I know of no glassblower that wouldn't prefer high reflectivity of both first and second surfaces.

A completely silvered dewar gives no clue as to the internal reflectivity. Since this white coating distressed me, I prepared six test tubes and silvered them with London solutions. The inner reflectivity could then be examined. I stopped the reaction at increments of five minutes, through thirty minutes, and annealed to check temperature tolerance also. All the tubes retained reflectivity, although after twenty minutes, a slight haze could be detected on the samples. To my joy, London solutions do not become flocculent and so do not deposit any significant bloom.

I then tested Brashear's. First, I prepared a tube and silvered one half so that the inner surface could be seen. Five minutes later, the solution was removed and like magic that same undesirable white deposit appeared. To be honest, the ammonium hydroxide had been added to the silver nitrate and potassium hydroxide with some impatience on my part and the solution was overly rich in ammonium hydroxide.

Six more test tubes were used and silvered with Brashear's. The ammonium hydroxide was added more slowly this time. Again, the reaction was stopped at five minute intervals as before. They were then treated with stannous chloride (5) and annealed. All the tubes retained reflectivity with no white coating. The tubes left in the longest did have the grey bloom. Visually compared to the London tubes, Brashear's came off second best.

To summarize, a satisfactory job can be done with Brashear's formula, but you would be well advised to pay careful attention to the drop by drop addition of ammonium hydroxide. This procedure may be difficult for an often interrupted university glassblower. Removing Brashear's solution rapidly enough to prevent excessive grey or white bloom during strip silvering, is even more questionable. Especially, since one can never be absolutely sure that his solution was prepared with exactitude.

IV. CONCLUSIONS

There are other ways to mess up silver plating, but my hope is the information in this paper will improve percentages considerably. With fresh NS2 and RX sensitizer combined with London Company's NS1 silver nitrate and NR reducer solutions, silvering will produce effective vacuum space reflectivity even if the phone rings at the most inopportune time, since it is very forgiving on silvering reaction time. If a customer smashes a dewar, have no fear; for there will be nothing to see but beautiful shiny silver on all the broken pieces.

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FIG. 1 Arrangement for Eliminating CO_2 from Hydroxide Solutions

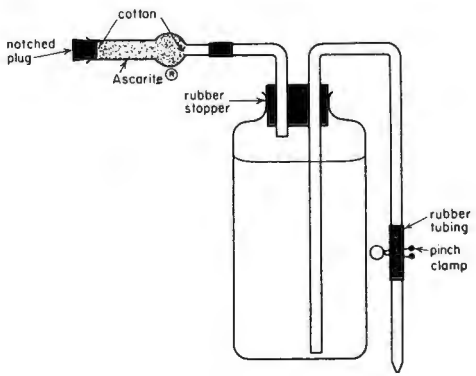
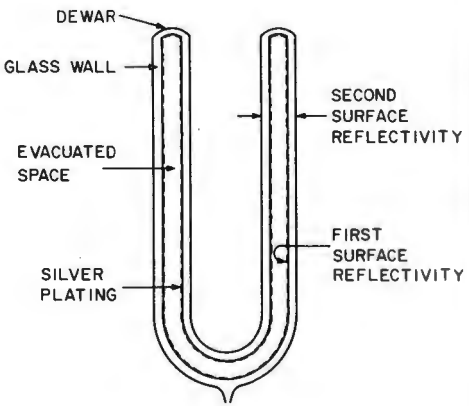


FIG. 2 First and Second Surface Reflectivity Defined



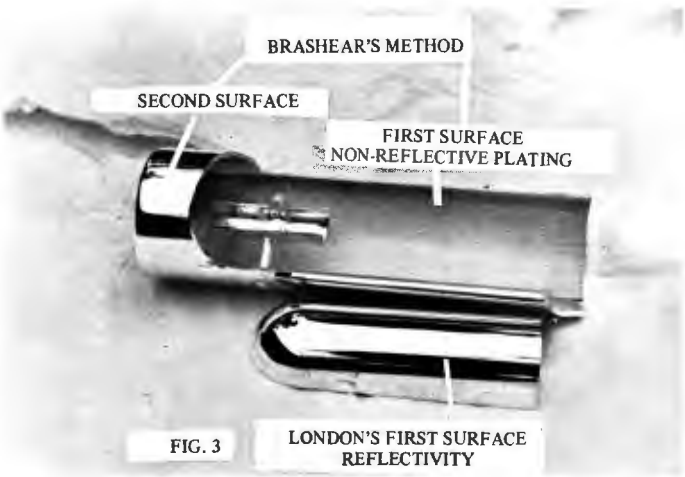


FIG. 3

CLEANING GLASS AND METAL SURFACES IN THE GLASSBLOWING SHOP

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ABSTRACT

Producing professional quality glass apparatus requires mastering pertinent glassworking skills and proper preparation of materials and subassemblies. Of paramount importance is proper cleaning. This paper will present cleaning procedures for tasks ranging from everyday repairs, to critical cleaning of vacuum systems, exotic electronic and biochemical apparatus, and also advice on how to keep things clean.

CLEANING GLASS SURFACES

Insisting that glassware brought into the glass shop be clean necessitates the assumption that the client knows how to properly clean the apparatus, and has done it. Cleanliness is both a practical and a safety issue for the glassblower. A safer policy is to assume that everything that enters the glass shop is dirty, and for glassblowers to assume the responsibility themselves. Ultimately, if you want something done right, you must do it yourself. Your health is at risk, and only you can adequately safeguard it.

The removal of radiological contaminants is beyond the scope of this paper except for the following advice:

a) make a habit of inquiring if the apparatus has been used with radioisotopes before accepting the repair.

b) recommend that all apparatus used with radioisotopes be disposable.

c) insist that all apparatus (including common flasks and beakers) used with radioisotopes have clearly visible, permanent radioactive decals fired on.

d) maintain good rapport with radiation safety personnel to assist in identifying and monitoring sources of potential hazards.

A little planning in the design phase can pay-off many times over in the cleaning that won't have to be done.

There can be no substitute for a well trapped vacuum system, or proper selection of materials (including gaskets, etc.) for a reaction vessel. If a system is so delicate that a stuck joint or stopcock could cause a catastrophic fracture, then by all means incorporate a teflon sleeve, stopper, or stopcock. If designing an apparatus to handle strong corrosives or solvents, again use all teflon valves rather than any with O-rings.

Another problem that need never occur is the fouling of vacuum systems with stopcock grease. It is amazing to see the percentage of laboratory personnel who don't know how to properly grease a stopcock plug or glass joint! The idea seems to be that if a little is OK, then a lot is better. Nothing could be further from the truth. The proper technique for applying grease is to apply a tiny line straight down one side of the male member of a small plug or joint, and two tiny lines down a standard taper 24/40 or larger joint. Insert it into its mate, and with several rotations, inerspersed with removals and reinsertions, distribute the grease evenly over the ground surface. Then, remove the male member and with a clean tissue wipe off the bulk of what little grease is there. Reinsert into its mate, and again rotate to redistribute the remaining grease. The seal should appear clear and

uniform without any obvious gaps. If gaps are present, either discard or regrind the joint, but in any case don't add more grease.

An excess of grease will not improve an inferior seal even though it may temporarily solve a leak problem. The quick and dirty grease solution to leaks will catch up with you down the road after the system becomes fouled with grease, which in time will itself become grossly contaminated with whatever is being used in the system.

Excess grease tends to smear over everything and contaminate reactants, it will itself become contaminated with particulates which can cause joints to freeze, and in high vacuum systems the contaminations in the grease can themselves be the source of persistant or sporadic virtual leaks. It seems that these drawbacks are unknown to most laboratory personnel, so it's up to us glassblowers to rectify this.

Designing the construction of an apparatus to facilitate flame cutting whenever possible can be a great time saver. Flame cutting is the procedure of intense heating, pulling, and thinning of tubing in lathework. Properly done, the diameter, wall thickness and concentricity of the tubing remains unchanged, and the resulting ends will be smooth, clean and uniform. This technique obviates the need to unchuck or contaminate the work with scratches, and can be performed on hot, freshly worked glass. In production work it is indeed the technique of choice.

For everyday laboratory ware brought into the glass shop for repair the following procedure will be satisfactory for all except apparatus contaminated with metals (e.g. mercury, solder, or thin films):

a) remove teflon and elastomeric components (e.g. plugs, sleeves, O-rings).

b) 'ash' the glassware by putting it through a conventional annealing cycle. This can be accomplished along with uncontaminated ware in the oven.

c) when cool, dip the ashed glassware in a 10% HF (hydrofluoric acid) solution for thirty seconds or less.

d) rinse extensively (thirty seconds or more depending upon the situation) with cold tap water.

e) scrub with detergent and water, and rinse.

f) if necessary, repeat steps c), d), and e).

g) if necessary, apply a little concentrated HF to the soiled spot and rinse with cold tap water after 15 seconds.

h) finally, rinse with DW (distilled water) or DI (deionized water).

If this procedure doesn't work, the contaminant has probably entered the glass surface itself, and must be removed by localized grinding, or heating and then picking the contaminated glass off the surface. In the worst case, the apparatus, or a piece of it may have to be discarded and replaced.

After the ashing step, but before the cleaning step, the piece should be trimmed-up on the cut-off saw. Annealing is important to do before trimming, because strains which might otherwise cause cracks to run can be relaxed.

The HF solution should be kept in a 10 liter polyethylene (high density linear polyethylene-HDLPE type is best) large mouth container with a screw lid (available form Cole-Parmer, and other lab suppliers). When using concentrated HF, allow the excess to drip into the container. A finite quantity of HF solution can be made to last almost indefinitely this way. Dirty HF will clean just as well as a fresh solution, so there's really no excuse for dumping it down the sink.

There is a debate among glassblowers whether glassware should be given a long or short dip in dilute or concentrated HF, and also on whether the dipped piece

should be neutralized in an alkalai solution, or simply rinsed with no neutralization. As a rule, the shortest possible dip in the most dilute solution that results in satisfactory cleaning should be employed. Too long a dip in too concentrated a solution can result in a noticeably etched surface. Also, consider that the very process of flame working irreversibly alters the chemical composition of the glass, removing the glass network modifiers (notably sodium) and making the resulting regions more vulnerable to HF attack, resulting in an irregularly etched surface.

If the piece will be dipped more than once before completion, shorter and more dilute dips are in order. Alternatively, try replacing a dip with the direct application of concentrated HF to the soiled area followed by immediate rinsing. This will avoid the over-all etching effect of dipping, however the fumes of concentrated HF are corrosive (resulting in a frosted surface), so the entire piece should be completely rinsed with water.

The cleaning regimen employed should be kept to a practical minimum. There's no point in costly and unnecessary effort, especially if it may result in unnecessary etching or leaching of the glass surface. Consequently, neutralization after dipping is rarely necessary for conventional laboratory glassware. Neutralization can in fact result in contamination of the glass surface with the salts formed by neutralization.

Throughout this discussion the term 'extensive rinsing with water' will be used. Just how extensive is extensive? In keeping with the intent of designing practical procedures, it should be no more than required to achieve a reasonably uncontaminated surface. It's unnecessary to achieve a scrupulous surface prior to simple flame working. As long as the operator won't be contaminated by any residue, and the seal can be effected without unsightly blemishes, then the minimal cleaning is the best. For this purpose, up to a minute in running water, or five flushings of a closed vessel may be adequate. The persistance of residual HF can be tested for with pH or litmus paper. Any residue able to pass this test is unlikely to cause any problems.

After cleaning, allow the apparatus to dry completely before working or annealing. This can prevent a lot of headaches, as droplets of water exploding into a boil can cause (especially complex) apparatus to break.

Detergents tend to leave a tenacious residue which, if dried on, will resist rinsing. The detergent of choice should not only clean stubborn contaminants, but should itself rinse quickly and completely. In the author's experience the best commercially available detergent is Micro (sold by International Products Corp., P.O. Box 118, Trenton, NJ, 08601-0118, 609/394-5480), which admirably accomplishes this latter goal, and its cleaning action doubles in strength with every 10 C that it is heated. It also forms emulsions with water and solvents, thus attaining the cleaning capabilities of both polar and nonpolar solvents in one bath. This can be amazingly helpful, especially in removing mysterious gunk from organic chemistry labware.

Ultrasonic baths are hard to beat for easily scrubbing off stubborn surface contamination. They are best used with hot detergent or solvent solutions. Also, the ultrasonic effect will transmit through an upright beaker containing corrosive chemicals if immersed or floated in the bath.

Vermiculite dust from the annealing oven floor is unusually tenacious. As a substitute, try an equal depth of clay-type kitty litter. The clay dust is much easier to wash off of annealed workpieces.

It is worth mentioning that good housekeeping practices become more important as the degree of cleanliness increases. Simply organizing the glass shop

such that splash from the cut-off saw and grinding equipment doesn't contaminate cleaned glassware depicts the lowest level of cleanliness. Storage in a hot vacuum box in a cleanroom lies near the highest level.

For some operations a clean room with filtered downdraft ventillation and protective particle-free outer garments for personnel is called for, but this is obviously beyond the means of most glassblowing shops. There is, however, a way to achieve nearly the same degree of cleanliness on a small budget and scale; namely, the commercially available filtered downdraft hoods and glove boxes that are table top size. The glove boxes are the least expensive, and may be quite adequate for clean assembly of apparatus in an otherwise dirty workshop.

Vapor degreasing is an easy and very effective method of cleaning machined and soiled glass and metal parts. The workpiece is continuously bathed in fresh, warm distilled solvent. Freons (e.g. tri– or tetrachloroethylene) are typically used. When ordering, be sure to specify that you want the 'inhibited' types which contain special additives that prevent the pot residue from becoming acidic and corrosive to the still. Other solvents can be used and are necessary depending upon the nature of the contaminants to be removed. Extreme caution should be exercised if flammable solvents (e.g. methyl ethyl ketone, xylene, toluene, acetone) are used.

Upon withdrawal from the vapor degreaser, a single remaining drop may be noticed at the lowest point of the workpiece. A holder should be designed such that it forms on the holder instead, otherwise the last drop can be removed with a clean absorbant tissue, to prevent deposition of a residue.

If further solvent cleaning is necessary, methanol or ethanol should be used. As a wiping solvent, methanol dries too quickly, so ethanol should be used.

As a final rinse prior to sealing with silicone (RTV) adhesive, acetic acid will clean and activate glass surfaces.

There are two easy tests for cleanliness of glass surfaces. The first is the water break test which consists of wetting the workpiece with DW and setting it aside to drip dry. If the water sheets off evenly and without suddenly pulling away from any regions, it passes. A notable exception is if the surface is contaminated with a hydroxide residue which is hydrophillic and will give a false positive result to the water break test.

The second test consists of placing a single small drop of very pure concentrated (glacial) acetic acid on the workpiece. Any surface contamination will be revealed by a violent swirling and rainbow film on the droplet surface. In the event of gross contamination the droplet will quickly spread over a rather large area. If clean, it will just sit there and slowly evaporate without leaving a trace. A significant drawback of this method is that it tests only a small spot on the workpiece.

Once you have cleaned something it may be necessary to keep it clean while in storage. The principal contaminants will be dust and water vapor. In some laboratory and industrial environments there will be other contaminants, notably machine and vacuum pump oils. The best way to prevent contamination is to store cleaned things in a vacuum oven. If this is not feasible, then store things wrapped in aluminum foil which has itself been degreased by wiping with methyl ethyl ketone. Crimp the edges to seal out dust and store in cardboard boxes or sealed cabinets. Prior to final use, these 'clean packages' can be heated in either a vacuum oven or a conventional oven to drive off adsorbed water.

If storage in paper is preferred, brown kraft paper should be used, as white papers contain bleaching and whiting agents which can contaminate the workpiece.

Polyethylene should be avoided, as it can leave a residue. If a plastic wrapping is desired, mylar film is the most inert and inexpensive available.

Silicone vacuum (stopcock) grease has a reputation of being difficult to remove from glass surfaces. The manufacturer recommends wiping off excess, then soaking the affected parts in a warm concentrated potassium hydroxide solution for ten minutes, rinse with water, wipe dry, dip in chromic acid, rinse in water, and then dry. Some glassblowers prefer to use various solvents (e.g. benzene, toluene), or hot detergents (e.g. Micro). While all of these will work, the basic ashing procedure described is the easiest method of all. In a simple experiment, it can be demonstrated that none of these methods is any more effective than simply removing excess grease with clean disposable wipers, and then scrubbing with Micro and water. In fact, scrubbing with hot water alone can remove silicone grease. In the author's opinion, silicones have an undeserved reputation.

A common source of surface contamination in assembling an apparatus is the asbestos or other material used to support components in place. An alternative which will not shed particles, can withstand considerable heat and (unlike asbestos or its substitutes) need not be removed until assembly is complete, is common copper screening available in hardware stores. Pretreat the screening with dilute nitric acid for a few seconds to produce a uniform, clean dull surface. This pretreatment helps insure that the copper is particle free. Roll, crinkle or crush the screening to fit snugly in place, and then proceed with assembly. The copper screening can be incorporated completely into the apparatus and then dissolved out with dilute nitric acid after completion of the workpiece. Be sure that the hot zone doesn't get so near the screening that it softens, crumbles, or melts into the glass surface.

Do not anneal the apparatus without first dissolving out the copper screening, otherwise the glass will be permanently discolored green.

In working quartz, surface bloom is always a problem. It can usually be 'burned' back into the work piece if it's not heavy, otherwise it may have to be etched off with concentrated HF, scrubbed with detergent and water, water rinsed, rinsed with DI or DW, and then any remaining burned in. Preventing its formation in the first place is always preferable. Proper flameworking technique can help; always work with as sharp a flame as possible, and avoid letting the flame 'splash' onto adjacent cool regions.

Bloom is a devitrified material which actually forms in the air, condensing from the quartz vapors. It settles out and adheres to the adjacent cooler regions. Careful flame control can direct the vapors away from the work, largely eliminating the problem. Expecially when working with large apparatus, this may not be possible. In these cases try painting on a layer of clean Aqua-dag diluted three fold with DW. It is imperative that the Aqua-dag be clean, otherwise surface contamination may result. Paint it on right up to the hot zone and let it dry completely. In bringing the piece up to working temperature the Aqua-dag in the hot zone will evaporate without a trace, and that which remains will provide a barrier preventing deposition of bloom. After the piece is completed, the Aqua-dag can be removed with detergent and water.

Another technique is to allow the fuel gas to bubble through ethylene dichloride. This can prevent the very formation of bloom, however it produces corrosive and toxic HCl fumes which can quickly fill a room not equipped with special ventilation.

Occasionally the glassblower is called upon to work on an apparatus which, it is requested, not be cleaned. This presents the unsatisfactory situation where surface contaminants can burn into the glass surface causing surface boil, and also the

liberation of possibly toxic fumes which will enter the mouth via the blow hose. The former should be pointed out to the client, and there is nothing more that can be done. The latter health hazard can be minimized by the use of a device constructed as follows.

Take approximately 18 inches of 70mm diameter tubing, and fit one hole stoppers into each end. Into one, insert a short length of tubing to which the mouth end of a blow hose tube is connected. The swivel end of this blow hose is attached to the apparatus to be worked on. Into the other one hole stopper insert a funnel such that the large portion of the funnel will insert into the large tube, and attach the swivel of a second blow hose to the protruding funnel stem. With a rubber band, attach a latex surgical glove or condom to the large end of the funnel, and insert this assembly into the remaining end of the large tube. What you now have is a tube containing a latex barrier that effectively separates the contaminated atmosphere in the apparatus from your mouth. By blowing into the blow hose, the latex will balloon into the large tube and thus pressurize the apparatus.

In some other instances it is necessary to work on an apparatus in a controlled atmosphere. An example of this is working on something that is oxygen sensitive, or where your breath may contaminate the workpiece (as in repairing an ultraclean electronic apparatus, like a laser). If the above described device will not suffice, perhaps the following technique will do.

Splice a T or Y into your blow hose and allow clean dry nitrogen to bleed slowly through it and into the workpiece. Excess nitrogen will flow gently into your mouth without pressurizing the workpiece. To pressurize, don't blow, just place your tongue over the mouthpiece and the workpiece will pressurize with nitrogen.

Although nitrogen is itself not harmful, be warned that it will dilute the oxygen that you breathe through your mouth, and you can become light headed. To avoid this, breathe through your nose while the nitrogen is running.

CLEANLINESS IN VACUUM ENVIRONMENTS

Glasses high in metal oxides (e.g. lead and alumino silicates) are relatively resistant to He (helium) permeation. The harder glasses (and especially quartz) are more prone to He permeation.

The leak rate for a 7740 sealed container at 10^{-10} Torr is 10^{-11} Torr/min, therefore the ultimate vacuum attainable is He leak limited. If an apparatus is intended to hold a static vacuum (without pumping) in excess of 10^{-7} Torr, or a dynamic vacuum (with pumping) in excess of 10^{-12} Torr, it must be constructed of a less permeable glass (e.g. 1720).

He permeation is particularly troublesome for liquid He glass dewars. Unless constructed from a less permeable material, the jacket must have provision for continuous evacuation.

Quartz is permeated not only by He, but also by hydrogen and neon.

All glasses will adsorb gasses. Additionally, glasses in a high energy flux environment will generate gasses due to a variety of degratory processes.

For 7740 surface adsorbed water will be liberated upon heating to 150° to 200°C, and further heating to about 300°C will generate water vapor from the dehydration of the Si-OH-OH-Si bonds at the surface to yield Si-O-Si and water. Further heating to over 450°C will cause water in the bulk to diffuse to the surface. Heating to about 490°C will be sufficient to degas the outer 10 to 20 microns of 7740. Some of this water is dissolved in the glass from manufacture, and if not

removed, will diffuse through the bulk and thus contaminate the vacuum. At 500°C the rate of outgassing is some one billion times greater than at room temperature, so this should be considered for vacuum systems operated at elevated temperatures.

High energy electron bombardment can cause oxygen to be dissociated from the glass surface.

UV and gamma radiation can dissociate both hydrogen and water from glass.

High energy neutron bombardment of borosilicates can liberate hydrogen and evolve helium from the fission of boron to form lithium and helium. This transmutation of boron breaks its bonds to two oxygen atoms, thus liberating atomic oxygen into the glass matrix, however the oxygen will not diffuse through the bulk and tends not to contaminate the environment. Due no doubt to this altering of the glassy structure, glasses exposed to high energy neutrons accumulate tensional stresses which can result in structural failure (fracture). As these stresses are a consequence of chemical changes, they cannot be relieved by annealing.

The liberation of water from glass can be a considerable nuisance when attempting to hermetically seal something by 'tipping off'. As a consequence of the heating necessary to make the seal, more water may be liberated than was removed by prior processing, therefore the following points should be considered:

a) Partially constrict the zone to be sealed prior to tipping off.

b) Strip the hydrated surface with a dip in dilute HF, rinse with cold tap water, then with DI or DW. Follow this with a typical annealing cycle.

c) The thinner the wall thickness and the smaller the bore to be sealed, the less time and heat required to effect the seal, therefore less water will be generated.

d) A cold trap close to and downstream from the tip off will speed the pumping of any water generated.

e) Keeping the upstream apparatus heated will tend to drive out any water generated.

f) Pinching-off the softened glass with suitable forceps can achieve the seal at a lower temperature and with less water generated.

g) Incorporating a getter into the closed system will result in chemical pumping for long after the sealing operation.

Gettering is the process of utilizing the reactive characteristics of a material to maintain a vacuum. The two general types of getters are dispersive and nondispersive. Dispersive getters, as their name implies, create a little explosion, increasing their surface area by dispersal. After working so hard to clean up an apparatus it's sometimes ill-advised to utilize a dispersive type getter if the apparatus will be damaged by the liberation of millions of particles; however they do have their uses. The most commonly found getter is the dispersive barium getter, evidenced by the mirror-like appearance at the top of common radio tubes. The barium sputters out from a tiny cup as a result of either resistance or radio frequency induction heating, and plates onto its surroundings. It reacts principally with oxygen and water vapor, and appears milky when spent. The milky appearance of the getter material is the indication looked for to determine leaking or 'gassy' tubes.

Several other metals are used as gettering materials, among them zirconium which getters hydrogen, nitrogen, and oxygen, and titanium which getters these same three gasses in addition to carbon monoxide, carbon dioxide, and water vapor. These two metals are sintered with carbon to form nondispersive getters which again must be heated for activation.

The choice of and firing regimen for getters should be determined by the nature of the expected contaminants and the shelf life of the apparatus. Dispersive getters are usually a one-shot affair, but the nondispersive types can be soft fired (partially activated), stored, and then hard fired, thus increasing the product's shelf life.

Especially with expensive electronic apparatus, the time and expense of making the proper selection and use of getters can more than pay for itself.

A glow discharge cleaning is sometimes necessary as a final in situ step for vacuum systems or components, especially prior to sputter or evaporation coating. The cleaning is achieved by low energy ion and electron bombardment. The usual procedure is to introduce a few Torr of either ultra pure argon or oxygen which becomes a conducting plasma in the system. Materials are desorbed by heating due to bombardment by the positively charged plasma ions. The binding energies of the molecules to surfaces is exceeded above 500 eV. Effectiveness of the process is dependent upon the conformation of the particular system and its electrical filaments. If oxygen is used, it dissociates into chemically active atomic oxygen that reacts with organic surface contaminants to produce volatile oxidation products that are readily pumped away. Gentle gas flow can be used to flush out contaminants. If argon is used, it tends to imbed into the surface and may need to be baked out (280° to 300°C). This process is called plasma ashing or plasma stripping.

Silicon wafers are plasma etched in a carbon tetrafluoride atmosphere, producing volatile silicon tetrafluoride and carbon dioxide.

Sapphire can be cleaned by a process called gas etching where the workpiece is placed in an atmosphere of sulfur tetrafluoride or sulfur hexafluoride and is heated in an oven to 1150°C.

A related exotic cleaning technique is sputter cleaning where the workpiece's surface is sputtered off.

The foregoing techniques produce surfaces which are clean on an atomic scale, and can be undone by simply exposing to the atmosphere.

Heavy wall rubber tubing, commonly used in vacuum work, is unsuitable for vacuums greater than 10^{-3} Torr. Cleaning of the tubing is accomplished by detergent and water washing, rinsing, and then bathing in a 30% sodium hydroxide solution, followed by more water washing and then drying, preferably in a vacuum oven set at a temperature low enough to not damage the tubing (e.g. 100°C).

CLEANING METAL SURFACES

The term polish is used to indicate a process, either mechanical, chemical, or electrochemical, which results in a more level surface as the result of removal of surface material. This objective is attained when the surface layer containing microcracks and imbedded materials from manufacturing, assembly, and handling is removed, ideally without the chemical contamination of the underlying bulk material. A matte surface tends to pick up and retain surface contamination. This can especially be a problem with sealing surfaces, which should be polished.

Mechanical polishing is achieved by either rubbing or blasting with an abrasive. Both of these techniques result in surface scratches, pitting, and imbedding of abrasive particles. Glass bead blasting is the mildest treatment, and any imbedded beads can be etched out with pickling solutions that contain HF.

Chemical polishes are commercially available proprietary acid solutions, each with specialized instructions. Inquire of electroplating shops to obtain these.

Electropolishing is a process whereby the workpiece is made the anode (electrically positive) and material is plated from it into the solution and onto the cathode. Due to the nature of electrical field concentrations, metal removal is most rapid from high points, and the resulting surface tends to become level. Some hydrogen from the acid solution may dissolve into the metal, but it can be removed by heating in a vacuum.

Kovar should be solvent degreased after machining, and then degassed. The easiest method is to simply heat in a pure hydrogen flame for a few minutes. This may not be satisfactory for certain applications and the following method described by Wheeler should be used: In a wet hydrogen furnace, heat to 900℃ for two hours, then to 1000℃ for thirty minutes, then to 1100℃ for fifteen minutes. Allow to cool to below 300℃ before removing from the reducing atmosphere. In preparation for glassing, reoxidize in a slightly oxidizing flame or atmosphere (650℃) briefly.

Tungsten is commonly found as an electrical feed-through in glass apparatus. In ordering tungsten for vacuum apparatus, be sure to specify electronic grade and not welding grade (commonly called 'stingers'), as the lesser grades contain minute longitudinal striations that can be the source of slow leaks. Cut tungsten wire on the cut-off saw and not with wire cutters, as it otherwise tends to splinter.

Tungsten can be etched in a solution of equal parts hydrofluoric and nitric acids (tungsten with nickel sleeves attached should be boiled in 20% sodium hydroxide for one hour, water rinsed, rinsed in dilute nitric acid, then DW rinsed). Tungsten can also be electrically cleaned in a saturated potassium nitrite solution at 10 v AC.

The clening method of choice in the glassblowing shop is to heat in a torch flame and then quickly plunge into sodium or potassium nitrate or nitrite powder and then withdraw it. The tungsten will undergo an exothermic reaction in the powder, and if not withdrawn quickly, will quickly be consumed by it. Ater this treatment it should be rinsed in water and then inspected for an even silver gray appearance. If it is not uniform, repeat the procedure or discard. Prior to sealing into glass, lightly oxidize the cleaned surface in a sharp torch flame.

Oxide layers on copper are thick and highly adsorbent, and form readily above 200℃. Copper is impervious to helium and insensitive to hydrogen and water at room temperature, but is attacked by acids in the presence of oxygen. Copper should be avoided in systems utilizing mercury (e.g. diffusion pumps) as it will form an amalgam. In ordering copper for vacuum and high voltage use, be sure to specify OFHC grade (oxygen free, high conductivity) which is 99.98% pure.

Copper can be bright dipped in a solution of 60% sulfuric and 15% nitric acids, with 1/2 oz. hydrochloric acid added per gallon. This solution is used for 5 to 45 seconds at room temperature, followed by an alkaline rinse to neutralize, a water rinse, and immersion in a sodium cyanide bath to destain.

If used on brass, this procedure will result in a matte finish, which is satisfactory when preparing for an elastomer seal.

In preparing copper for a Housekeeper seal, the feathered edge is attained by etching in a dilute nitric acid solution, then water rinsed.

304 stainless steel is commonly found in vacuum systems. Due to a spontaneously formed, invisibly thin layer of chromium oxide, it is highly corrosion resistant. 304 stainless has a very low vapor pressure, however it has a surface appearance similar to cadmium plated steel (a common anti-corrosion coating), and should not be allowed to become confused with such parts, as cadmium has a relatively high vapor pressure.

The formation of the chromium oxide surface can be speeded up and its uniformity controlled by immersing the freshly machined part in a 50°C bath of dilute nitric acid for 30 minutes, followed by water rinsing.

Because of its protective film, 304 stainless should not be cleaned with steel wool, as this will remove the protective oxide film, and fragments of the steel wool will embed into the surface and result in corrosion and pitting.

303 stainless welds and brazes more easily than 304, but 303 contains sulfur which has a high vapor pressure and will contaminate the vacuum, and so should be excluded from vacuum systems.

Chemical cleaning of stainless and other mild steels is as follows:

a) vapor degrease to remove oils and grease.

b) soak in a hot alkaline bath to remove stubborn greases.

c) soak in a potassium permanganate solution to condition the oxide scale.

d) dip in a hydrochloric acid solution to sensitize the metal surface.

e) soak for 3 to 30 minutes in pickling solution (by weight: 30% nitric, 3% hydrofluoric, 66% DW) at room temperature. To remove more metal and yield a roughened surface, the pickling solution should be 33% nitric and 33% hydrofluoric acids. For carbon and low alloy steels, 5 to 15 minutes immersion in an 8 to 12% (by weight) hydrochloric solution at 39°C is sufficient.

f) rinse with water.

This procedure should only be performed with workpieces that are without open seams and fissures from which the acid solution may not be entirely flushed. As troublesome as virtual leaks from dirty components may be, leaks from chemical corrosion due to ill-advised cleaning procedures is far worse.

As oxide layers on iron are thick and highly adsorbent, it is seldom found in vacuum systems.

Nickel is often found in laboratory apparatus as it resists corrosion in air, water, salt water, alkalais, and most organic acids, but not hypochlorites, nitric acid, and wet gasses containing sulfur dioxide, bromine or chlorine above 580°C. Nickle is impermeable to He, but may conatin a high fraction of hydrogen, oxygen, carbon monoxide, and carbon dioxide. The bulk of the hydrogen can be removed by heating in a vacuum to 500°C.

Aluminum finds limited use in high vacuum due to its relatively high vapor pressure at temperatures above 300°C. An exception is its high resistance to cathodic sputtering, due in large part to the presence of a thin protective coating of oxide. For use as a high voltage cathode, aluminum should be etched in a hydroxide solution (thus increasing its surface area) and then oxidized in situ by making it anodic in a low pressure oxygen atmosphere. Alkali etching will yield a white matte finish that is sufficient for elastomer seals.

Proprietary chemical and electropolishes containing phosphoric and nitric acids are commercially available, but are not typically necessary for vacuum work.

Gold and platinum leaf can be removed from glass surfaces with aqua regia, which is a mixture of one part nitric to three parts hydrochloric acids. If this method proves unsatisfactory, try putting a little mercury (dirty mercury is fine) into a beaker and immerse the plated surface into it. The mercury will adhere to the metal and form an amalgam. Wearing rubber gloves, rub the mercury gently into the metal. Dip the piece into dilute nitric acid to strip the amalgam. Repeat this

treatment as required to remove all the metal. It's always a good idea to work with mercury in a plastic dish pan in a sink to contain any spills. Mercury spilled down the sink will be retained in the sink trap and can be easily retrieved, and its submersion under water in the trap will prevent the liberation of toxic mercury fumes into the workplace.

Mercury should be triple distilled in a partial vacuum to insure its purity for laboratory use. As the presence of large quantities of mercury carries with it the hazard of exposure to its toxic fumes, the cleaning of instrument grade mercury should not be attempted in the typical glassblowing shop.

Two tests to determine the gross contamination of mercury are to observe whether a droplet in a container appears mirror-like and tends to ball up off the surface, and whether a droplet rolling across a piece of white notebook paper leaves a dirty gray streak.

Any system incorporating mercury either to create or measure vacuum needs to be suitably trapped. Surge traps and constrictions, in addition to dry ice or liquid nitrogen traps, should be incorporated.

APPENDIX

Formula for 10% HF:

Out of the bottle, HF is 49 to 52%. Dilute one part of this with 5 parts of DW. Example; for one liter of 10% HF solution, add 200 ml of concentrated HF to 800 ml of DW. REMEMBER – ALWAYS ADD ACID TO WATER – NEVER add water to a concentrated acid as it may react violently.

ALWAYS work in a proper ventilation hood and wear a protective rubber apron, gloves, and safety glasses when working with acids and caustics. In general, getting even dilute HF in the eyes means INSTANT PERMANENT BLINDNESS.

First aid for HF burns:

Inhaled: Remove to fresh air immediately. Give artificial respiration or oxygen if necessary. Summon paramedic help IMMEDIATELY!

On skin: Remove contaminated clothing immediately. Flush affected areas with cool or cold water for 15 minutes (note, 15 minutes is NOT 15 seconds!). If available, treat burn with clacium gluconate gel or zephiran (benzalkonium hydrochloride) solution. Pay particular attention to regions under and around fingernails. With burn wrapped in water or zephrin soaked wrapping, seek medical help IMMEDIATELY!

In eyes: Flush with cool, fresh tap water for 15 minutes (NOT 15 seconds!). Summon paramedic help IMMEDIATELY!

In all cases, don't be shy about summoning assistance at once. If you doubt this, just consider how shy you might be with a finger or eye missing!

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EXPERIMENTAL EXAMINATION OF A SIMPLE EXPRESSION FOR STRESS RELEASE IN GLASS

H. E. Hagy
Corning Glass Works
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ABSTRACT

An experimental examination has been conducted using a simple expression for stress release in glass. The expression contains a single relaxation time proposed by Weymann and used by Spoor and Burgraaf for a basic Maxwell model. Since the expression attributes stress release to be predominately due to viscous flow, experimental stress data are used to calculate glass viscosities. Examples cited show that viscosities thus derived are in good agreement with known values and demonstrate viscosity-time dependence in the lower transformation range as reported by Lillie. It is concluded that this expression is reasonably accurate for practical applications of stress release in glass.

1. INTRODUCTION

Much has been written on the phenomenon of stress release in glass. A complete bibliography on the subject would number dozens of references. Depending on the thermal state of the glass and the temperature involved, stress release can be complex, which explains the variety of equations proposed in the literature. To quote Novotny and Kavka[1], "at the present time various models for the mathematical description of relaxation at temperatures above the strain point are somewhat in disagreement. Most, however, agree that the relaxation mechanism at a given temperature is controlled by viscous flow."

It is the purpose of this paper to experimentally examine a simple stress release expression based on viscous flow. The analyses yield convincing data that support recommending the expression for engineering approximations.

2. THE STRESS RELEASE EXPRESSION

Based on a simple Maxwell model with a single relaxation time proposed by Weyman[2] and used by Spoor and Burggraaf[3], the mathematical expression to be examined is:

$$\sigma = \sigma_o \, e^{-\frac{t}{\tau}} \tag{1}$$

where:

σ = stress at any point in time
σ_o = original stress at time zero,
t = time, sec., and
τ = relaxation time, defined as:

$$\tau = \frac{6(1-\gamma)\eta}{E} \tag{2}$$

where:

γ = Poisson's ratio,
η = viscosity, poises, and
E = elastic modulus, dynes/cm^2.

Since the experimental examination of Equation 1 is based on the calculation of viscosity from stress release data, it is appropriate to combine equations in that form:

$$\eta = \frac{Et}{6(1-\gamma)\ln \sigma_o/\sigma}$$

or

$$\eta = \frac{Et}{13.8\ (1-\gamma)\log_{10} \sigma_o/\sigma} \qquad (3)$$

It is an interesting exercise to calculate stress release at the annealing and strain points and compare results to the old definitions for these reference points: stress will be substantially relieved in 15 minutes at the annealing point and in 4 hours at the strain point. Using Equation 1, calculated stress release values are given in Table 1 for a typical commercial glass. In these calcualtions, 1.5×10^{13} and 4.7×10^{14} poises were used as the annealing and strain point viscosities, respectively, as found by beam bending[4] and believed to be the most accurate. This exercise shows that the old definitions hold quite well, except that the time is overstated for the annealing point. It is postulated that those drafting these early definitions were concerned about thermal equilibrium within the glass article and adjusted the time at the annealing point accordingly.

3. EXPERIMENTAL EVALUATION

3.1 Television Glass

Figure 1 shows stress release curves for Corning Code 9010 glass, a black-and-white television composition. These data were experimentally generated in the 1950's for engineering purposes. Mildly air quenched rectangular cross-section glass beams were periodically removed from the treatment furnace and the optical retardation related to the central plane tension was measured at room temperature with a Friedel polarimeter. Results for the other two glasses in this study were also obtained by this method. The curves for temperatures 380 and 383°C are nonlinear due to an increase of viscosity with time, a phenomenon discussed later in this paper.

Using the curve for 418°C and the known properties for this glass, the viscosity is calculated with Equation 3:

$E = 6.2 \times 10^{11}$ dynes/cm^2 at 418°C
$\gamma = 0.24$ at 418°C
$\sigma / \sigma_o = 0.20$; $\log_{10} \sigma_o/\sigma = 0.699$
$t = 1/2$ hr $= 1800$ sec.

$$\eta = \frac{(6.2 \times 10^{11})\ (1800)}{(13.8)\ (0.76)\ (0.699)} = 1.52 \times 10^{14} \text{ poises}$$

Refer to Figure 2 which plots the glass annealing and strain points (442° and 412°C, respectively) on a viscosity-temperature graph. The calculated viscosity datum falls exactly on the straight line which properly describes the glass viscosity-temperature behavior over this short range. Note that the elastic properties at 418°C were used for the calculations to be more exact. Actually these properties change very little with temperature and in this example, only a 4% adjustment results.

3.2 Titanium Silicate Glass

Air quenched Corning Code 7971 ULE[tm] titanium silicate glass specimens periodically removed from a furnace served to photoelastically generate the two stress release curves shown on Figure 3. This glass has an elastic modulus of 7.4 x 10^{11} dynes/cm^2 and a Poisson's ratio of 0.19 at room temperature.

Figure 4 shows the viscosity-temperature curves for a specimen of this glass quenched with the stress release beams. These curves were determined by the beam bending method both on heating and cooling. The heating curve was taken first at a rate of 5℃ per minute to determine the initial quenched low viscosity state. Upon reaching 10^{11} poises, cooling was established at a rate of 4℃ per minute and data were taken to establish the near-equilibrium, higher viscosity structural state. From the stress release data average viscosities were calculated using Equation 3 for both temperatures. These data are indicated on Figure 4 and display a gratifying behavior; firstly, because they show the expected rise in viscosity with time characteristic of quenched glass, as reported by Lillie[5]; secondly, they fall within the boundaries established by the beam bending data.

3.3 Tempered Code 1723 Gage Glasses

Some recent investigations were carried out to update engineering stress release data for Corning Code 1723 tempered aluminosilicate gage glasses. Data up to 1000 hours were established at one temperature, 427℃, which was controlled to ± 2℃ over the duration of the experiment. These data are shown in Figure 5. The 1000 hour value of 0.82 for σ / σ_o is in excellent agreement with that reported by Shand[6] for a glass of this type.

Again, viscosities were calculated using Equation 3. Two points thus derived are shown on Figure 6. Also plotted are the near-equilibrium curve established by the annealing and strain points, and an estimated quenched state curve representing the initial viscosity. The data points are assigned the elapsed time for the measurement, but in reality the viscosity calculated is for some weighted average over the time span.

4. Practical Usage

To estimate stress release at some termperature in the transformation range, one should first plot the annealing and strain points on a semi-logarithmic graph using viscosity levels of 1.5 x 10^{13} and 4.7 x 10^{14} poises. This curve will serve for relatively slow cooled glass. Very rapidly cooled glass will follow the curves shown in Figures 4 and 6. The viscosity chosen for the application depends on the concern for the amount of stress release to be accomplished. If overkill is not a problem, the higher viscosity is chosen with an appropriate time. If stress reduction must be limited, a viscosity at or near the lower level must be assigned with a conservative time. In the latter case, a conservative first try is recommended, expecially if the glass article has high value or retempering is impractical. Equation 3 applies, and η, t, and $\sigma_{\overline{o}} / \sigma$ are adjusted to fit the situation.

5. Conclusion

A simple mathematical expression for stress release in glass, based on viscous flow, has been experimentally assessed. Three sets of stress release data, cnosen without any bias, for three different glasses, yield viscosity data that appear credible.

This expression is recommended for approximations of stress release to those who are practically involved in heat treatment of glass. Elastic moduli and Poisson's ratios can be supplied by glass manufacturers.

6. References

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5. Lillie, H. R., "Viscosity-Time-Temperature Relations in Glass at Annealing Temperatures.., J. Am. Ceramic Soc. 16 (1933).

7. FIGURE CAPTIONS

Figure 1. Stress Release Curves for Corning Code 9010 Glass at Four Selected Temperatures.

Figure 2. Viscosity-Temperature Curve for Corning Code 9010 Glass with Datum Calculated From Stress Release Data.

Figure 3. Stress Release Data for Air Quenched Corning Code 7971 Glass.

Figure 4. Viscosity-Temperature Curves for Air Quenched Corning Code 7971 Glass Including Points Calculated From Stress Release Data.

Figure 5. Stress Release Data for Corning Code 1723 Tempered Gage Glasses at 427°C.

Figure 6. Viscosity-Temperature Curves for Corning Code 1723 Glass Including Points Calculated from Gage Glass Stress Release Data.

TABLE I
Calculated Stress Release Values at the Annealing Point and Strain Point for a Typical Commercial Glass

Annealing Point		Strain Point	
Time, min.	σ / σ_o, %	Time, Hrs.	σ / σ_o, %
1	52	1	29
5	3.8	2	8.5
10	0.14	3	2.4
15	0.006	4	0.7

$E = 7.5 \times 10^{11}$ dynes/cm^2

$\gamma = 0.23$

FIGURE I
Stress Release Curves for Corning Code 9010 Glass
at Four Selected Temperatures

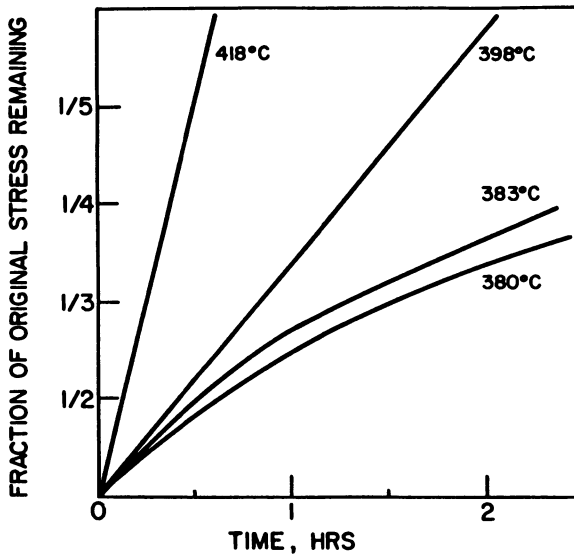


FIGURE 2
Viscosity-Temperature Curve for Corning Code 9010 Glass
with Datum Calculated from Stress Release Data

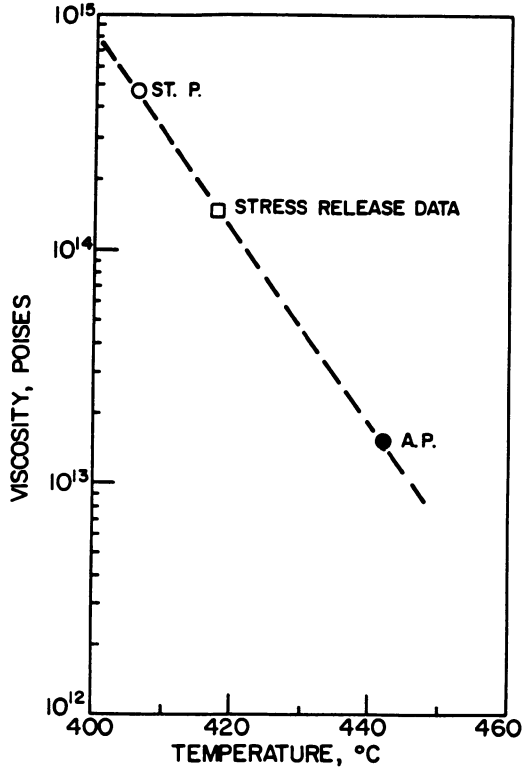


FIGURE 3
Stress Release Data for Air Quenched Corning Code 7971 Glass

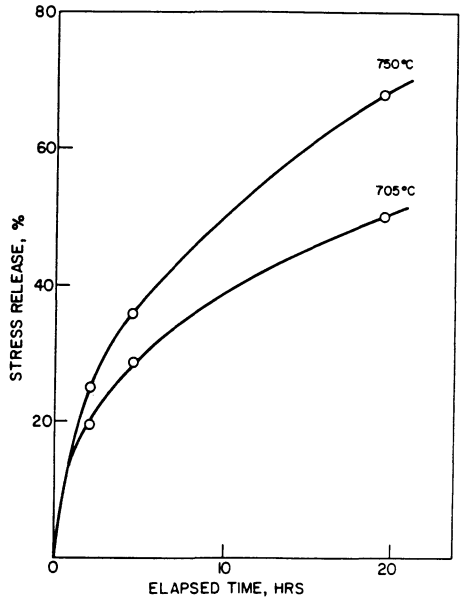


FIGURE 4
Viscosity-Temperature Curves for Air Quenched Corning Code 7971 Glass Including Points Calculated from Stress Release Data

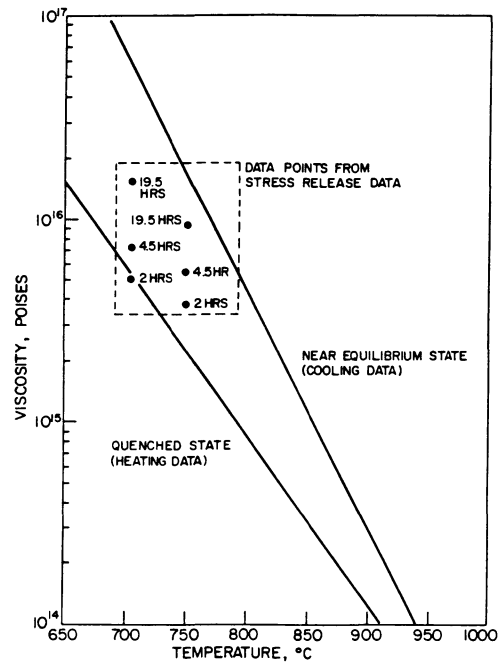


FIGURE 5
Stress Release Data for Corning Code 1723
Tempered Gage Glasses at 427°C

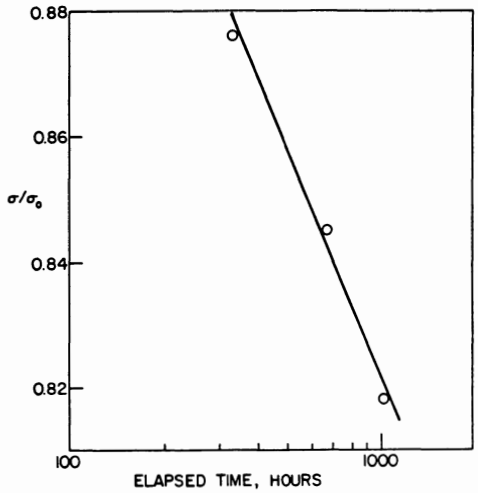
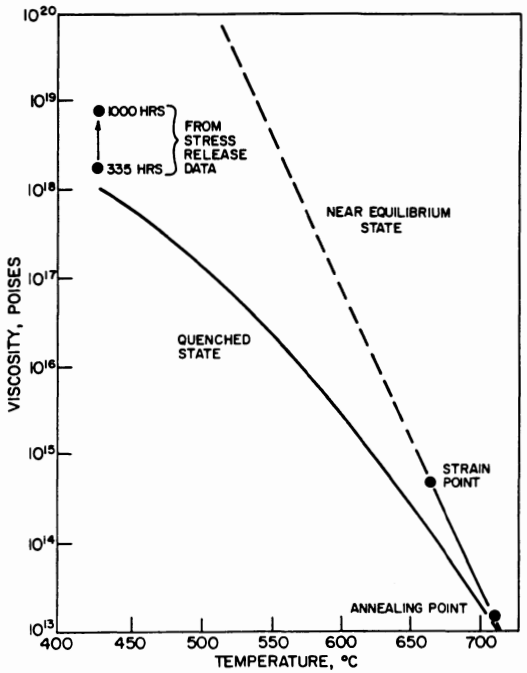


FIGURE 6
Viscosity-Temperature Curves for Corning Code 1723 Glass
Including Points Calculated from Gage Glass Stress Release Data



"CROSS ARM SAW AS A GLASS CUTTING MACHINE"

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INTRODUCTION

The glassblower has used many methods for cutting glass with a diamond, with heat, with mechanical pressure and with cut-off machines. This paper is about using an existing machine which has a number of attachments that make it versatile in a very small shop operation. It has had the capability primarily of sawing, drilling, joining and disc sanding of soft materials, To be absolutely clear, there are several well manufactured glass cutting machines on the market and demonstrated here, that beyond a reasonable doubt, answer the problem of cutting glass accurately, efficiently and effectively. This is to say, that the machine to be described cannot compete with the higher precision ones in quality but it can cut satisfactorily for an experienced operator in most situations required. The results depend largely on the operators skill and ability.

The purpose of this paper is to give you an idea of what can be done with equipment already in a small shop. It may not be the answer for your application but, from this presentation you may get a perspective that, hopefully, will be helpful.

This machine is used by many, either at home or in their place of business. There have been numerous articles written about it, so it actually, is nothing new. Cutting woods, plastics, and sometimes soft metals are standard . . . . but cutting glass? Well, that is quite different and probably unheard of.

MAIN BODY

The machine is a 1960 Sears 10" cross arm saw with a 1 HP 220/110 V 3450 RPM motor with all attachments. In Figure 1 the machine is shown using a carbide blade ripping a piece of hard wood. The head is turned 90 degrees from the cross cutting operation in order to rip any length of material. The machine is ruggedly constructed of cast iron, steel and aluminum.

In Figure 2 the machine is shown as doing a drilling operation. The saw blade is removed and the head is set in a verticle position with a 1/2" chuck mounted so that the spindle is verticle to the table and the entire unit can be moved up and down to drill a hole to the proper depth. This change-over takes less than a minute for an experienced operator. The machine is so designed that holes can be drilled with the spindle in a horizontal position also. It is possible to drill holes in glass with proper cooling lubricant such as is used in more expensive machines.

Figure 3 shows another attachment application, namely; the joiner. This unit might perhaps, with some ingenuity, be used on glass finishing but, if there is, the author is not aware of it. There are possibilities, perhaps. The setup change takes two minutes.

In Figure 4 the machine has a 10" disc sander mounted on the right end of the spindle. This disc, mounted with the proper cutting material, has a great cutting and finishing capacity on a variety of materials including glass. For glass a cutting fluid, of course, should be used. Onto this steel disc wheel can be mounted a silicon carbide 10" wheel that needs mechanical support for any side pressure put on it. This technique is very effective.

In Figure 5 a tool grinding wheel is mounted in place of the carbide saw blade. This setup is used very commonly for grinding tool bits, drills, in particular, but

may be utilized for some glass finishing or cutting operation provided with proper cooling. Again, these setup changes take just a minute of the experienced operators time.

In Figure 6 a 10" silicon carbide cutting wheel (or even better a diamond wheel) is mounted on the right side of the motor. A water collecting bath is placed below the wheel with a metal shroud to collect the water thrown from the wheel. A water supply line is directed on the wheel and the water can either be fed by gravity or by a small fountain pump (Sears - Cat. No. 71H27743). This system does work satisfactorily.

The cross arm movement feature with the glass stationary is limited to cutting 70mm glass tubing. Larger tubing may be cut by rotating it in place against the cutting wheel. The glass may be rotated on small rollers.

In Figure 7 the setup shows how an angle may be cut on tubing or rod using the same approach and technique as in Figure 5. Needless to say, in this picture, there should be further work done on holding the glass, but the idea is there and can be completed by the individual glassblower's innovativeness.

In Figure 8 the fixturing shows ways by which small rod or tubing can be cut straight or on an angle using plexiglass which is V-grooved to hold the glass securely. This setup was used in cutting at accurate angles glass envelopes for small laser units. The finishes came out very well and produced satisfactory results for the customer concerned.

CONCLUSION

Hopefully, it is possible that any innovative glassblower so inclined and who works in a small shop operation can see what may be accomplished with this versatile machine, which was designed for other materials than glass cutting. The basic machine lends itself to a number of applications and, if changing setups is no problem, then many different jobs can be completed.

CREDITS

The author wishes to thank Wilbur C. Mateyka, Technical Papers Chairman; Peter Severn, Glassblower, Chemistry & Metalurgical Engineering, U. Of Michigan; and Jim Panczner, Editor of *Fusion*; all of whom gave me inspiration and support to write and present this paper.

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Figure 1.

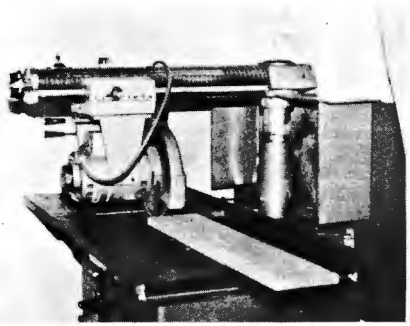


Figure 2.

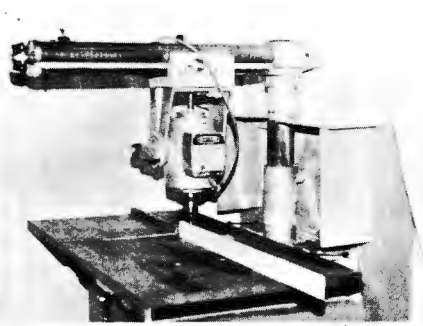


Figure 3.

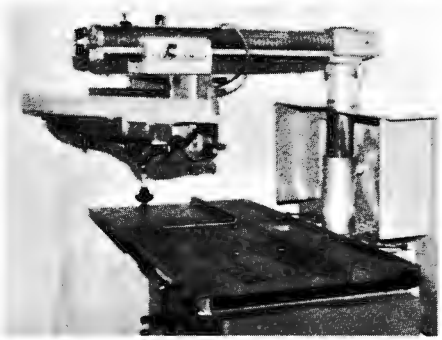


Figure 4.

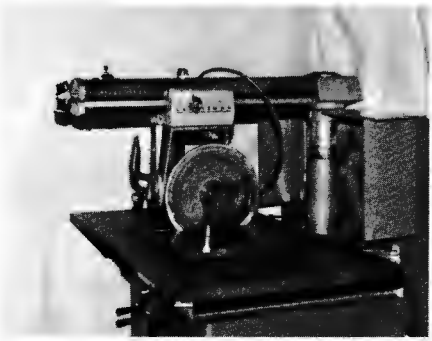


Figure 5.

Figure 6.

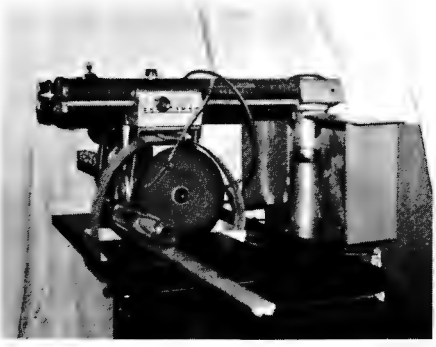


Figure 7.

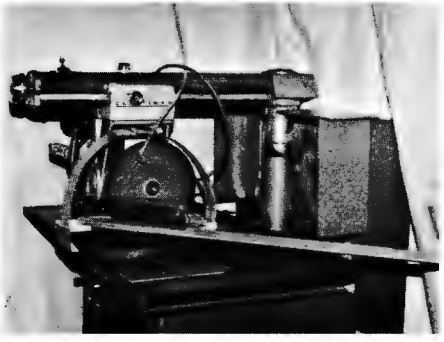
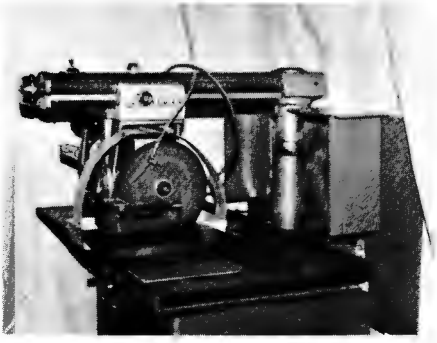


Figure 8.



THE DEVELOPMENT OF TOOLS AND FIXTURES

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INTRODUCTION

Last month the Zenith Electronic Corporation announced it is planning to build FTM tubes starting in 1987. In our labs in Glenview, Illinois, we have been working on the development of this type of CRT for several years. I would like to tell you, in this paper, a little bit about what a FTM is: Some of the work that went into building a few prototype tubes and most of all, about the tools and fixtures that were developed in the process of constructing a bulb.

FLAT TENSION MASK TUBE

The "Flat Tension Mask" cathode ray tube is a new kind of CRT that offers a host of benefits, including a perfectly flat faceplate, and improved resolution brightness, contrast and color fidelity. Unlike a conventional CRT (which uses a curved shadow mask suspended by springs inside a tube), the FTM display has a flat shadow mask that is held under tension and supported directly by the tube's glass.

CONSTRUCTION PARAMETERS

To build this type of tube, it was decided to use existing color television glass funnels and panels, modify them to our needs, and assemble a Flat Tension Mask cathode ray tube. This involved cutting of rings from the skirt of a conventional color panel, grinding them to a specified thickness, and ultrasonically drilling of very accurately located grooves.

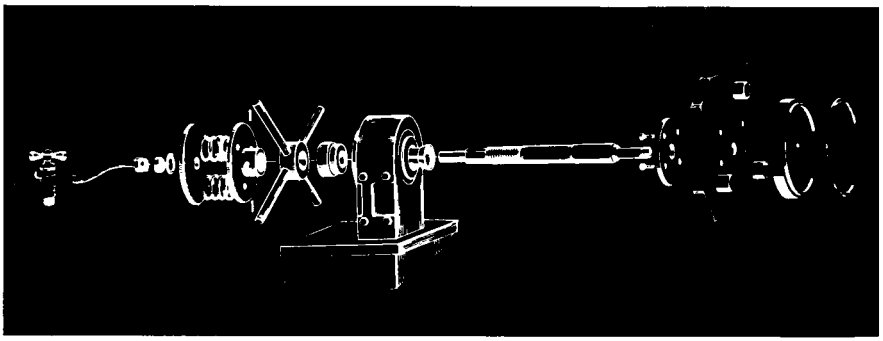
Drilling of grooves into a flat panel and a funnel. Also changing the neck of the funnel to be centered with respect to the grooves.

CUTTING OF RINGS FROM A PANEL

There are several ways of cutting rings out of the skirted panel such as thermally shocking with a hot wire cutter, or just grinding the panel off.

We decided the best way for us would be to build a vacuum holder. This holder would be mounted onto our diamond-blade cut-off saw.

VACUUM HOLDER



Panel Vacuum Holder

This holder is made of two hollow steel shafts. The inner shaft has a 6" dia. aluminum disc with an 'O' ring attached to one side and a hose-connector on the other end.

The outer shaft has a 3/4" thick aluminum disk which has three one-inch dia. posts mounted to it.

This shaft is held in a bearing block with a collet, the type that machinists use in a mill.

It has a lever: When it is pulled out it allows the collet to rotate to the desired position and is locked in place by pushing the lever in.

On the other end of the shaft are three compression springs held between two metal flanges. On the inside flange is a collar with spokes, threaded to the shaft.

A short piece of rubber tubing is attached to the hose barb, which is then attached to a 3-way stopcock which is connected to a vacuum pump with another hose.

CUTTING THE SKIRT OFF A PANEL

First we find the center of the panel by marking the approximate center with a felt marker. Then, while the ink is still wet, turn the panel over and spin it on a flat piece of glass. This smears the ink into a bullseye, the center of which is the center of the panel.

The center of the panel is visually aligned with the center of the aluminum disk while the stop-cock is rotated to allow the vacuum to pull the panel to the 'O' ring. Or to say it more correctly, let atmospheric pressure push the panel against the 'O' ring.

The collar with the spokes is then rotated to draw the panel against the 3 aluminum posts. This serves to take up the vibration we would have if the panel was only held by the 'O' ring.

The panel can now be moved, transversely, by sliding the outer shaft in the collet. When it is properly aligned with the saw blade, it is tightened by rotating the collar.

The saw is then started and a cut is made into the corner of the panel as deep as the saw blade permits, about 4". Then the panel is backed out of the blade, the handle on the bearing block pulled out, which allows the panel and shaft to be rotated to the next corner to be cut. This is done on all four corners. Only a small piece of glass is allowed to remain at the end of each sawcut. This is to support the ring in four places. These supports are then slowly removed by cutting them a small amount at a time until the ring drops gently into the operator's hand.

For each bulb two rings are required. One, one-inch thick is the backup ring, the other the O ring is about .200" thick. When cutting, it is important to have a good sawblade that runs true and the bearings on the saw spindle have no play in them.

This is especially true when cutting the relatively thin ring, where a little bit of vibration can snap the ring very easily, generally just before it is cut away from the panel.

GRINDING OF GLASS RINGS

A mixture of Silicone Carbide and water is made into a slurry. It is applied to a flat wheel either with a paint brush or a squeeze bottle. The wheel is then started and adjusted to run at about 60 rpm. The glass ring is hand held against the wheel in a big clockwise oval motion. This will remove the little glass support posts left during the cutting operation and result in a flat surface, but not parallel to the surface of the other side.

HYDRAULIC SURFACE GRINDER

To grind the ring to a uniform thickness we use a surface grinder. A 3/4" thick steel plate large enough to support a 14" glass ring is placed onto the magnetic

chuck and locked into position. Two clamps with soft rubber tips are bolted to the plate in the proper location. The glass ring is placed onto the steel plate and is held down by the clamps. The diamond impregnated wheel is started and allowed to spin about 5,000 rpm. A hydraulic pump is activated which will oscillate the table from side to side. The wheel is lowered to the surface of the glass ring. At the end of each pass of the table, the wheel is lowered .005". Water and rust inhibitor is used as a coolant during the grinding of the ring.

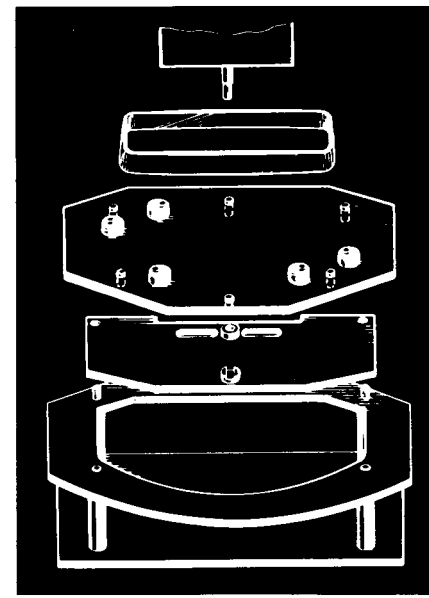
DRILLING OF GROOVES

For this operation we decided to use the ultrasonic drill we have in the shop. Unfortunately, it is only a bench model with a limited amount of table travel. A large support fixture had to be designed and built before we could start to drill the grooves.

SUPPORT FIXTURE FOR ULTRASONIC DRILL

It consists of 3 aluminum plates; a baseplate is bolted to the bottom of the drill. It has four posts mounted to it. On the posts are two plates; one is fixed to the posts, the other is movable. On top of that is the product plate.

With this arrangement it is important to align the baseplate with respect to the drill bit location. A centering fixture is placed on the baseplate. It engages a hole and a groove with dowel pins that are located on the upper and lower end of the baseplate. It is then moved until it is centered in the fixture. Two bolts are used to lock the two baseplates down. The locating fixture is replaced with the product plate. The glass ring is now placed onto the product plate and held by 5 cams. The drill is lowered to touch the glass ring. A small amount of silicon carbide and water is brushed on the drill area. The ultrasonic unit is then activated and allowed to cavitate to the correct depth into the glass.



Support Fixture for Ultrasonic Drill

To drill the next groove the product plate and glass ring are rotated until the next set of dowel pins can be dropped into the hole and groove in the baseplate. The same process is also performed for the 3rd groove to be drilled.

Grooves are also drilled by this method into the panel and funnel for a total of 12 grooves per bulb.

SEALING OF MASK TO GLASS RINGS

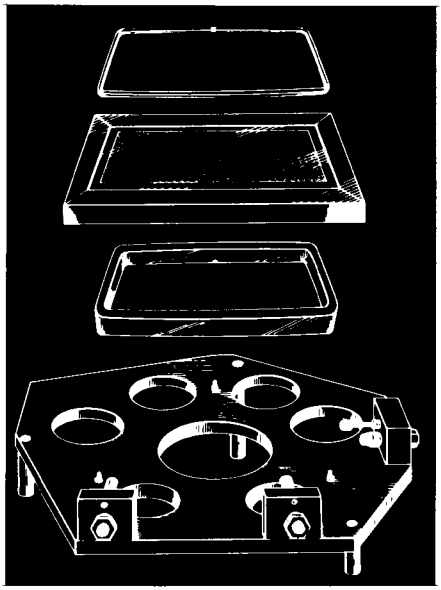
A layer of solder glass, or frit, is dispensed on the ground side of the rings. In the green state it is about .060" thick and about as wide as the glass ring. It is then set aside to allow the binder to dry.

The mask, which is a .001" thick cold roll foil with holes, is then stretched over a cold roll iron "L" shaped frame and is spot-welded into position.

To hold the rings and mask in place during the frit seal cycle we needed another fixture.

FRIT FIXTURE

We started with a baseplate of 3/4" thick steel cut in 7 holes and mounted three studs into it. On the bottom are three support legs at different lengths, to tilt the plate about 15°. On the lower side and on the right hand are steel blocks with threaded holes. These holes are designed to accept threaded rods with graphite caps.



Frit Fixture

ASSEMBLY OF MASK TO RINGS

To assemble the parts, the thick glass ring is placed onto the studs on the bottom of the plate engaging the grooves that were previously drilled. Then the mask and frame assy. is draped over this ring. The threaded rods are adjusted to locate the mask in the center of the ring and then are kept in place by locking nuts. The thin ring is placed on top of the mask and a drilled panel, which in this case acts as a fixture, is placed on top of the ring. It is held in place by small glass balls that engage the grooves in the ring. Then it also is centered by the second set of threaded rods and locked into position.

Now the whole assembly is placed into an oven and run through a heat cycle of 435℃ for one hour.

During this cycle the mask and frame expand more than the glass rings. At 435℃ the frit begins to devitrify and after one hour at this temperature the mask is sandwiched between the glass rings. During the cooling of the assembly the mask wants to shrink more than the glass will permit: This causes the mask to be held in tension.

SCREENING OF PANEL

It is very important in this operation to have good reregistry. The ball and groove arrangement allows for the panel to be removed and replaced very accurately.

FUNNEL NECK ALIGNMENT HOLDER

A steel plate 1/4" thick is cut to the contour of the sealing edge of the funnel. Three grooves are ground at the proper location. A 2" round shaft that is 2" long is attached to the back with two (1/4 - 20) bolts.

The funnel is held sealing edge up and three glass balls are dropped into the grooves. The steel plate is placed on top of the funnel allowing the balls in the funnel to engage the grooves. Ordinary masking tape is wrapped around the lip of the funnel and the holder. This tape is strong enough to hold the funnel and make a good enough seal to allow us to blow into it during neck sealing.

ALIGNMENT PROCEDURE

The funnel and holder are chucked into a lathe. When the lathe is turned on, the neck will not be running true, only the grooves are centered at this point. The neck is therefore cut off, using a scribe and a small flame to heat shock it.

A new neck that has been chucked into the tailstock is brought to within about 1/4 of an inch of the funnel opening. Both the funnel and the new neck are preheated. When they reach the proper preheating temperature, they are spliced together with a gas-oxy 4 burner. After the seal is completed and the glass is still a little soft, a preheated graphite paddle is placed on the neck and slowly pushed toward the funnel to get the contour necessary to later allow a yoke to slip on. The yoke area is then annealed with a gas air radiant burner.

BULB ASSEMBLY

At this point we have a panel with a screen on its inside surface. A mask held between two glass rings, a funnel that had its neck changed, and then coated with dag.

Electrical contact is made with the use of springs from the screen to the mask and from the mask to the funnel.

To assemble the bulb, the funnel is placed into a holder seal edge facing up. A frit bead is depostied on the sealing edge and three glass balls are pushed into the grooves and then allowed to air dry.

The mask assembly is placed onto the funnel and another frit bead is dispensed onto the seal edge of the small ring and three glass balls are pushed into the grooves. The faceplate is lowered onto top of this stack.

This assembly is placed into an oven and run through a frit cycle of 435℃ for one hour.

At the 380℃ the solder glass begins to melt. Gravity can now push the panel and mask assembly onto the funnel, closing the gap from .060 to about .005. The glass balls engage the sides of the grooves for good alignment of the components; excess frit will be squeezed out on the sides to form a fillet.

GUN SEAL

The bulb is then held in a glassblowing lathe. A level is placed on the outside edge of the panel in line with the phosphor dots of the screen. An electron gun is inserted into the neck of the bulb and is visually aligned with a scope. The press of the gun is flame sealed with a gas oxygen two burner, then it is annealed with a gas air radiant burner.

The FTM bulb is now ready to be evacuated on a pumping station.

OBTAINING THE ELUSIVE JOURNAL ARTICLE

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If I say the word "library," what come to mind? Think about it for a second, maybe repeat the word to yourself a few times. For many people the word conjures up images of rooms with booklined shelves, dusty volumes, bespectacled staff members (probably wearing sensible shoes . . .) doing their best to keep the noise level down. But is this a true reflection of today's libraries? In some cases, yes, but in most it is way off the mark.

Libraries and librarians have always been information specialists, but few called them that before the "Age of Information." As computers have changed the way scientists work, they have changed the ways that librarians do theirs. Armed with computers we experiment with electronic document delivery, telefacsimile, electronic mail and hundreds of databases filled with all sorts of information. Nearly everything from coffee prices to full-text articles from *Analytical Chemistry* is available with the touch of a few keys.

So, why am I telling you this? That's a fair question. I've been asked to talk to you today about what librarians call "document delivery." Translated into English, that means bringing a library patron and a desired piece of information together. Most of you read the A.S.G.S. journal, *Fusion*. In each issue is a listing of papers that are relevant to glassblowers and glassblowing. Picture yourself in your lab, skimming your latest issue of *Fusion*, and an article catches your eye. You don't own a subscription to the journal it's in, so what do you do? The answer is obvious, but not simple. You go to the library.

As a national organization, your members come from all over – universities, corporations, government labs, etc. You may have access to large research libraries, small special libraries, or perhaps no library at all. Depending on your own situation, your next step will differ, though we hope the end result will be the same.

The scientific literature is based on a core of journals for each discipline. Societal publications, such as those published by the American Chemical Society or the Royal Society of Chemistry, form an integral part of the core journals for chemistry. In an effort to build complete collections, university and college libraries try to subscribe to all the core journals. Exactly which journals belong in the core, or, for that matter, how many journals comprise the core, is open to debate. Once source suggest that there are 5000 or so core journals in science, but that includes everything from botany to high energy physics. *Chemical Abstracts* contains articles from more than 19,000 serials, so you can see estimates vary. Only the very large, well-financed libraries can afford to buy all of them, so you may be disappointed when you check your card catalog. If you have any questions regarding the use of your catalog, please ask for help.

If you work in the private sector, you may or may not have access to a special library. Although some special libraries are quite extensive, like those owned by Bell Laboratories, others are rather small, and may not have collections of the older years of journals. If you do have access to a technical library, check with your librarian for the issues you need.

For those of you who don't have a library where you work, there is, of course, your local public library. Today's public libraries have more than the latest fiction,

auto repair books and cookbooks. Even small libraries are connected one way or another to computer networks that link libraries around the world. It is true, though, that very few public libraries have extensive technical collections. A few public libraries, such as New York Public Library or Milwaukee Public Library, are well-known for their scientific collections. Those that do not rely on state and local area networks to borrow the materials that they do not own.

Which brings us to my next topic – interlibrary loan. When a library doesn't own a particular item that a patron wants, they contact other libraries that do own it, and ask to borrow the item. Nearly everything is available through interlibrary loan, including books, journal articles (but usually not entire issues of journals), patents and government documents. In order to use interlibrary loan, it's not necessary that **you** know who owns what you want. Libraries have a wide range of resources available to them that list locations or holdings. For books published before 1956 we use the *National Union Catalog, pre-1956*, a 754 volume set listing hundreds of thousands of authors and titles, and the libraries that own them. More than 700 libraries in the United States are listed. While not 100% comprehensive, it's pretty close. Later years are covered by other editions of the *National Union Catalog*. For journals, Interlibrary Loan departments use a similar set, called *New Serials Titles*. It, too, has supplements to keep it up-to-date. However, computer networks, such as OCLC or RLIN, now list locations for books and journals, and many libraries take advantage of these systems. OCLC, for example, is used by thousands of libraries nationwide, and recently added its ten millionth record.

Aside from the national networks like OCLC, states and regions also form interlibrary loan networks. The states in the Southwest form a group called AMIGOS, and public and academic libraries in the state of Kentucky loan and borrow books with the help of a network called KEN-CLIP.

Interlibrary loans are not free. Some libraries cooperate with exchange programs, where items to and from these libraries are loaned for free. Others charge $10 - $15 per item. Photocopies of articles can cost more. There is usually a surcharge of $2 or $3, and then a per page charge of 10¢ - 15¢, depending on the lending library. If you work for a corporation, your interlibrary loans may be paid for. Academic libraries sometimes pay for all loans or copies, but others pass on some or all of the costs to the patron. The same applies to public libraries. When in doubt, ask.

You can obtain copies of government documents or patents through interlibrary loan, too. If you are fortunate you may be living near a patent or government documents depository library. Most states have at least one depository for federal documents, and they are often affiliated with universities or research institutions. Other libraries may function as partial depositories, selecting documents in a particular range of subject areas. A depository library is required by law to keep one copy of every unclassified document and have it available to the public. Patent depositories keep one copy of every U.S. patent. Many hold inventors workshops to teach would-be inventors how to do their own patent searching. The Cincinnati Public Library is a regional patent depository library.

Time can be a problem with interlibrary loans. Delays caused by the mail, processing, even three-day weekends can create three to four week delays in the arrival of your book or article. If you need your article right away there are other alternatives. One is the use of telefacsmimile. A telefacsimile machine is very similar to a photocopier. It transmits a copy of a document, one page at a time, electronically over telephone lines to a telefacsimile machine on the other end. The transmission is instantaneous. There are two drawbacks: both the price of the machine and the resulting price of the copy are very high, and most libraries do not

own telefacsimile equipment. Another alternative would be overnight delivery services, but you must be prepared to pay the extra amount.

Some journals are found in online databases. The eigthteen American Chemical Society journals are available full-text through a vendor called BRS, Bibliographic Retrieval Services and also through their own database, CAS on line. The rates for accessing the database or BRS are high, averaging $85 per connect hour and a 51¢ per page print charge. You must also have a contract and password to access the databases.

Still another alternative to interlibrary loans is to order documents through a document delivery service. A variety of companies offer document delivery, including University Microfilms, ISI (the people who publish *Science Citation Index*), the British Lending Library and Chemical Abstracts. The availability of documents varies from vendor to vendor, usually by year or by subject category. Chemical Abstracts charges $14.00 per item, regardless of length. You can use regular mail, electronic mail or their toll-free number to order the documents, and some even accept Mastercard! Your article will be on its way within 24 hours, and you should receive it within a week.

I have spent these past few minutes telling you different ways that libraries get materials for you, and how you can get them yourself. However, none of this will be of any help to you if you forget that first crucial step – asking for help. If you don't find what you are looking for, please don't be afraid to ask for help. After all, that's what we're there for.

I would like to thank Roxanna Jones, Head of Interlibrary Loan at the University of Kentucky for her help in preparing this paper.

CONSTRUCTION OF SPHERICAL DEWARS

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This paper will discuss several different methods of constructing Spherical dewars without packing and without blowing, a step by step. Also covered will be construction of hemispherical dewars using a round bottom flask.

The flasks that we will use as an example will be A 3 L inside A 5 L. In all of the procedures the outer (5L) flask is prepared the same. See Figure 1. I changed the neck to 85mm diameter and about 55mm long and sealed a piece of 1/2" MW tubing on to the bottom, this will be used for holding and, in the final phase, for silvering and evacuation. At this point I oven anneal this piece.

The inner flask (3 L) can be prepared in a couple of different ways depending on how you are going to hold it while making the neck seal. Figure 2. The neck is changed to a 60 mm diameter by 60 mm long. Now, if you are not going to use an internal holder, you will want to seal a rod onto the bottom. I use 1/2". This piece is now oven annealed. The 5L flask is now put back in the lathe and cracked in half using a diamond pencil. See Figure 3. What happens next will depend on how you decide to hold the inner flask. Figure 4. If you have a lathe with a six jaw chuck you may want to consider the top example, holding the rod with the 3L flask in the inner jaws and the outer flask in the outer jaws. I usually wrap a couple of layers of masking around the chuck arms to cushion the 1/2 outer flask in all of these methods and avoid any other wrap. The bottom method is my least favorite. See Figure 4. That involves the use of a tube wrapped with quartz tape as an internal holder. Figure 5. This is my favorite method and it involves the use of an internal adjustable holder. At this point I make the seal using a graphite rod to bring the inner tube up to the other tube.

Figure 6. After the neck seal has cooled we are ready to finish the bottom. If you used the rod as a support, this will be removed at this time. Holding the neck in one end of the lathe and the bottom half in the other end, the final seal is made. This can be done quite simply without blowing, although you may want to connect a blow hose to clear the flask of any trapped gas before making contact. We have a large number of these flasks in use in our department. Some have been in use over 20 years and as large as a 6L flask inside of a 10L flask.

The second dewar I would like to describe is hemispherical and is made from a 2L flask. The flask is mounted in one end of the lathe and a graphite rod in the other. See Figure 7, top. The bottom of the flask is heated with a large bushy flame and flattened. See Figure 7, bottom. The graphite rod is brought into contact with the flat section you continue to heat and slowly push the bottom into the desired depth. See Figure 8. As a final step you will want to suck slightly to give the desired shape. See Figure 9. You will need to take some care in doing this because what needs to happen is that you stretch the inner part slightly, the 75 mm depth inside will be contained in 90 mm of outside which is longer than half of the diameter of the original flask. The neck of the flask is then constricted to proper size for the silvering and evacuation.

The silvering procedure that I use is from London Labs and the dewars are pumped to a vacuum of $5X10^{-7}$ Torr. As bases for my dewars I have the machine shop cut and roll aluminum .040" - .050". I drill them and fasten them together with pop rivets. See Figure 10. I pot them using blocking pitch.

In closing, I would like to mention that the drawings here were done on a computer and say a word of thanks to my son, Dustin, for doing them.

Figure 1

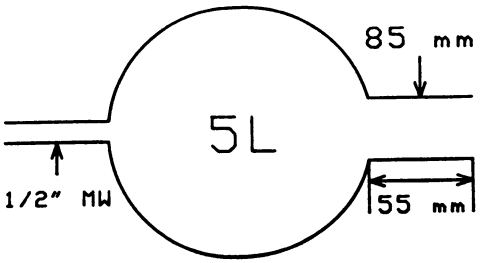


Figure 2

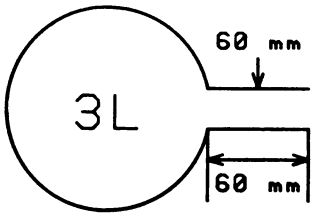
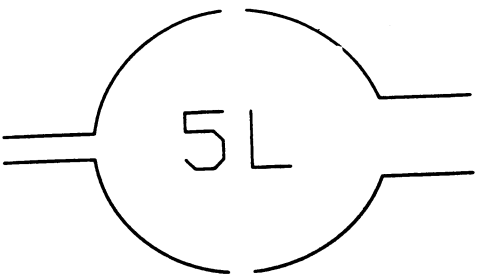


Figure 3



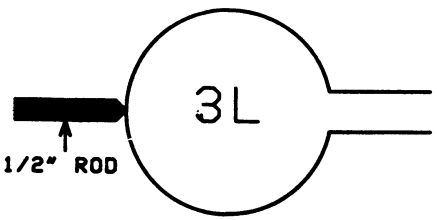
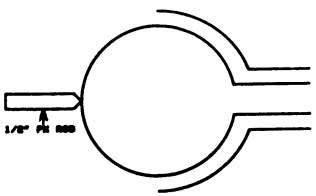


Figure 4



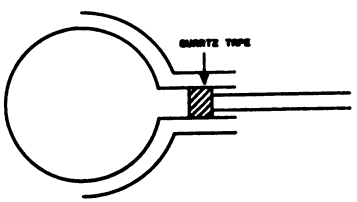


Figure 5

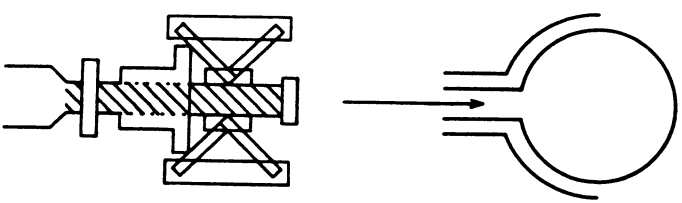


Figure 6

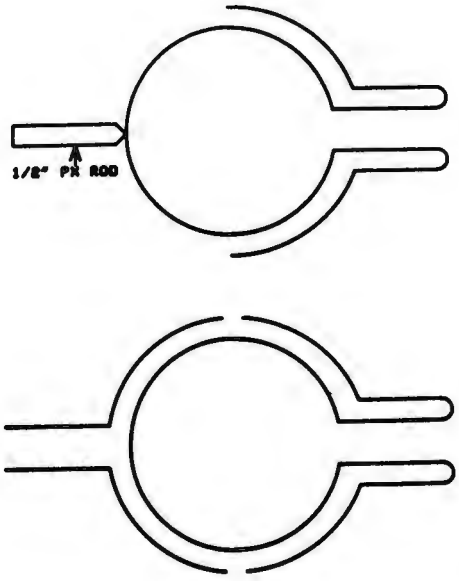


Figure 7

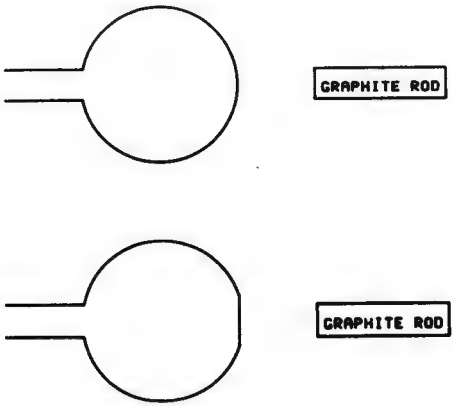


Figure 8

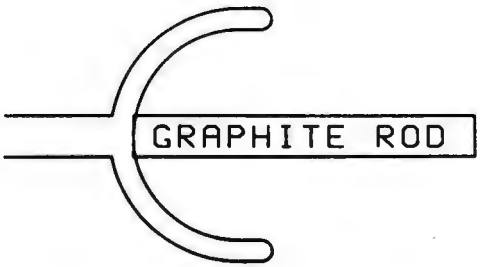


Figure 9

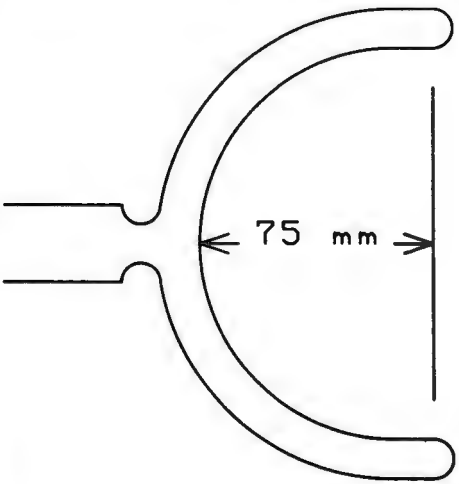


Figure 10

NEW COMMERCIAL SEALING GLASSES

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ABSTRACT

This paper will attempt to guide the glassblowers in their selection of newly developed low temperature sealing glasses. The following topics will be emphasized:

What Sealing Glasses Are Available, How to Use Them Effectively, What Is Commercial Production Experience In Using These High Technology Glasses.

Tables of physical and chemical properties will include the commercial designation of these materials. Special solder glasses used for sealing low thermal expansion materials as well as relatively high expansion materials will also be covered and their properties tabulated.

1. INTRODUCTION

Glass technologists recently developed new low melting sealing glasses called solder glasses suitable for sealing assemblies which deform at elevated temperatures (e.g. color television or electronic devices which may be damaged by heat exposure (e.g. phosphorous, silicon, MOS, sensors and others). These new sealing glasses cover a relatively wide range of application temperatures from 350°C to 620°C (662°F to 1150°F).

Logical grouping of these new solder glasses is shown in Table I. From these five types Television, CER-DIP and Special sealing glasses for low and high thermal expansions will be covered in details.

Furthermore, these glasses are vitreous (commercial designation SG = solder glass) or crystalizing (C.V. = crystal-vitreous) and single or multicomponent. (Glass only or Glass and adjusting additives.)

TABLE I – SEALING GLASSES TYPES

TYPES	EXAMPLES
GLASS-GLASS	TELEVISION - MONOCHROME
GLASS-METAL	FEED THROUGH WIRE
GLASS-CERAMIC	ROM - WINDOW
GLASS A - SOLDER GLASS - GLASS B	TELEVISION - COLOR
CERAMIC-GLASS-METAL-GLASS-CERAMIC	CER-DIP

Vitreous solder glasses remain glassy during and after sealing. On the other hand, **crystalizing** solder glasses are changing into ceramic structures with crystals and glassy phase.

Furthermore, these two types of solder glasses differ in desired properties which make them applicable for a variety of sealing processes and final products with specific seals specifications.

Vitreous solder glass seals have the following advantages:

1. **Salvaging** of parts is achieved by remelting the sealed joint without significantly changing the glass properties.
2. **Annealing** can adjust the seal joint stresses.
3. **Expansions** and contraction can be adjusted by additives.
4. **Sealing time** will decrease at increasing peak holding temperature.

Between the two disadvantages, neither of them is so serious as to limit the vitreous solder glass application.

1. Slightly sensitive to thermal shock, due to cooling temporary stresses.
2. Chemical corrosion is usually somewhat higher than the crystalizing sealing glasses.

Without available **crystalizing** solder glasses, color television bulbs could not be exhausted and evacuated at desired temperatures. The main **advantage** of these glasses is their rigidity at temperatures not far from sealing temperature. (slightly higher) Also, **chemical corrosion** resistance and **mechanical shock resistance** are usually higher than vitreous solder seals. Although the crystalizing solder glasses accomplish their intended mission very well, the processing must be well controlled to achieve desired crystaline structures.

2. COLOR TELEVISION APPLICATIONS

Table II. The selected solder glasses are sold in a powder form which the customer processes (prepares) into a relatively thick paste with additions of selected vehicles. This paste with high glass content is extruded by dispensers on the funnel part of the assembly. The deposition process is controlled to yield high production with miniscule losses (1/1000). The controlled shape, spacing on sealing edge and weight of paste is achieved daily. The weights may be within $\pm$ 2 gms in control. Funnel with dried solder glass paste is placed into a proper fixture with the neck (narrow portion) down. Face is placed in contact with solder glass in desired orientation maintained by fixtures. This assembly is sealed in lehr with the following typical schedule:

Heat-up rate: ranges from 7℃. to 10℃. per minute to minimize the thermal shock of relatively thick glass parts with somewhat higher expansion and to assure complete burning-out of organic vehicles. Incomplete removal of vehicles could chemically reduce the lead glasses and they in turn can cause high voltage punctures.

Around 400℃, the solder glass "fluxes" (= reacts with = dissolves) both glass parts of the color television assembly. After about 10 minutes at peak temperatures, the original vitreous glass begins to crystalize and the fluxing action is slowed down. The crystalization takes about 20 - 28 minutes to achieve desired strong structures. Then the assembly is cooled down at the rate of 5℃. to 10℃. per minute to eliminate any breakage due to thermal shock.

TABLE II – TV SOLDER GLASSES

	CV-808HD	1307B	1304	8363
Density GM/CM^3	6.56	6.3	6.3	6.42
Expansion x 10^{-7}/℃.	100	99	99	100
Contraction x 10^{-7}/℃.	106	102	102	104
Annealing Point ℃.	300	330	320	304
Softening Point ℃.	370	400	400	375
Volume Resistivity				
250℃ Log OHM-CM	8.1	7.8	7.7	9.2
350℃ Log OHM-CM	6.6	6.4	6.4	7.5

TABLE III – CER-DIP SOLDER GLASSES

	CV 111	XS 1175-Mi	XS-1190	SG-95	SG-202	SG-200	LS-0802	LS-0803	LS-0120
1. Density	5.85	4.25	4.75	6.8	5.2	5.61	6.78	7.19	6.92
2. T. Expansion							77	67.5	67.5
3. Contraction	70	74	74	73	70	63			
4. Annealing	318	305	305	310	307	295	310	300	315
5. Softening	380	350	350	370	355	345	360	350	385
6. Volume Resistivity									
at 150℃.	9.7	11.5	11.5	12.0	11.7	11.1	10.5	11.0	11.5
at 250℃.	8.6	9.1	9.1	9.5	9.2	8.8	8.5	8.9	9.0
7. Dielectric Constant	16	12	12	40	12	16	31	35	31
8. Alpha Radiation		2	0.2	10	1.0	0.2			

NOTES AND UNITS

1. Units GM per CM^3
2. Range –– 30℃ - 250℃.
3. Units x 10^{-7} per ℃.
4. Units Point ℃.
5. Units Point ℃.
6. Units Log OHM-CM
7. At 25℃. 1 MHZ
8. Counts per CM^2 per hour

3. HERMETIC PACKAGING CER-DIP

Table III. Hermetic Package includes two ceramic parts (base and lid), metal lead frame and two solder glass sections which make the final seal.

The supplier to this industry preglazes the two ceramic parts by silk screen printing and glazing the ceramic parts. The **glazing** is done in lehrs in air atmosphere to remove the organic vehicles. The glazing temperatures range from 320℃. to 430℃. at the peak with times from 4 - 12 minutes.

Lead Frame of proper metal (Kovar - 42, 45 alloys) is sealed to the ceramic base by rapidly heating the glazed ceramic base above the recommended final sealing temperature (±30℃. ± 50℃., - 2 minutes). The lead frame is pushed into molten glass and this subassembly is cooled. After this operation the silicon active device is bonded into the ceramic cavity and the thin wires attached to it and to the lead frame. This operation is followed by the "closing" sealing part of the process which seals the active device by attaching the lid to the base. The heat-up rate varies from 20℃. to 140℃. per minute. The peak temperatures range from 390℃. to 500℃. with hold times from 4 to 12 minutes. The last part of the process, the cooling rate is from 7℃. to 60℃. rate per minute.

TABLE IV – SPECIAL SOLDER GLASSES

	SG - 7	SG - 67	CV - 432	SG - 100	CV - 455
1. Density	4.07	5.38	6.55	6.7	5.9
2. Expansion	41	83	117	81	86
3. Contraction	60	102	127	90	89
4. Annealing	469	365	290	312	315
5. Softening	571	441	327	361	365
6. Volume Resistivity					
at 250℃, Log OHM-CM	12.4	11.1	6.6	11.4*	10.4*
at 350℃, Log OHM-CM	10.5	8.9	5.0	9.0	8.3
7. Dielectric Constant	8.2	12.5	27.3	31.8	18.3
8. Applications	Kovar EN-1 N-51-A S.S. Alloy	SodaLime Platinum #4 Alloy	Metals Irons Nickel Aluminum Coppers	Glass Panels	Glass Panels
	Low Exp	Med Exp	High	Vitreous Med.	Crystal Med.

Notes and Units

1. Units GM per CM^3
2. Units x 10^{-7} per ℃.
3. Units x 10^{-7} per ℃.
4. Point ℃.
5. Point ℃.
7. at 25℃. 1 MHz
8. N-51-A

* at 150℃

The lead frame is tin plated and the DIP assembly, including the sealing glass, is immersed into acid de-scaling solutions. The solder glasses with relatively high lead oxide content are normally subjected to sulfuric acid. Hydrochloric acid is used for glasses with relatively lower lead oxide content (e.g., SG-200 and SG-202). Nitric acid is used only in very low concentrations (e.g. 2%) to minimize any solder glass removal.

4. SPECIAL SOLDER GLASSES

Table IV lists the most useful sealing glasses for low temperature joining of metals, glasses, and alloys in order of increasing thermal expansions with the last two glasses sold for sealing display panels. SG-7 with lowest thermal expansion of 41 is used for Kovar, S.S. Alloys - EN-1 and N-51-A glass. CV-432 is good sealing glass for high expansion metals like nickel, aluminum, copper and their alloys. SG-67, one of the first developed vitreous glasses, has medium thermal expansion which made it useful for original color television panel sealing but now it is sold for sealing soda lime glasses, No. 4 alloy and platinum. The development of sealing glasses is continuing primarily in hybrid electronics industry – in lead-free glasses for alumina to eliminate the reduction of lead oxide glasses fired in reducing atmosphere. The low expansion lead-free glasses for Kovar and S.S. alloys are also available. Glass technologists are putting some effort into decreasing temperatures of all these glasses.

5. CONCLUSIONS

1. Commercial low temperature sealing glasses were described and applications detailed for color television and CER-DIP.
2. Special sealing glasses for expansion from 50 - 160 were reported and their physical properties were tabulated.
3. The low melting glasses are expected to meet the needs of joining high technology materials at safe temperatures and time cycles.

PRIVIAL PURSUIT

William E. Caldwell
J. Matthew Hughes

INTRODUCTION

Urban archaeology has taken place for years by people in search of artifacts from generations before us. With a broad range of artifacts to search for, the authors narrowed their interest to blown glassware and have limited their search areas to privys (outhouses) and pre-civil war dumps.

DISCUSSION

Greater Cincinnati to include Covington and Newport, Kentucky, was settled in the latter quarter of the 18th century. These settlers came down the Ohio and built homes on each side of the river. By 1840 there were 40,000 people living on what is now referred to as the downtown area.

The homes being constructed were small frame and brick houses with maybe as many as four rooms. One very important room was going to be missing for a long time to come; a bathroom. Indoor plumbing was not yet available so an outhouse, was the only answer. The construction of the outhouse was by owner's choice; he had three. One, a brick-lined outhouse; two, a rocklined outhouse, or three, the earliest example and easiest, a woodlined outhouse. These holes on the Cincinnati side of the river have been excavated by hand to a depth of 45 feet. Each area of town has its own trend of depth, more than likely based on the water table. The Kentucky side of the river outhouses run shallow compared to Cincinnati, with the deepest maybe at 22 feet. Almost always circular, the brick and the rock-lined use no mortar as the walls are stacked carefully because the hole receives its strength from the uniformity of the circle. Woodlined outhouses were simply a large rectangular hole dug with a wooden box without a bottom, dropped into the ground. In all 3 examples a shack of wood was constructed over the hole.

At this point with the most important amenity to a home constructed, how is the author going to attribute bottles and glassware winding up in the outhouse? There was not any form of trash collection for years to come so all trash (bottles, glassware, pottery, etc.) went into the outhouse almost as regular as, **Wednesday** the trashmen come.

With information of types of and how the trash (bottles, etc.) got into the outhouse, the most critical part left to come is the location of the proverbial privy. The line "fish where the fish are" applies here. If you are looking for pre-civil war glassware, locate pre-civil war dwellings. After a familiarization with architecture, it wasn't long before certain areas of town became our favorites, simply due to when the area was constructed. Almost all of the lots where these early homes were built are very narrow and not always deep. Upon qualification that the dwelling is pre-civil war, permission is sought and suprisingly more often than not, obtained. With a rod made of spring-steel to use as a probe, the first order of business is to find the lot lines. The aroma of these outdoor toilets back then, I'm sure, resembled the odor of a port-a-john. With this in mind the construction of the hole was as far from the house as possible. So the back lot line is probed first. Of all the rules and trends that you would figure could be established, one of the most recurring is the outhouse is most generally on the side of the yard not visible from the street. The houses were seldom built in the middle, so the narrow sidewalk that runs to the back yard is a tip-off that the outhouse is on the opposite side. Probing is an art gained only from experience. The feel of the different soils is the key to the privy.

Probing the ground for soft area along the back lot line is the quest. Virgin soil being very tight makes the material in the top of the outhouse really stand out. Upon finding the soft soil, the tip of the probe is checked for an example of the soil. Probes differ in length from 4 to 7 feet, but in our area dark dirt in a soft spot recovered 4 to 7 feet beneath the ground usually means that the sub-terrain has been distrubed; but not always is there an outhouse. With the probe angled into the ground, we try to find the walls by walking in a circle probing outward. Looking for a consistent wall by continuing the probing is the best shot for establishing location. After confirming the consistency of the wall, a test hole is begun to uncover the wall. In digging the test hoels close observation is kept in regard to shards of glass, pottery etc. that may verify the age of the hole and its contents.

At this point an explanation of our success to failure rule is in the best interest of the reader. More often than not behind these dwellings there is more than one outhouse. The bonanza is to find the old one on your first try. Not easily done. You also might ask why more than one. If the original was too shallow it would fill quite often and the owner would then have to pay to have his outhouse cleaned. So the construction of a new, deeper one might be cheaper than continual cleaning. Also the original home was small and large additions were commonplace and might require relocation of the outhouse. The other success failure rate stems from when the home received city water. The earlier the better, because of the point where the toilet moves inside the outhouse was used exclusively for trash. Another tid-bit; some outhouses were cleaned very close to the time the home got indoor plumbing, so the hole consisted of mostly fill material instead of actual trash. All outhouses excavated have fill material. This must be removed in order to dig the actual privy layer (human waste and trash).

Items used to excavate are shovels, ropes, garden scratcher, clevis hook, and a walkboard to cross the hole so buckets of dirt can be removed without rubbing the sides of the outhouse.

The dig itself is basically hard work until the excavation reaches the privy layer and then the work seems easy. Careful extraction of the dirt is a must because many of the bottles may be grouped together because they fell together. Breakage is not our favorite word; of all the bottles and glassware a 50 percent recovery rate might be high. The excitement level rises as the rate of material leaving the hole slows because of glassware of all kinds, broken pottery, mineral waters, flask, puffs, etc. There isn't any average number of bottles pulled from a hole; it varies from none to 200.

IN CONCLUSION

One of the questions most often asked about this pursuit sometimes referred to as Privial Pursuit is; how do you know when you're at the bottom of the hole? The hole is over when you have hit packed clay or more important, when the walls stop. When the walls stop, get out . . . With 150 plus digs behind us there has only been one accident and it did result in a death. However, with proper precautions this unfortunate type of accident can be avoided. When one toils beneath the surface in a hole that was used 150 years ago, your success and fate fall heavily on luck.

SPOON GAUGES

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The spoon gauge is named because of its resemblence to the shape of a spoon. However, correctly described, the name changes to Deformed Bulb Type Bourdon Gauge. The sensitivity of these gauges is primarily dependent upon the thickness of the walls of the bulb, a fact which imposes limitations with respect to handling during assembly and to the capability of the gauge to respond to a workable range of pressure. An additional chore is the high reject rate during manufacture.

This paper deals with the procedure for making strong, easily assembled gauges which utilize the rotation of a quartz fibre about its longditudinal axis when subjected to tangetal forces exerted upon it by the movement of a rod attached to the closed end of a deformed bulb responding to pressure differences applied to its interior and exterior surfaces.

The first step in the construction of the gauge is to blow a few bulbs. Ideally, the elongated bulb should measure 60 mm in length and 35 mm in diameter. Starting materials are 9 mm OD standard wall tubing, and 2 mm rod. Seal a length of rod of not less than 200 mms to one end of a piece of tubing. Practice blowing up thin wall bulbs which have even wall thickness. Allow a soft flame to collapse one side of the bulb slightly beyond the central line. If suction is applied to the bulb and the rod tip moves sideways 8 to 10 mms, the bulb possesses a usable degree of sensitivity without being unreasonably fragile. A selected bulb is sealed into a 45/50 cone so that only the rod protrudes beyond the terminal end of the cone. The cone portion of the gauge has a side arm sealed on in order to adjust the pressure within the body of the gauge. It is important to position this side arm for convenience, bearing in mind that the spoon rod will move vertically from up to down with the deformed (or concave) side to the spoon facing downwards when negative pressure is acting on the inner surface of the spoon.

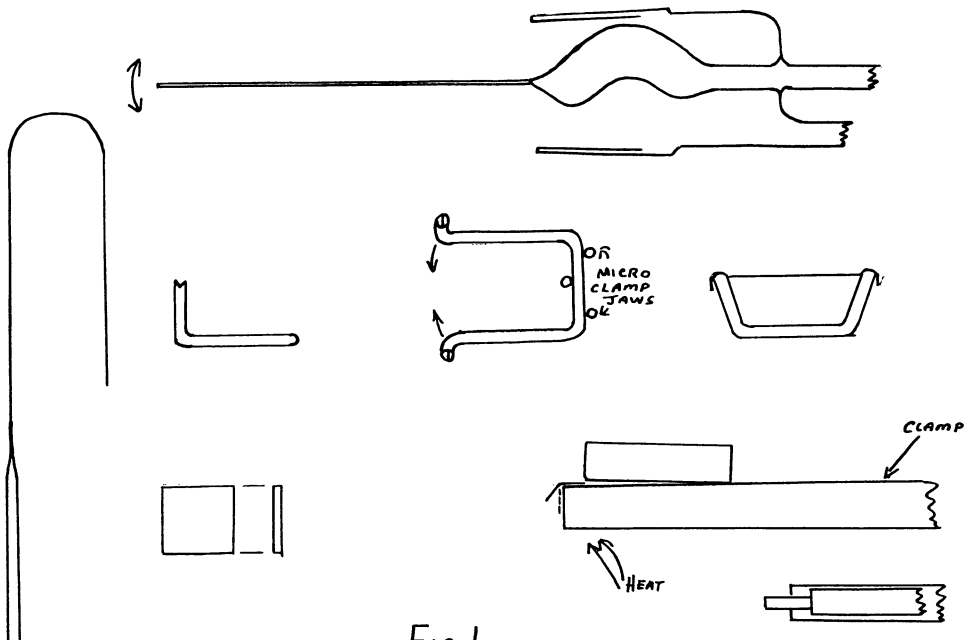

FIG 1

The socket portion of the gauge is of a length such that it will contain the rod plus 10 to 12 mms clearance to the end, which is flanged out 8 to 10 mms greater than the tube; this flange is ground flat and finished with 600 grit.

The remaining components of the gauge consist of a carriage upon the upward pointing arms of which is cemented a fine quartz fibre which is under constant tension, and a small galvanometer mirror waxed onto a glass bracket cemented to the centre portion of the fibre. These several subassemblies are shown in Figure 1.

The width of the carriage is such that it will sit within the tube with its upraised arms level with the diameter of the tube in the horizontal plane. The length of the carriage isn't critical; 35 to 40 mms is good. The surfaces of the ends of the carriage arms are notched to provide a seating for the fibre, and the notched surface should be rough so that it provides a keyed surface for the cement. The next step is the pulling of the fibre. The fibre need not be excessively fine because of the mechanical advantage of the gauge movement. It has been found that if a fibre 15 to 18 inches long is held vertically upward so that the free end curves gently downward to form an inverted U, then the middle third of the fibre is suitable for mounting on the carriage.

The length of fibre must be such that it will overhang the arms of the carriage on both sides by 4 to 5 mms. Both ends of the fibre are bent at right angles to form a shallow U; the base length is 1.5 to 2 mms less than the outside length of the carriage arms. When mounted for use, the fibre must be prestressed in order to prevent it from bowing downwards when subjected to pressure on the mirror bracket. The bowing movement would introduce errors in the early stages of the reading cycle until the bow limit has been reached. In order to introduce tension, the rear cross member of the carriage is clamped to a three jaw conventional laboratory micro clamp aligned so that the single jaw is on the inside surface of the cross member and the parallel sides of the carriage are at right angles to the closing plane of the clamp. By gently tightening up the clamp, the arms of the carriage will be forced together sufficiently to accept the fibre with the two ends laying along the outer surfaces of the arms. Carefully adjust the geometry of the carriage so that the sides of the arms are just touching the arms of the fibre without causing the middle section of it to distort in any way. Apply a small amount of quick set epoxy cement to the top of the arms so that the material wets both the fibre and the rough surface of the notch. Place additional cement so that the bent down ends of the fibre will be secured to outer sides of the carriage arms. Allow the cement to cure thoroughly; then release the tension clamp. The fibre will be pulled taut, and will resist gentle finger pressure. The remaining item of the carriage assembly is the mirror mounting bracket. Several materials were tried out and glass was chosen becuase it combines rigidity with the same resistence to chemical attack as the rest of the system. The brackets are made by cutting off strips 2 to 2.5 mms wide and 15 mms long from a pyrex microscope slide cover slip. Wheel type cutters don't work very well because of the pressure needed but, with a little practice, good strips can be cut with a diamond. To bend the strip to form a right angled bracket, place the strip on the top of a graphite bar 1/4 inch wide by 1/2 inch deep and 5 to 6 inches long, which is held in an asbestos jawed clamp. Place a second piece of graphite on the end of the strip to prevent it from overbalancing when it is set so that the midpoint of the strip is aligned with the edge of the graphite bar. Heat the bar from underneath, taking care to avoid flame spill from touching the strip; keep heating until the strip bends down to form a right angle.

Even a well mounted fibre will display a small amount of bow when force is applied perpendicular to its longitudinal axis until its limit of yield is reached. In

order to absorb this source of error which manifests itself during the beginning of the reading cycle, the fibre is deliberately prestressed in the following manner: The carriage is gently clamped so that the arms are tilted 10 to 15 degrees above the horizontal. Place a small drop of cement on the centre of the fibre and make sure that it flows around the diameter of the fibre, then balance the bracket like a rider on the fibre, moving carefully about to ensure that the bracket is wet by the cement across its width. The inside arm of the bracket should be at an angle to the base of the bracket and more or less horizontal. When the cement has cured, a glavanometer mirror with a focal length of 50 or 100 cms is fixed to the outer arm of the bracket using a minimal amount of vacuum wax. The mirror is positioned so that its center lays along the axis of the fibre. These procedures are illustrated in Figure 1.

Begin to assemble the gauge by first sealing the cone into the socket using vacuum wax. Soften the rod at a point 25 to 30 mms inside the tube using a micro torch, then bend it upwards so that at a point 10 to 12 mms inside the tube it is 5 mms above the centre line of the gauge. Now heat at the point 10 to 12 mms in and bend the rod to form a tip with a small rounded end at right angles to the rod and 5 mms long. Prepare two strips of vacuum wax about 25 mms long and 2 to 3 mms diameter. Clamp the gauge body horizontal then insert the carriage, positioning it so that the rod tip is very close to, but inside,the line where the fibre lies. It will be necessary to manipulate the bracket in order to allow it to pass under the tip. The ideal relationship between the bracket and the tip of the rod comes about when the distance between the point of contact of the tip on the bracket arm and the fibre is 1 mm judged visually from the side, and when the pretension pressure of the rod causes the bracket to rotate clockwise to bring the focal plane of the mirror into the bottom portion of the flanged end. It will be necessary to heat and rebend the rod in order to achieve the desired result. At this point the two pieces of wax are

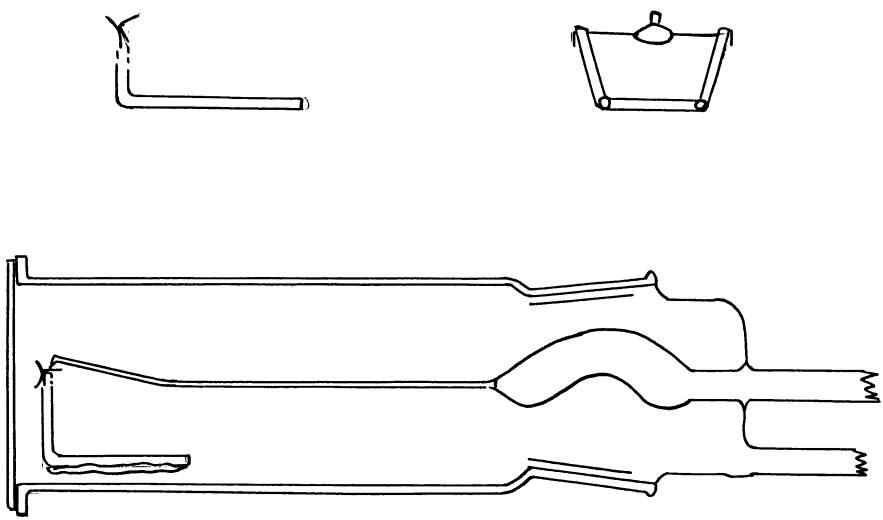

Fig 2

laid along the outside edges of the carriage. Gently heat the outer walls of the tube adjacent to the wax in order to melt it sufficiently to flow and adhere to the inner surface of the tube and the carriage rods. Before the wax hardens check for any malalignment, which may have been introduced by moving the carriage during this procedure. Finally, when everything has set solid, the body of the gauge is clamped vertically with the flange surface horizontal. Spread a minimal quantity of cement onto flange and carefully set a flat pyrex window onto the flange (the window can be square). When the cemnt has set the gauge is ready for use. See Firure 2.

The instrument is quite sensitive enough for most gas phase studies at low pressure, and is simple to make. Furthermore, in these days of low funding, it is inexpensive to make.

BASIC ANNEALING
JUST COMMON SENSE ANNEALING

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It has often been said that you can go to annealing temperature as fast as the glass can take it. **But . . .**

What does this mean? Try it . . . and it also means how fast does your oven go up to annealing temperature?

For all around general annealing of the borosilicate glasses, or the ones used in the lab with a coefficient of expansion per C of 32 X 10^{-7}, it's not complicated. But in many shapes, condensers, flasks, etc. where the oven goes to a temperature in forty-five minutes to one hour, it proved to be most adequate because of penetration.

It doesn't matter if you are doing one piece or gradually increase the oven up to full capacity as it will take longer for the same oven to reach annealing temperature because of the work load or volume of glass to be heated. You cannot go to annealing temperature too slowly.

Now we have other factors to consider if your oven is constructed of certain fire brick that absorbes a lot of heat. You have to put in a lot more power or BTU's to overcome the heat drawn in to the fire brick and the bricks continue to draw a lot of heat out of the oven, after reaching annealing temperature. Thus, you have to hold at annealing temperature much longer because of this situation. This problem also decreases the efficiency of the oven since it takes much longer to get the heat out of the oven and the fact that you can most likely make only one annealing cycle per day.

Because most ovens that have a lot of metal inside them use blowers to get uniform heat, the blower could be inadequate. One may think that they have a terrific blower because at room temperature they have a very high velocity, but one must remember that at 60°C, a small airplane will use one half the runway it uses at 90°F. Can you imagine how little the air moves when you are up to 1000°F., Unless you have a **very, very** large blower, or in the case of the airplane, a powerful engine. But I must state it is an almost must to use a blower in all vertical ovens and vertical ovens are usually used only for long straight columns.

I have found in a bell type oven one could go to 585°C and have the oven shut right off. But, then again, it depends on the type of firebrick used. It also depends on what you put in. I would not suggest using this method on complicated glassware.

A simple crude test is to take a length of borosilicate tubing, 8 - 10 mm dia. by 4' long. Hold at both ends placed a few inches off the bottom. By going to 585°C and shutting the oven right off, the tubing will sag a little. In fact, this is a crude test for annealing. You can generally anneal flasks and most uncomplicated apparatus by this method. Also, the internal heigth is a factor, since heat rises.

In repair work, one would fill the oven, run up to the annealing temperature of 585°C, (this will burn out the contaminents), then set the controls on a holding temperature of 350°C to 450°C.

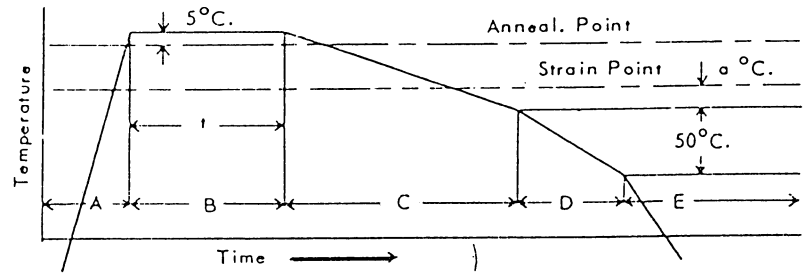
In the case of flasks with star cracks, take them out one at a time. Using a big bushy flame, heat up the area, then use your sharp flame and seal up the star crack. (always have some 4 mm glass rod handy in case the crack opens up.) Then flame anneal with a big bushy flame and return to the oven and, at your leisure, take out piece after piece and repair them.

Table 4 - 1

SCHEDULES (IDEAL) FOR COMMERCIAL ANNEALING - ORDINARY WARE

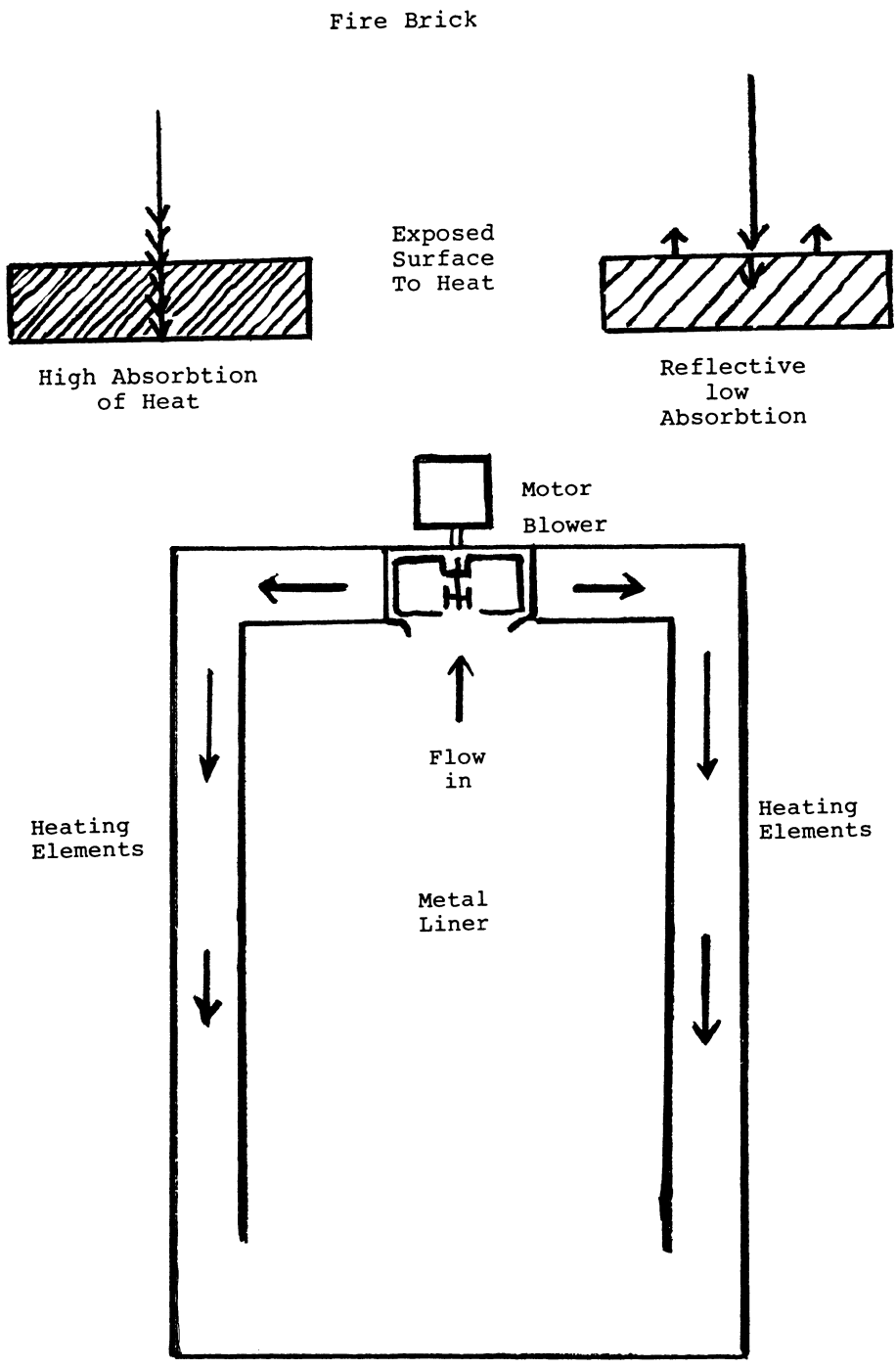
Annealing Periods -

A - Heating to 5°C. above Annealing Point.
B - Hold Temperature for time - t.
C - Initial Cooling to a °C. below Strain Pt.
D - Cooling - Next 50°C.
E - Final Cooling.



Expansion Coeff. of Glass per °C.	Thick. of Glass		Cooling on One Side					Cooling on Two Sides						
			A	B	C		D	E	A	B	C		D	E
	In.	MM.	Heat Rate °C/Min.	Time t - Min.	Temp. a - °C.	Cool Rate °C/Min.	Cool Rate °C/Min.	Cool Rate °C/Min.	Heat Rate °C/Min.	Time t - Mir.	Temp. a - °C.	Cool Rate °C/Min.	Cool Rate °C/Min	Cool Rate °C/Min.
33×10^{-7}	1/8	3.2	130	5	5	12	24	130	400	5	5	39	78	400
	1/4	6.3	30	15	10	3	6	30	130	15	10	12	24	130
	1/2	12.7	8	30	20	0.8	1.6	8	30	30	20	3	6	30
50×10^{-7}	1/8	3.2	85	5	5	8	16	85	260	5	5	26	52	260
	1/4	6.3	21	15	10	2	4	21	85	15	10	8	16	85
	1/2	12.7	5	30	20	0.5	1.0	5	21	30	20	2	4	21
90×10^{-7}	1/8	3.2	50	5	5	4	8	50	140	5	5	14	28	140
	1/4	6.3	11	15	10	1	2	11	50	15	10	4	8	50
	1/2	12.7	3	30	20	0.3	0.6	3	11	30	20	1	2	11

Only under extremely abnormal conditions would you lose a piece of work when the oven is holding at 350℃ to 450℃. When you have finished your repairs, run the oven to 585℃ and shut off with no ill effects.



THERMOCOUPLES

If you use a thermocouple with:

1) No protection (open wires), you have a very sensitive response and your electric relay is clicking on and off with the slightest variation of heat.
2) A thin protection shield or pipe shield seem most desirable.
3) The ceramic shield is an almost must in the quartz annealing oven.

Because of the variations that are caused by various thermocouples, it is extremely important to know your oven.

THERMOCOUPLE

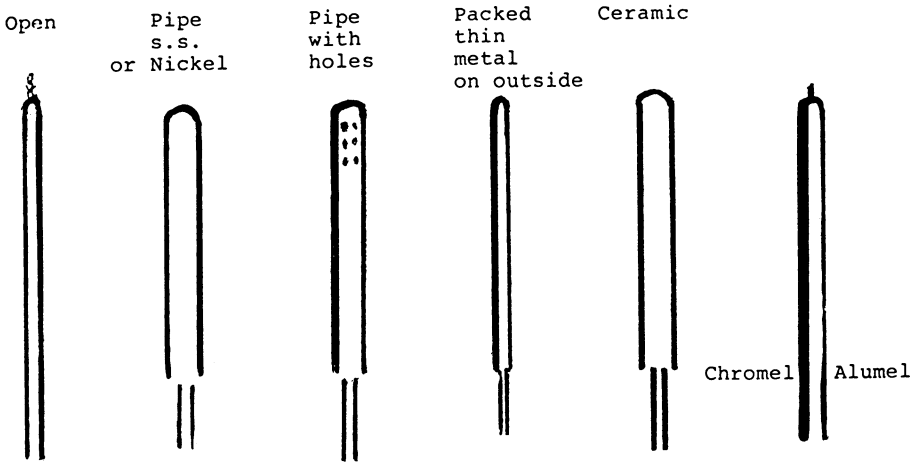
You will find as your thermocouple gets older or is used a lot, the two dissimilar metals become more similar and you are going to higher temperatures to anneal: this usually means thermocouple trouble.

REPLACING THE THERMOCOUPLE

It is extremely important that you use the same type as the old thermocouple.

Iron constantan, chromel-alumel, platinum, rhodium, (with a high percentage of platinum). These are common thermocouples used on annealing ovens.

Be sure to connect thermocouple wires as old thermocouple was connected. (red is always negative).



BASIC OVENS
FIVE BASIC TYPES

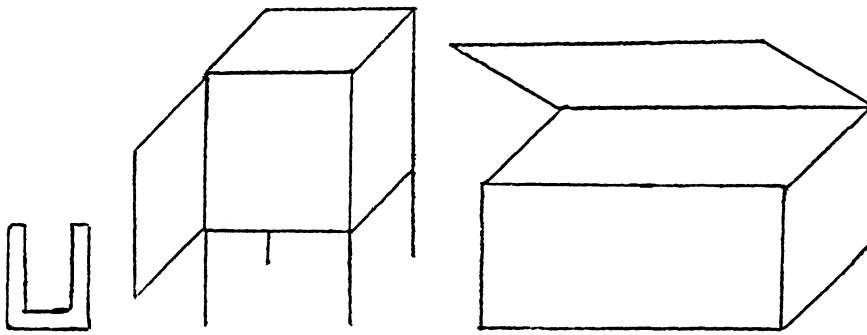
1) POT 2) DOOR 3) COFFIN 4) BELL 5) VERTICAL

With the most recent type a combination, a furnace that will anneal the softer glasses right through to quartz. This has been brought about by the following:

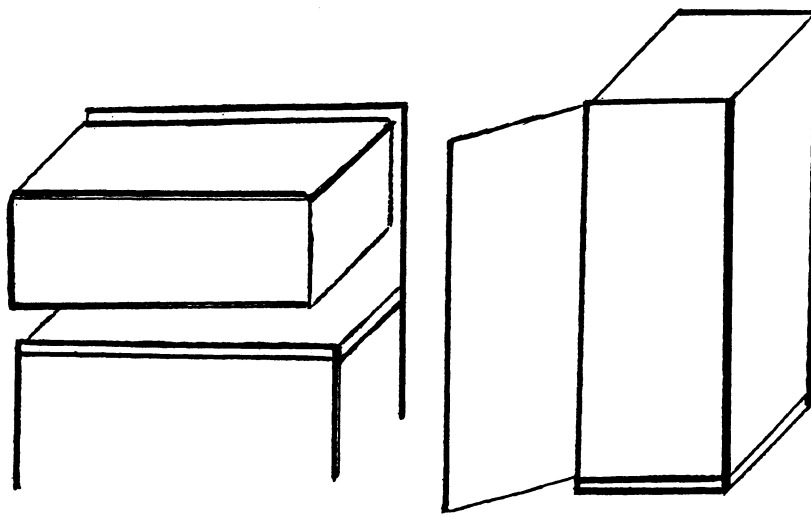
1) Good efficient ceramic fiber blanket insulation. There are many types: Some were not very good when they first came out. The blanket was flaky or could be pulled apart easily. The newer types are much more durable, but still vary in purity.
2) **Microprocessors** – Put in a program for what you want, whether it be soft glass or borosilicate glasses. Through quartz, some microprocessors are capable of many steps for ramping up, soaking, and ramping down.

3) Example: If the oven goes to temperature too fast. instrumentation and or microprocessors are available to reduce your rate.
4) Careful consideration should be used in selecting control systems.
5) Also, do not try to run this type of oven without first reading **ALL** the instructions.

Five Basic Types of Ovens

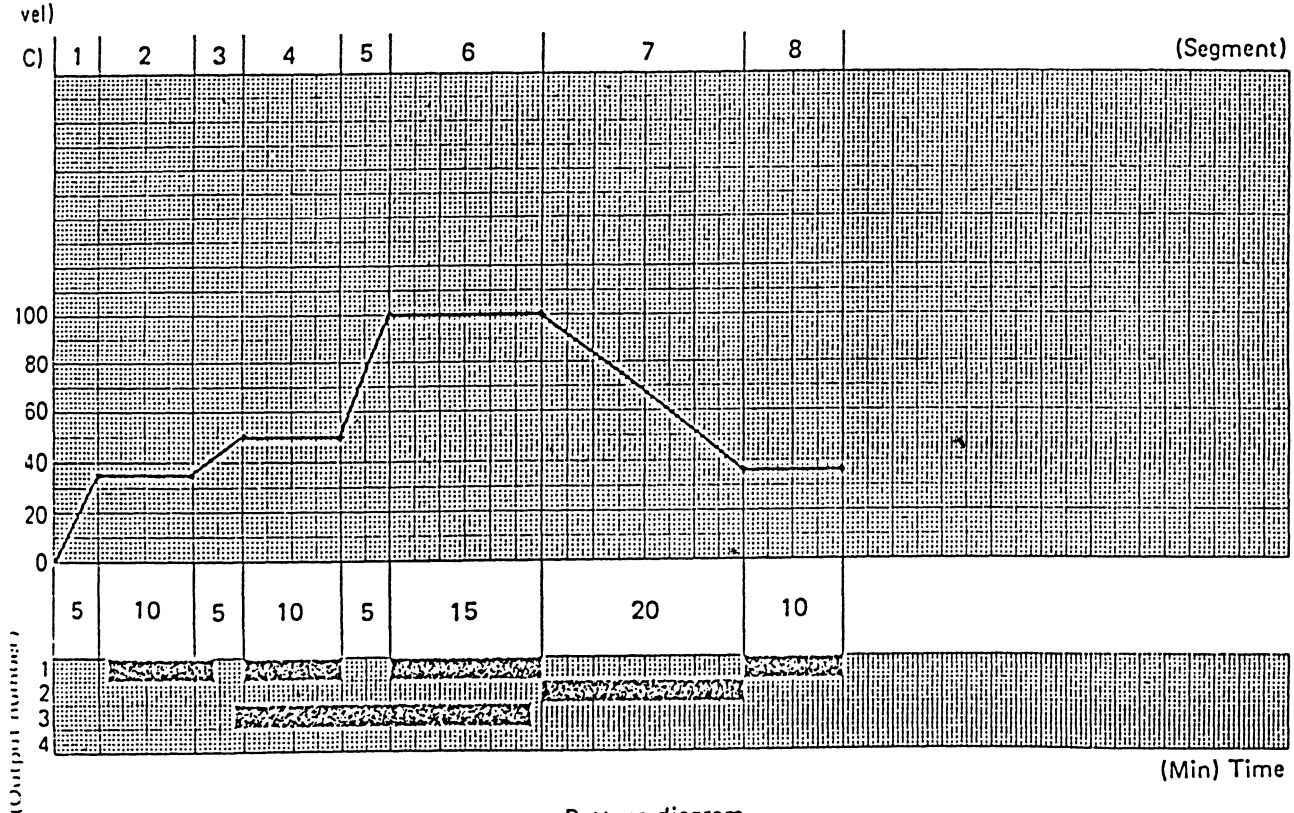


Pot Door Coffin



Bell Vertical

What I wanted in an oven lined with firebrick here is what I felt most desirable when blowing glass and making all types of apparatus, diffusion pumps, repair work, etc. – an oven that would do the following.



Pattern diagram
Time signal diagram creation format

1) Be able to have a fast recovery when adding pieces.
2) Low heat loss and operate efficiently.
3) Could comfortably add pieces or take out glassware when opening and closing.
4) Automatic shut-off when glass was annealed.
5) Be able to have controls that when set would hold a given temperature such as when doing repair work or baking out.
6) Could seal on and off comfortably vacuum tubes, dewars, etc. – parts to be evacuated.
7) Lined with proper insulating materials that reflected the heat and not absorb it.
8) Go to 585℃ in forty-five minutes to one hour with a light load.
9) Could do at least two annealings in a day of uncomplicated glassware.

SUMMARY

Through trial and error you will find you can do many things such as repair and anneal two - three batches a day.

Or in the case of quartz – go to 1140℃ and, when the furnace gets down to 900℃, unload and reload for another annealing.

I will admit you will need an aluminized jacket and gloves, plus a large fan. I do not recommend doing this constantly because of the damage to the oven it can cause, but we have found less damage with the use of blanket insulation.

What this is all about is experience and using your oven and getting to know it.

It is one of your most valuable tools.

MODIFIED DEVICE FOR RAPID MIXING OF LIQUIDS

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Department of Chemistry
University of Wisconsin – Milwaukee

This paper deals primarily with the construction of a divice used to homogeneously mix two solutions very quickly. The solutions completely mix in a matter of microseconds. The mixed stream of liquid can then be immediately analyzed. The chemical reactions can be observed immediately at the tip and at various points along the stream. This allows the researcher to chart the chemical reactions over time.

Figure 1 shows the basic design. It consists of one glass tube held concentrically inside a larger tube. This is accomplished by means of an Ace-Threds* joint. The solutions to be mixed are delivered through teflon ® tubing connectors. Mixing takes place as the solutions are forced around a 100 micron diameter platinum ball secured in the tip. This ball is held in place between the inner and outer tubes.

Fabrication of the apparatus was straight forward to the point of requiring a hole in the tip of each tube 90 - 100 microns in diameter. Having no access to an ultrasonic drilling machine, I saw the formation of the two holes as the major stumbling block of this job. After several failed attempts at drawing tubing down to such a precise opening, I determined a form or mandrel of some sort would be needed.

A fine piece of uncoated copper wire was chosen because it would seal to the glass but not make a vacuum seal. It is also easily attacked by common acids. After locating a piece of suitable copper, I drew down the tube to the approximate desired shape and opening. The piece was allowed to cool and the wire was placed in the constriction. See figure 2. Using a lathe, gentle but localized heat was applied until the glass sealed onto the wire. As soon as the glass sealed onto the wire the heat was removed and the piece was again allowed to cool. The tip was cut on the cut off saw and 100 ml reservoir was sealed to the top.

The copper wire was removed by drawing concentrated nitric acid through the tube with the aid of vacuum. Figure 3 shows the set up used to accomplish this. Because the copper doesn't make a vacuum tight seal, the acid will, over time, be drawn down through the tube into the bottle. You can speed the process up by using aqua regia (1 part HNO_3 - 3 parts HCL) if needed. Aqua regia produces objectionable quantities of chlorine gas. Extra caution should be exercised if this mixture is to be used.

Figure 4 illustrates the method used to secure the 0.05 mm diameter platinum wire in place. This was accomplished by scoring and snapping the tube approximately 2 inches from the tip and allowing the wire to hang over the now open ends and resealing the tube. The 100 micron platinum ball was made by heating the end of the wire with a sharp oxidizing flame.

Assembly and operation of the device are illustrated in figure 5. The two solutions, No. 1 coming in from the top into the larger tube and No. 2 from the side into the smaller tube, are then mixed. As stated earlier, the mixing takes place as the solutions are pushed through the tubes and simultaneously flow around the platinum ball.

* Product of Ace Glass Incorporated.

CONCLUSION

The intent of this presentaion was to show a simple method to make reasonably precise holes without the use of sophisticated ultra sonic drilling equipment. With a little practice this method has proven itself very useful for this as well as a number of other projects.

REFERENCE

Mixing Liquids in Microseconds by Peter Regenfuss, Robert Clegg, Mack Fulwyler, Francisco Barrantes and Thoms Jovin – Review of Scientific Instruments 56 (2), February 85.

® "Teflon" – registered trademark for TFE-fluorocarbon resin E. I. du Pont de Nemours & Co.

ACKNOWLEGEMENT

I would like to thank Dr. James Kincaid of Marquette University for his help on this project.

Figure 1

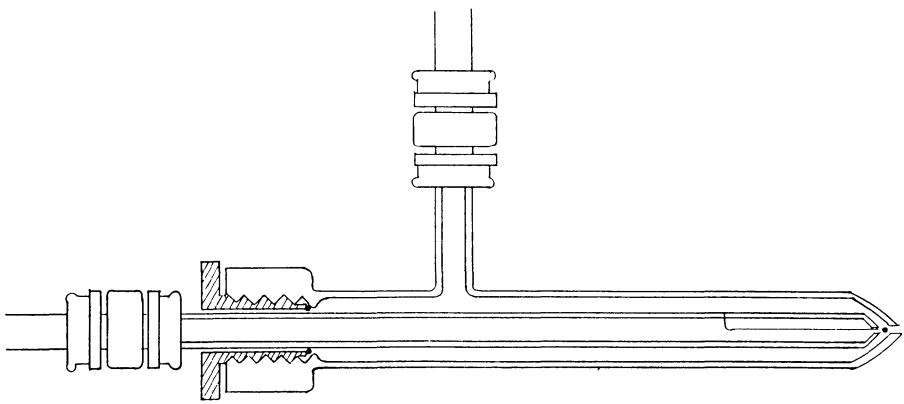


Figure 2

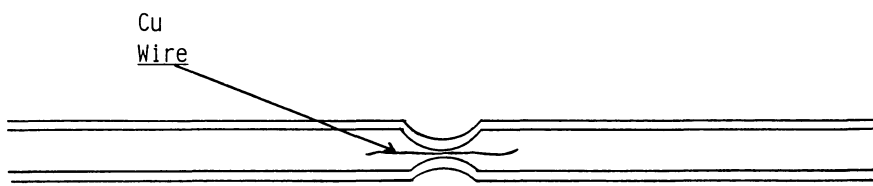
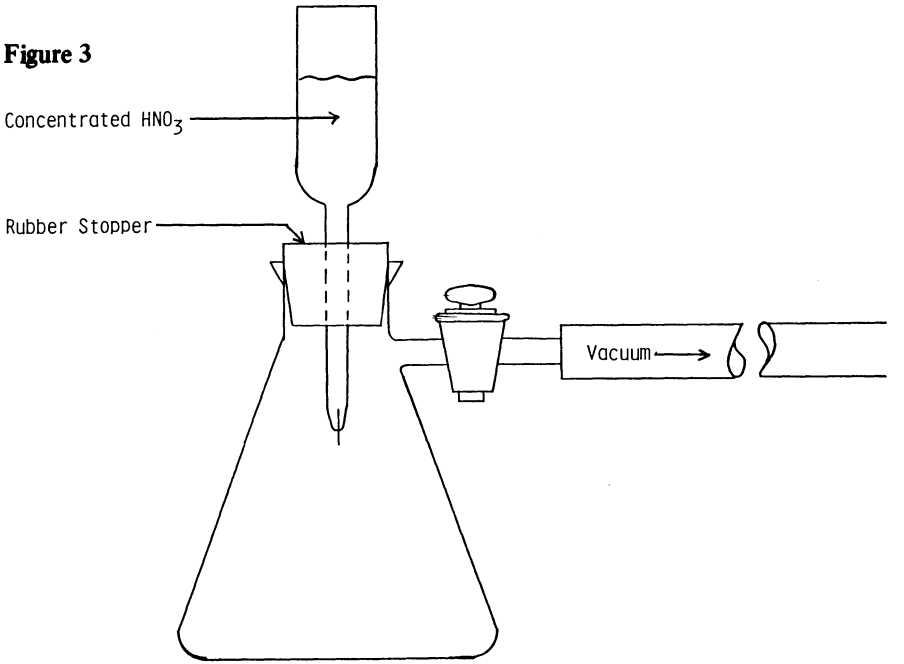


Figure 3



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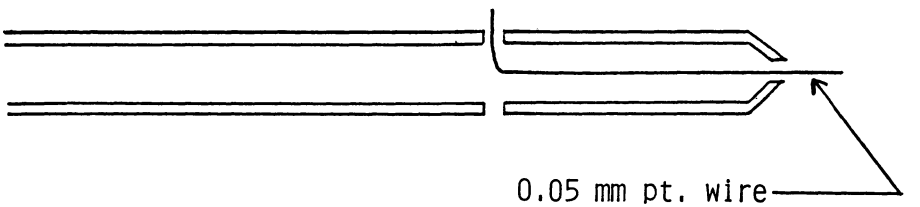

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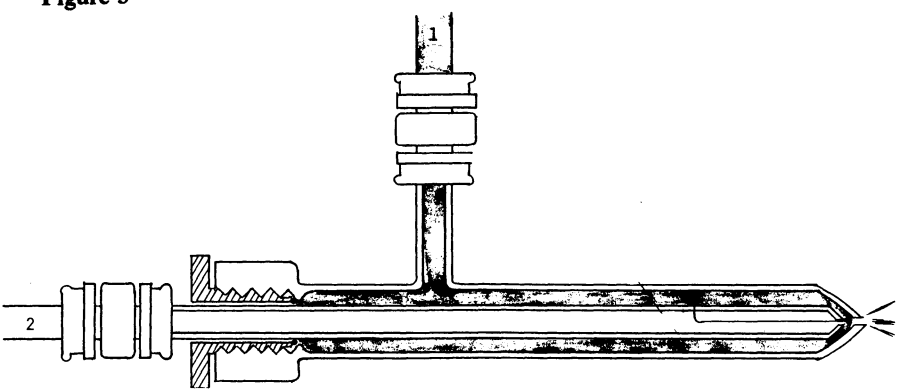

FLOW AND STOPPED-FLOW APPARATUS, SOME USEFUL TECHNIQUES

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In the investigations of reaction mechanisms of solutions by the observation of high speed reactions, two novel pieces of apparatus were developed. Both satisfy the requirements of requiring small volumes of reagent and providing extremely rapid mixing of the solutions. The first piece to be described will be an improved tangential jet mixer which can accommodate both flow and stopped-flow techniques. The second is a microdroplet mixer for use in flow methods. The design and use of these pieces of apparatus will be described briefly as they have both been adequately described in the literature (1, 2). What we will attempt to do is to elaborate on the glassblowing techniques involved, showing the fixtures and equipment utilized in the construction of the glass components. It is hoped that this will provide the glassblower with several useful techniques that can be utilized in the construction of similar devices and the development and construction of other types of apparatus.

THE TANGENTIAL JET MIXER

Description of the Mixer. Figure 1 shows the basic mixer and its connectors, and Figure 2 shows the mixer connected to a spectrophotometric observation cell. Both pieces can be connected to a computer-operated syringe drive system by using teflon tubing and Alltech liquid chromatography fittings (3) as shown in Figure 1.

Construction Details. To construct a single-stage mixer as in Figure 2, plug the end of a piece of 2 x 7 mm quartz or pyrex tubing and insert it into the grinding jig, shown in Figure 3. The grinding jig consists of a stainless steel block with a hole just large enough to accommodate the glass tubing. Intersecting this hole, a 1-cm wide grinding slot is milled into the block to a depth which extends 1 mm into the hole. Indexing marks are scribed at right angles on the end of the block. The tube is inserted into the hole and is locked in place with a plastic screw, A. Place the indexing ring on the tube next to the block and lock it in place with a plastic screw, B. The flat is ground with a 1/8" diameter cylindrical diamond tool mounted in a hand-held motor tool (Dremel). After the flat is ground, loosen the plastic screw A, and rotate the tube 90°using the marks on the indexing ring as a guide. The screw is then retightened and the next flat is ground. After all four flats are ground, the tube is removed from the jig, and the flats are polished using 400- and 600-mesh emery paper. Flatness can be maintained by wrapping the emery paper tightly around 1/4" square metal bar and using this to carefully hand lap the flats. To drill the tangent holes in the flats, we have utilized a metal lathe with a S.S. White airbrasive unit (4) mounted on the tool compound as shown in Figure 4 A-B. The chuck of the lathe is indexed at right angles and a pointer is positioned next to the chuck so that the chuck can be rotated in exactly 90° steps. The holder for the airbrasive unit has a rotating indexed head so that it can be positioned at any desired angle. The lathe compound allows movement in all lateral directions. Care must be taken to position the tube so that the airbrasive tool drills a hole from the exact center of the flat and still gives the hole the desired tangential angle as shown in Figures 1 and 2. Placing a piece of pipe-cleaner in the bore of the glass tube will keep the bore from being abraded when the airbrasive stream cuts into the bore.

If a secondary mixer (split bore) is required as shown in Figure 1, a plug is sealed in the middle of a glass tube and the channels for the split bore are drilled with the

airbrasive unit using the lathe set-up shown in Figure 4. After the channels are drilled, the entry holes are flame-sealed, and you proceed to the first step of the construction of a single-stage mixer.

The final step of construction is the grinding of the groove at the outlet of the mixer which attaches to the L.C. connector as shown in Figure 1. This is accomplished by mounting the mixer in the chuck of the metal lathe and grinding the groove with a diamond tool mounted in a Dremel as shown in Figure 5. Note the buret with the syringe needle which is used to direct a stream of water onto the diamond tool while grinding.

The mixer is connected to the required systems as described in the literature (1). Several of these mixers have been constructed and used in our laboratory with a variety of observation cells for several years, with excellent results.

In an effort to utilize these mixers at higher pressures, we are presently constructing them from 2 x 10 mm tubing rather than 2 x 7 mm tubing as shown in Figures 1 and 2. It is hoped that the greater cross sectional area in the tangential section will give it the additional strength required to tighten the inlet tubes to the tension required for higher pressures. Results of this change will be reported at a later date.

THE MICRODROPLET MIXER

Description of the Droplet Generator. The microdroplet generator is shown in Figure 6. It consists of 2 glass capillary tubes mounted through piezoelectric Bimorphs(oscillating crystals) and connected to a pressurized flow system. Droplets are generated by forcing solution under pressure through the oscillating capillaries. The 2 streams of droplets are impinged upon each other, as shown in Figure 7, to form a stream of coalesced droplets which may then be observed by laser Raman spectroscopy. A detailed description of this operation is available in the literature (2).

Construction Details of the Glass Capillaries. Matched pairs of capillary tubes with constricted tips that ranged in size from 0.001" to 0.005" as shown in Figure 8, were constructed as follows. Several capillary tubes were hand drawn from 12 mm standard wall tubing to a diameter of 0.028" to 0.030". The resulting wall thickness of these tubes ranged from 0.0025" to 0.003" as measured with a microscope using a Filar eye piece. One-foot lengths of these tubes were then cut in half and the resulting ends which have identical O.D. and I.D. are then constricted by inserting them into an electrically heated coil as shown in Figure 9. The heater is constructed of 18 gauge Ni chrome wire wound into a cone shape, with the top diameter being approximately 1/2". The electrical current is adjusted so that the coil attains a bright yellow-red heat.

Care must be taken when cutting the capillary tube in half that the resulting ends are square, without chips or nicks. Otherwise the constricted orifices will not be round and concentric. Insert one of the matched ends into the heating coil to a depth of approximately 5 mm and clamp it so that it is positioned as close to the center of the coil as possible. The depth can be gauged by inserting a thin metal strip between the coils and clamping the tube so that it is touching the metal strip. Be sure to remove the metal strip before starting the heating cycle. Turn on the heater and heat the capillary for about 1 1/2 to 2 minutes, timing the heat cycle with a stopwatch. Remove the capillary and measure the resulting constriction with a Filar eye piece. If the capillary tube is filled with distilled water when making measurements of the inside walls, false images and reflections will be reduced, allowing a true reading. The water can be blown out of the tube, leaving the bore clean and uncontaminated. If the constriction needs to be smaller, this is

accomplished by using shorter heat cycles until the desired size is attained. Be sure to time and record these additional cycles. The second capillary is then inserted into the heater and positioned to the same depth as the first. Use the same heating schedule as was used on the first capillary. The resulting constriction should be very close to that of the first capillary. If it is not, short heat cycles on the larger of the pair will bring it to the size of the smaller one. With s small amount of practice, you will find it is possible to match the tips to within 0.0001" or less. We have matched tips to within 0.0002" with this method. As experience is gained in positioning the tube in the heater and cutting the tube so that the ends are chip free, you will find that many of the pairs will be within 0.0001" upon completion of the matched heating cycles, and little or no additional heating adjustments will be required.

The matched capillary tips are then cut to length and connected to syringe needles with teflon heat shrink tubing, backed up with a layer of epoxy cement. They are then ready for connection to the droplet generator and its flow system.

This method has been used to make precision constrictions on tube ends as large as 4 mm diameter with I.D.'s of less than 0.010". The taper of the constriction can be controlled by the depth that the tube is inserted into the heater. The greater the depth, the longer the taper. Shorter depths result in sharper tapers.

If a square or beveled end is required on the constricted tube, it can be generated by sanding with fine emery paper. Air must be blown through the tip while sanding to prevent its being plugged with abrasive compound.

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2. Simpson S. F., Kincaid, J. R., Holler, F. J.; *Anal. Chem.* 1983, Vol. 55, No. 8, 1420-2.
3. Available from Alltech Assoc. Inc., Deerfield, IL.
4. Available from S. S. White Industrial Products, Piscataway, NJ.

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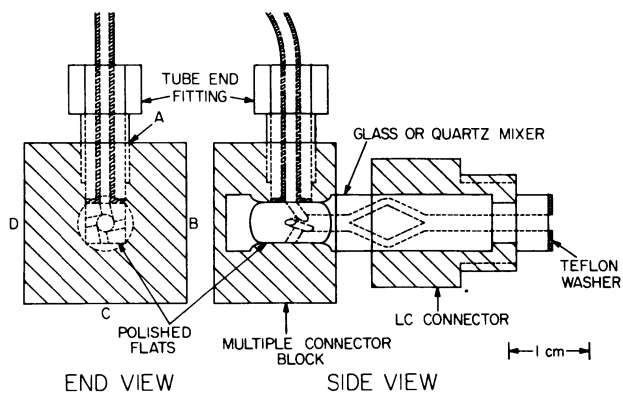
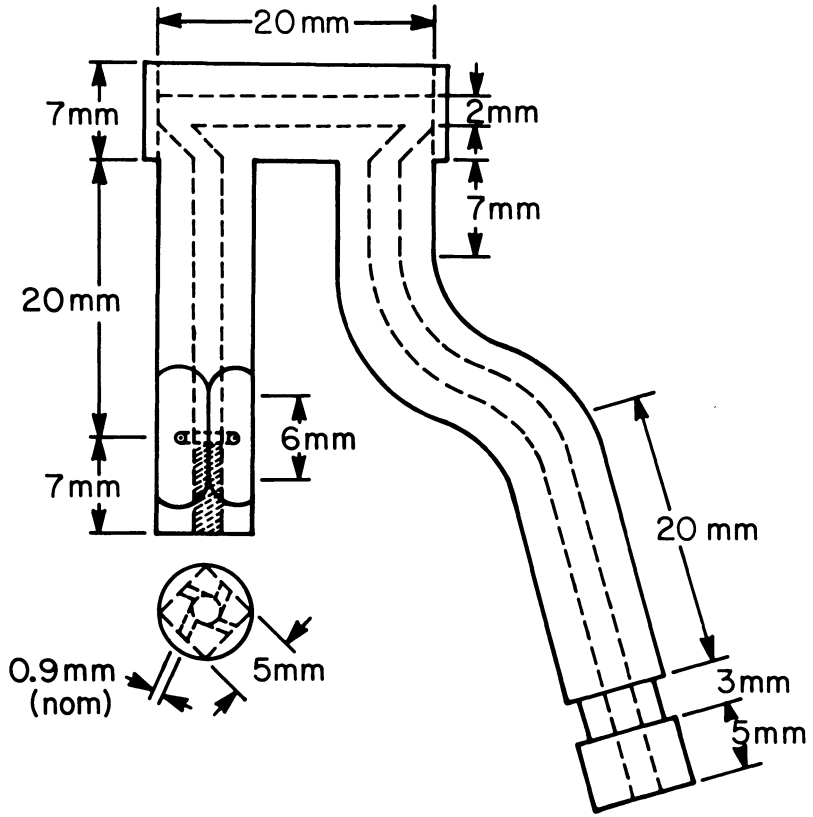
Figure 1





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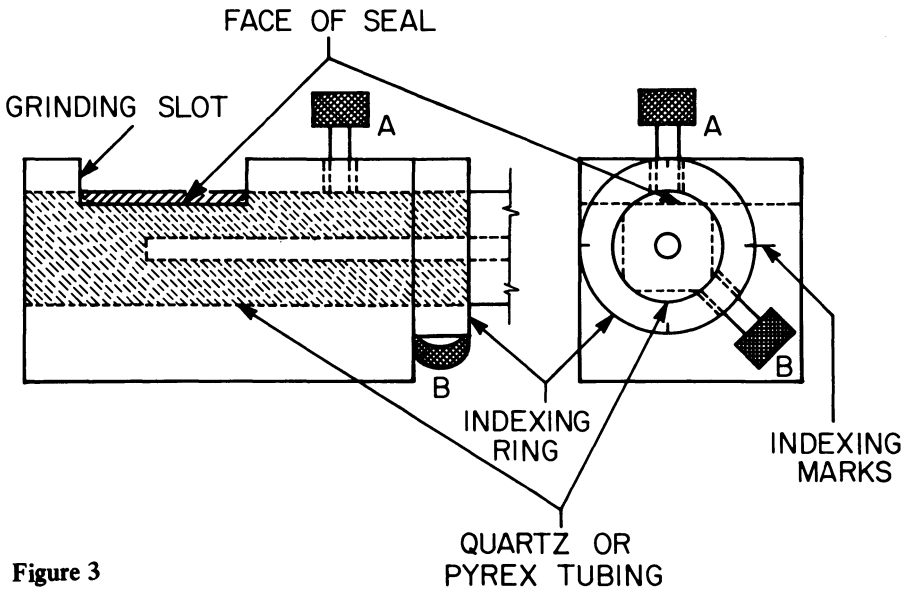

Figure 3

Figure 4

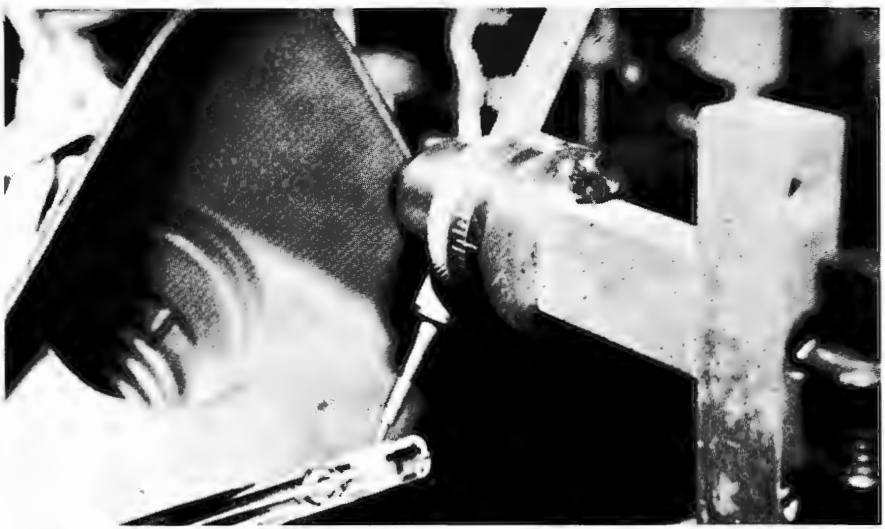

Figure 5

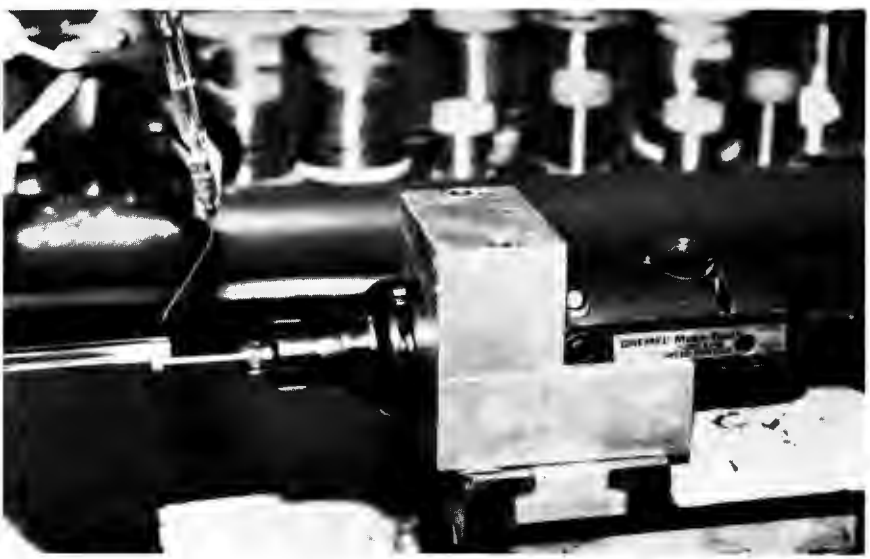

Figure 6

Figure 8

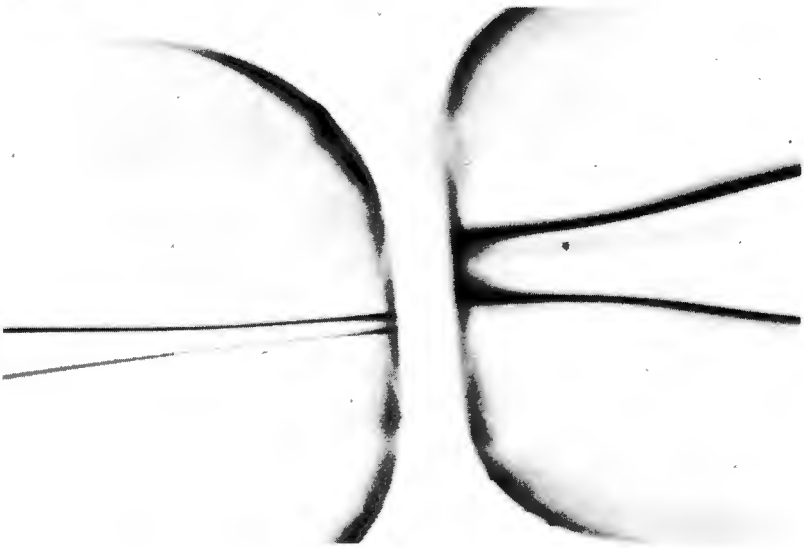

Figure 9

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