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Papers

Construction of an Organic Vapor Phase Deposition Reactor: A Proof of Concept for TFT & OLED Processing

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ABSTRACT

Molecular organic compounds are employed as active materials in a variety of applications, including organic light emitting diodes (OLEDs), photovoltaic cells, and thin film transistors. Typically, these thin (~100nm) film devices are grown by thermal evaporation in high vacuum, permitting the high degree of purity and structural control needed for reliable and efficient operation. However, control of film thickness uniformity and dopant concentrations over large areas, which are needed for manufactured products, can be difficult when using vacuum thermal evaporation (VTE).

An alternative to conventional VTE is a technique called organic vapor phase deposition (OVPD). It proceeds by thermal evaporation of the molecular organic compounds into streams of inert gas, which dilute and transport them to a cooled substrate where condensation occurs. An all glass reactor provided proof of this concept.

INTRODUCTION

At the start of this new millennium, the Nobel Prize in Chemistry was given to three chemists for their work on conductive organic polymers. The citation from their 2000 award read in part; "We have been taught that plastics, unlike metals, do not conduct electricity. In fact, plastic is used as insulation around the copper wires in ordinary electric cables. Yet this year's Nobel Laureates in Chemistry are being rewarded for their revolutionary discovery that plastic can, after certain modifications, be made electrically conductive...."

The three recipients were: Dr. Alex J Heeger, Dr. Alan G. MacDiarmid and Dr. Hideki Shirakawa. Their work opened a broad field of organic materials to electronics and electronic devices. Indeed, when we think of electronics and the sources of their innovations, we usually think of physicists such as Thompson and the electron or cathode ray tube, Bardeen, Schockley & Brattian. The devices and patents, which were later developed due to these innovators, came from companies like Bell Labs, Westinghouse, RCA, and IBM.

Figure 1. *A small TFT on flexible plastic.*[1]

Now chemists working at companies such as Eastman Kodak, 3M, DuPont and PPG are busily working on organic electronic

[1]Photo courtesy of Sarnoff Research Center in Princeton, NJ.

materials, which use molecules similar to some found in biology to perform electronic functions now done by silicon and other minerals and metals. These materials can be applied in economical non-conventional ways to create displays, transistors, modulators, memory storage devices, and even used as power sources on glass and most notably **flexible plastic substrates**.

THE ORGANIC ADVANTAGE

The traditional electronic device is often laid on a silicon chip that is processed and coated with rare earth elements such as gallium and arsenide. There are literally less than a hundred or so known compounds that can be used (conversely, organics provide over a million different compounds) in a high temperature process near 1,000°C in extremely controlled environments. In addition, they typically require vacuums in the range of $1x10^{-7}$ or greater. Present day TFT (thin film transistors) and LCDs (liquid crystal displays) are fabricated in large vacuum reactors using a process known as a VTE (vacuum thermal evaporation).

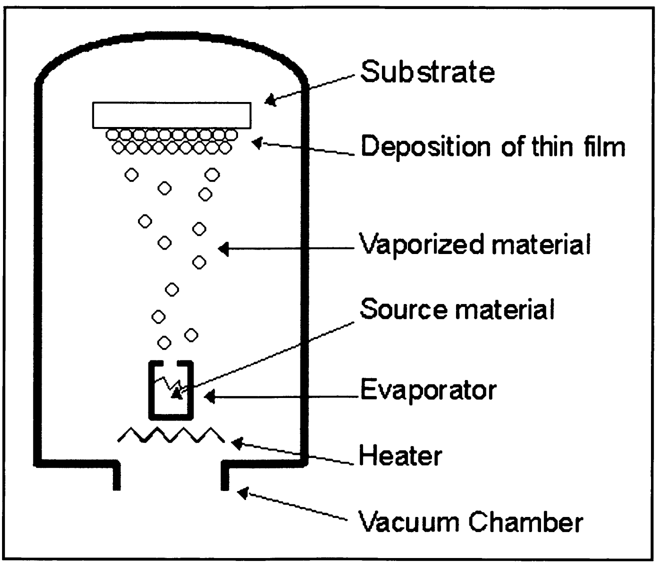

Figure 2. *A schematic of a typical VTE used in TFT and LCD panels.*

In conventional VTE, the reactor chamber achieves a vacuum of $1X10^{-7}$. The source material is heated to <800°C and the vapor it generates evolves within the reactor. At the top of the reactor, substrates are held upside down against a cooling block above the vapor. The source material condenses onto the substrate. However, several issues become problematic. For instance, the coating thickness requires very precise temperature controls. The difference of just 1 degree Celsius can affect the coating by a factor of x10. In addition, as little as 3% of the source material ever makes it onto a product, since most of the vapor coats the housing of the reactor. As a result, production runs are frequently shut down because the reactors start to build-up thick coatings that start to precipitate onto the substrates. These shutdowns are costly in time and affect product yields.

DESIGN CONSIDERATIONS FOR OVPD

The goal for the Princeton OCM (Optoelectronic Component Materials) Group was to design a reactor for TFT that could take advantage of the low temperature requirements of organic materials and to utilize a carrier gas such as N_2 to flow the evaporating gas towards the substrate. Making a glass reactor as a "proof of concept" model offered the fastest and most economical way to prove OVPD capabilities.

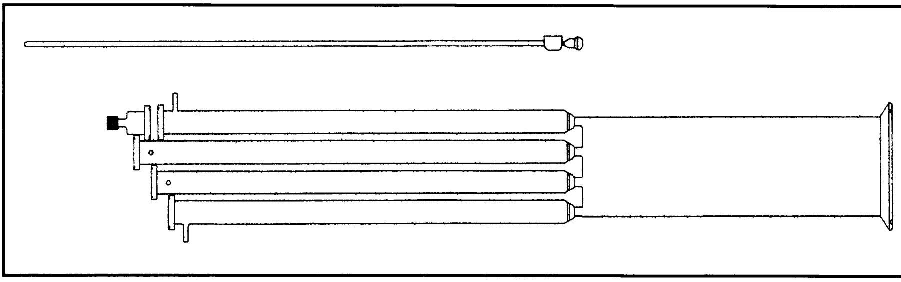

Figure 3. *The initial design for the OVPD.*

The design goals for the first OVPD were fairly simple. A large entry was required for loading the substrate holder. Normal borosilicate glass was acceptable because temperatures would not exceed 300°C. The center region of the reactor would have to fit into a clamshell tube furnace. Each open end on both sides of the reactor would be outside the furnace. Thus viton O-rings were acceptable as gaskets for the O-ring joints; this was a big advantage since it would be a low pressure system that would flow inert gas over non-volatile material at a pressure no greater than $1X10^{-3}$. Source boats had to be able to telescope in or out of the hot zone and still maintain an air free environment. This was achieved by using #7 Chem Threads® as the slide fitting for the source boat pull rods. At the time, the biggest design obstacle appeared to be that the users would want to be able to isolate and deliver four different source materials. For these purposes, the original design in figure 3 worked quite well. However, the construction and later repair of the reactor was problematic. This involved sealing the four 1.5" o.d. source tubes nearly one meter long to the seat inlets on the 5" reactor tube. Since the seat inlets were modified 18/9 sockets and the supporting material where it was sealed to the 5" material was a mere 1/2" tube, the strain was constant at this juncture. Moreover, as source boats would slide up and down, scratches would occur. This put into play, the well known **flaw plus tension** equation that no doubt led to some of the later breakages. I would also learn that the group would occasionally run the temperature past 450°C at regular intervals to ash clean the organics. In more than one case, this led to extreme sagging and the eventual replacement of the whole reactor. It seemed that every case was a very difficult repair job, even to replace just one broken flange or a component of the reactor.

THE SECOND GENERATION OVPD

Someone once noted that "Perfection is the obstacle to achievement…." The existing OVPD was proving to be functional despite the occasional disruptions from breakages. Indeed, because the OVPD was in constant use, the disruption of a repair would cause a backlog, which made it imperative to get the instrument fixed as soon as possible. In this mode, re-designs and improvements on an existing instrument are often shifted to the rear in priorities. Nevertheless, with the direction of graduate student Max Schtein, a new design was developed for the OVPD. The overall objectives were to make the reactor functions more modular and replaceable and to install new features such as an exhaust port and an observation port to insert an inferometer to monitor the thickness of the coating on the substrate.

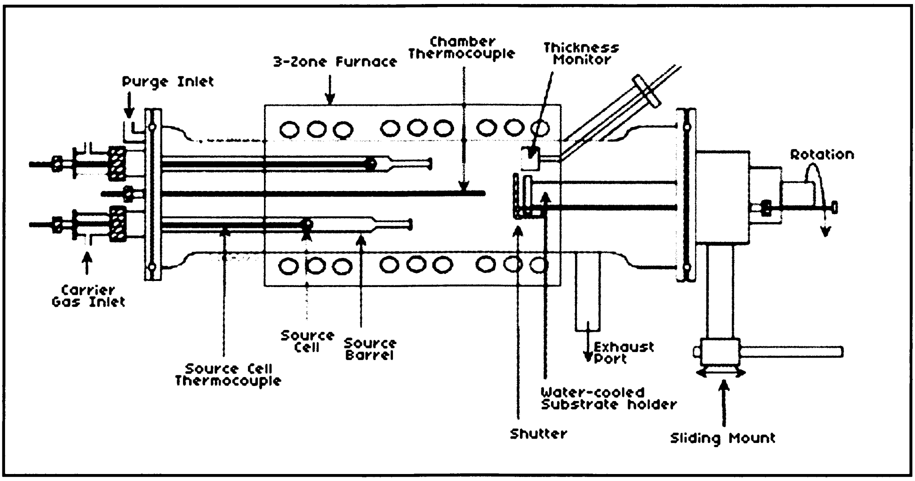

Figure 4. *The 2nd generation OVPD.*

Ultimately, this new design achieved these objectives. In Figure 4, we see that the reactor has a uni-body that acts as an envelope. Each source barrel is mechanically fixtured inside the reactor. A spacer made of refractory insulation block helps support the barrels inside the reactor. The source cells holding the organic can be slid into or out of the hot zone. Each source barrel has an inert gas inlet to allow gas flow to move the phase vapor through the nozzle outlet and onto the substrate holder.

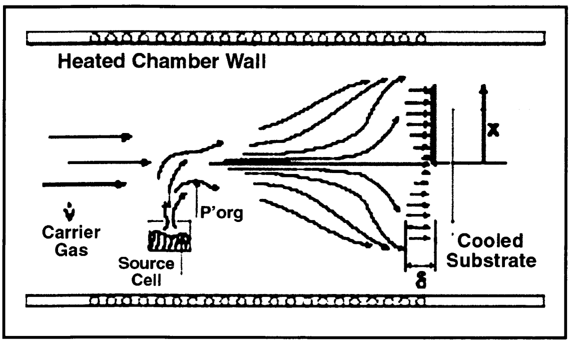

Figure 5. *The flow dynamics of the OVPD.*

With this design, each part became replaceable. Indeed, redundant parts could be made and if an occasional source tube cracked, it could be easily replaced. In the meantime, the broken part could be fixed and rotated into back-up parts. The reactor's performance was also enhanced with the addition of two outlets in the antechamber area. The inlet on the bottom acts as an exhaust port. If the reactor needs cleaning, the organics are burned off of the walls and the expensive compound is reclaimed by vacuum in the exhaust port. The inlet on top allows a light probe to measure the thickness of the coating as it occurs. Amazingly the molecules applied to the substrate emit extremely bright light when a current is applied. Yet the coating is approximately 1/1000th the thickness of a human hair. When the light display is not active, the screen is 90% transparent to light.

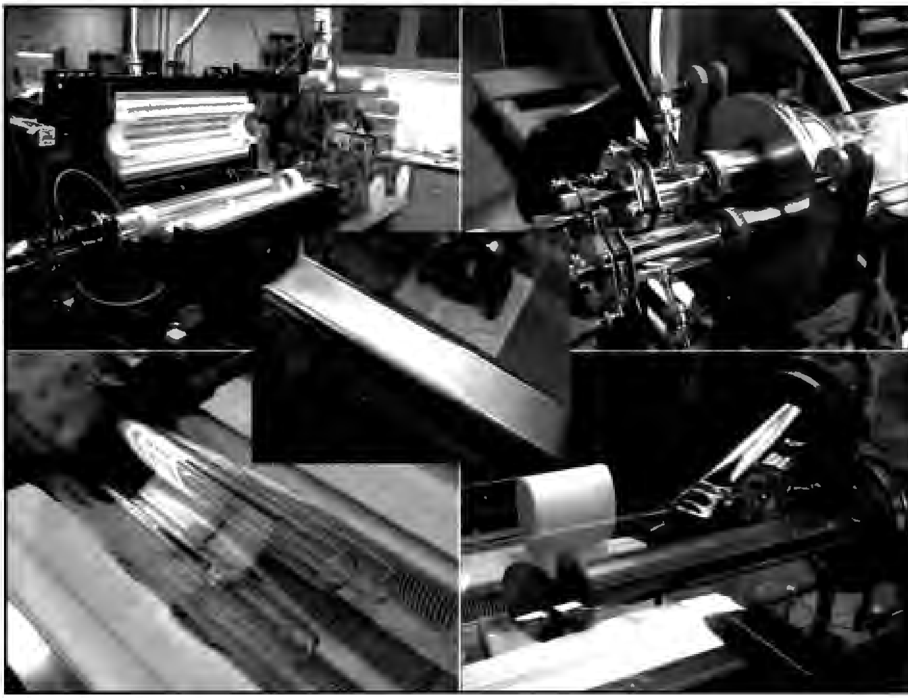

Figure 6. *A photo collage of the OVPD. The center panel shows an active OLED (organic light emitting diode) film. Courtesy of Max Shtein.*

Work on the OVPD started in 1994. In 2002, a fundamental patent was filed with the U.S. Patent Office. The license for this patent was sold by Princeton University to Universal Display Corp. (UDC) in Ewing, NJ. In 2001, a fully functional OVPD Pilot Plant was built for UDC under a development and licensing agreement with AIXTRON AG. The price tag for this equipment exceeds $2 million dollars. In May of 2003, UDC and AIXTRON released the following press release:

> **RiTdisplay selects AIXTRON's OVPD technology for OLED Display Production.** Aachen / Hsinchu, Taiwan R.O.C., May 7, 2003- AIXTRON is delighted to announce the sale and joint testing of AIXTRON's 2 generation Organic Vapour Phase Deposition (OVPD) equipment with RiTdisplay Corporation, one of the world's leading OLED manufacturers, situated in Hsinchu, Taiwan. RiTdisplay has successfully developed OLED main and sub-displays for mobile phones, instrument front panel displays, E-books, games, and personal digital assistants (PDA).. AIXTRON's development of this highly sophisticated OVPD tool, suitable for 2 generation mother glass size (~370 x 470mm) OVPD displays, began in 2002 as part of the OVPD joint development program with the Universal Display Corporation (UDC). The delivery of the OVPD system to RiTdisplay is planned before the end of this year.

It is impressive to note that the two-generation OVPD has the capabilities to produce nearly 300k OLED screens per month. The price tag for the Production Plant is nearly $10 million per unit. According to the USDC (United States Display Consortium), a similar process plant used to make LCD screens typically costs $100 million. Needless to say, the "proof of concept" bench model bears little resemblance to the present day OVPD.

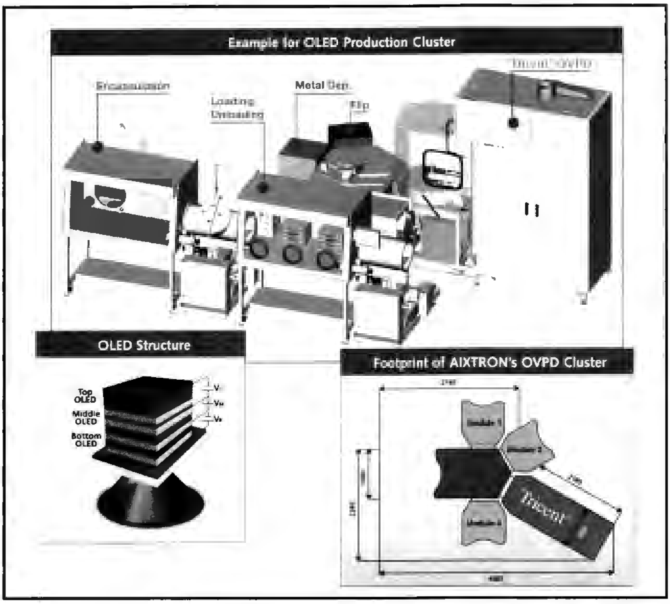

Figure 7. *Today's OVPD bears little resemblance to the model built as a "proof of concept."*

CONCLUSION

Organic electronics is still a very new field. Products using carbon-based materials to mimic what metals and rare earth elements presently do are just coming to market. OVPD promises to be an important tool in processing OLEDs which offer far brighter screens and use nearly 75% less power; these are just now emerging onto the market place in small cell phones. According to DisplaySearch, a leading consultant in the flat panel display industry, the market for OLEDs in 2003 will be $300 million worldwide. By 2007, that number is expected to reach nearly $2 billion annually. The salient point for scientific glassblowing at the research level is that the function of a scientific glassblower is to aid research on many levels. We are responsible for upkeeping traditional laboratories by repairing and fabricating old and new glassware. We also aid in the design of new glassware for emerging fields. In a recent article, Professor Forrest of the Electrical Engineering Deptment at Princeton remarked that "The role of a good scientific glassblower is to make a new instrument. With a new instrument, new measurements can be taken and that is when science is at work."[2]

I would like to acknowledge the support and help of individuals and companies associated with this project:

Professor Stephen Forrest, James S. McDonnell Distinguished Professor of Electrical Engineering at Princeton University

Max Shtein, Graduate student in The Dept. of Electrical Engineering at Princeton University

Universal Display Corp. in Ewing, NJ

AIXTRON AG, Aachen, Germany

Dr. Thomas Spiro, Dept. of Chemistry, Princeton University

The Dept. of Optoelectronic Component and Materials Laboratories at Princeton University for the use of their schematics

[2] M. Shtein, H. F. Gossenberger, J. B. Benziger, S.R. Forrest, "Organic Vapor Phase Deposition and its Applications to Electronic Devices," *J. Appl. Phys.* 89 (2001): 1470.

Glassblowing Holders: From Production to Research and Development

by
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ABSTRACT

This paper will discuss five separate holders that I have used over the past years. As a glassblower who has worked both in the production and research fields at various times in my career, I have come to the conclusion that the holders commonly used by production shops can come in very handy to the custom glass shop. Types of holders to be discussed include grind tool for extractor thimbles/side ring seals, condenser tool, filter funnel frit holder, resizing i.d. mold tool, and resizing o.d. mold tube.

In the past few years, I have had the opportunity to work in a large-scale production shop. There, it was necessary to develop a selection of holders that would enable me to produce a high volume of glassware with consistent results while maintaining a high level of quality. In this paper, I will be discussing a few of the holders that I developed while working in a production environment. I will conclude by discussing some holders that I have since used in my new shop at Procter and Gamble, a custom type shop.

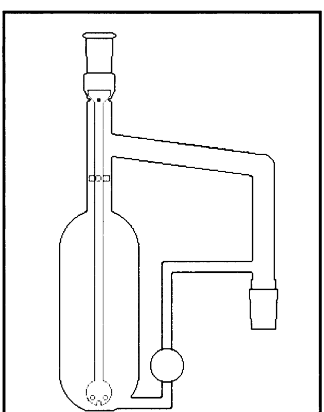

Figure 1

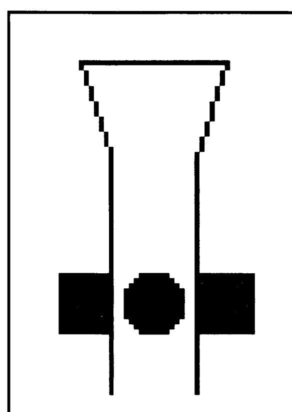

Figure 2

The first holder that I will be discussing I call a "Grind tool for extractor thimbles." Here you see a standard liquid/liquid extractor (Figure 1). To the right of the body, you see the extractor thimble. This next slide shows a close-up of the top of the thimble (Figure 2). The portions that are highlighted are the support arms. It is these parts that I will be grinding.

In order to assemble the holder, it is first necessary to make some measurements of various tubing. The first tubing to be measured is the tubing that will be used in the thimble itself, in this case, 9mm medium wall (Figure 3). It is then necessary to find a tubing that has an inner dimension (i.d.) that is slightly larger than your first tube, in this case, 1/2" medium wall worked well as it has a ~9.5mm i.d. (Figure 4). The next

Figure 3

Figure 4

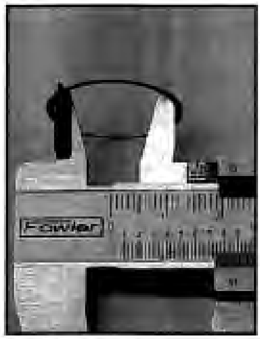

Figure 5

Figure 6

important measurement is the i.d. of the neck of the extractor itself; in this case, I have selected 25mm standard wall which has an i.d. of 22mm (Figure 5). The last tubing that will be needed is 22mm medium wall (Figure 6).

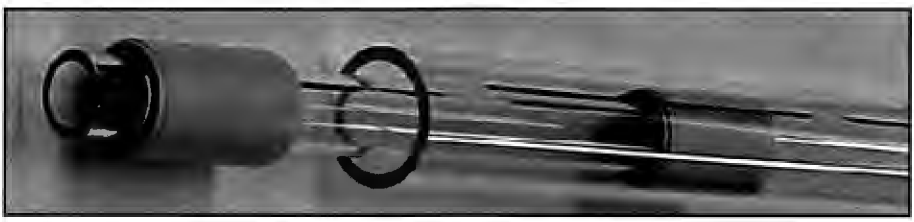

Figure 7

From here, the 1/2" medium wall tubing will be wrapped in masking tape until it fits snugly inside of the 22mm medium wall tubing (Figure 7). You now have essentially a tube with a 22mm o.d., and a 9.5mm i.d.

To use the tool, one first assembles the top portion of the extractor thimble with the flare and support arms. Note the support arms are roughly 1/2" long at this point (Figure 8). The unfinished thimble is then slid into the holder where it is taken to the belt grinder (Figure 9). I then grind the support arms until the holder touches the belt. The unit is then rotated until the entire scrap rod is ground away and there is nothing left sticking out past the holder. This process is repeated two more times. I had originally thought of having my machine shop make a stainless steel holder out of a piece of 7/8" rod with a 9.5mm hole down the center, but I decided against it due to the abuse that this holder suffers. As you can see in the picture (Figure 9), the 22mm tube does get ground down, and with this set-up, I can easily and cheaply replace the outer tube as needed.

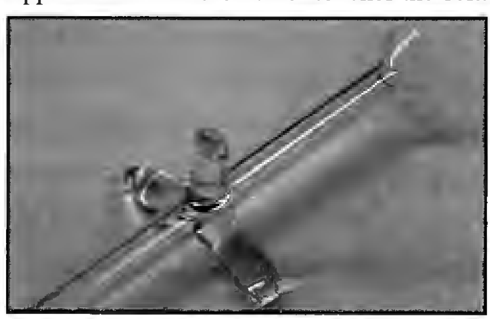

Figure 8

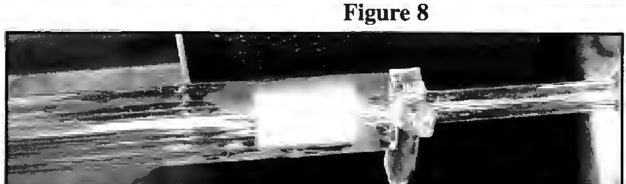

Figure 9

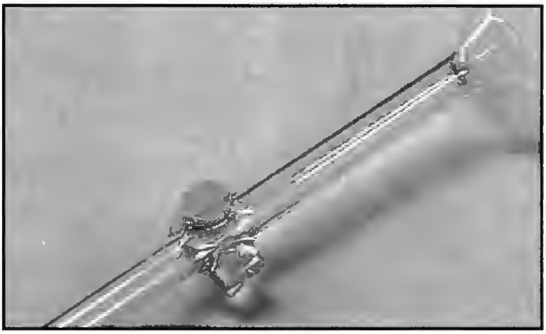

Figure 10 **Figure 11**

Here is a photograph of the final product before fire polishing (Figure 10). In Figure 11, you can see a close-up head on view of the thimble in the extractor neck. As you can see, the arms match up nearly perfectly with the i.d. of the extractor neck. This will prevent the thimble from swaying around in the extractor, minimizing breakage.

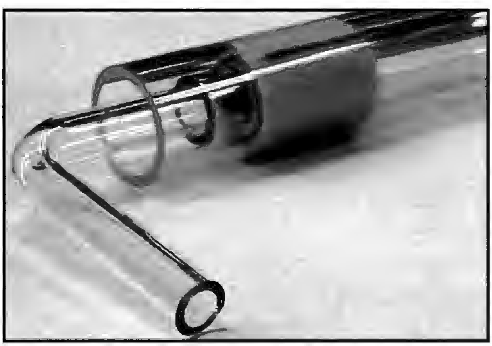

Figure 12 **Figure 13**

The above-mentioned holder also has a second purpose, and that is in prepping tubes for side ring seals. In order to do this, I have taken a piece of 9mm medium wall tubing and bent it to 90°. I then slid this bent tube into my holder (Figure 12). The excess is trimmed away, and it is then ground in a similar fashion as the extractor thimble. When finished (Figure 13), the 9mm tube will slide nicely into the 25mm tube. It is then ready for sealing. I prefer to use this method rather than simply cut the tube straight because, as you can see, using this grinding tool creates a curve on the ground surface of the bend (Figure 14)

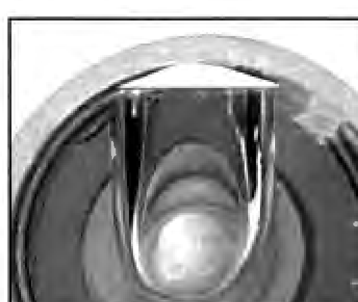

Figure 14 **Figure 15**

This eases the ring seal. If the 9mm tube is simply cut straight across (Figure 15), you are left with a gap at the top of the inner tube. I have highlighted this section. When the seal is made, the 25mm tube must be dropped down to the 9mm opening where, as in figure 14, the 25mm tube barely has to move before the two tubes are joined.

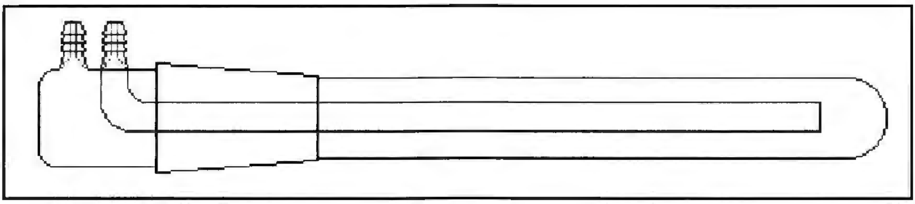

Figure 16

The next holder I want to discuss is a simple condenser holder. In this case, the holder will be used to create a cold finger condenser (Figure 16). When building a cold finger condenser, I have found it advantageous to locate the bend inside the condenser itself, as opposed to having the ring seal on the top of the condenser with the bend coming outside the body. By doing this, the condenser is much less likely to break but the functionality will be unaffected.

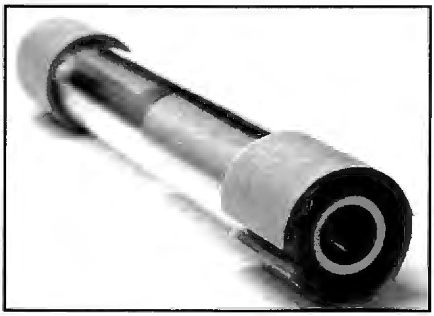

Figure 17

To assemble this condenser, I have stuck with the 9mm and 25mm tubing dimensions from the grind tool. To assemble the condenser holder (Figure 17), you again start with a piece of 1/2" medium wall as it has the proper i.d. for holding the 9mm tube. However this time, I have selected 3/4" heavy wall tubing as the outer tube for the holder. Since the 25mm standard wall has a 22mm i.d., 22mm medium wall tubing fits too tightly and in many cases will not fit at all. The 3/4" tubing is wrapped in masking tape until it fits tightly into the 25mm tube. This is done at the top and bottom of the holder. An air channel is then cut lengthwise into the masking tape, and this is repeated on the other end of the holder. Note that the two channels are 180° from each other. This prevents one side of the holder from

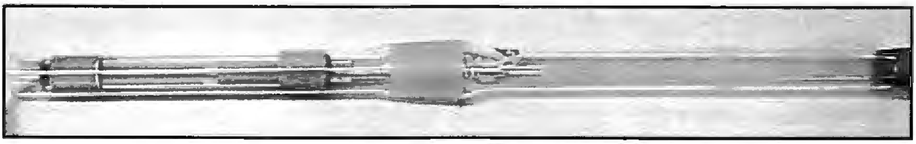

Figure 18

collapsing which helps to keep the holder running true. The holder is then loaded into the 25mm tube, and the pre-ground 9mm tube is slid into place. From here, the seal can be made (Figure 18). The condenser is not complete in this photograph (Figure 18), in order to show the clarity of the seal. Figure 19 shows a close-up of the 9mm ring seal.

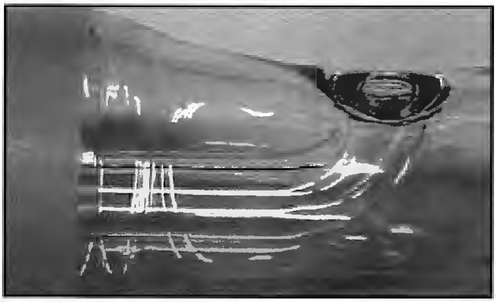

Figure 19

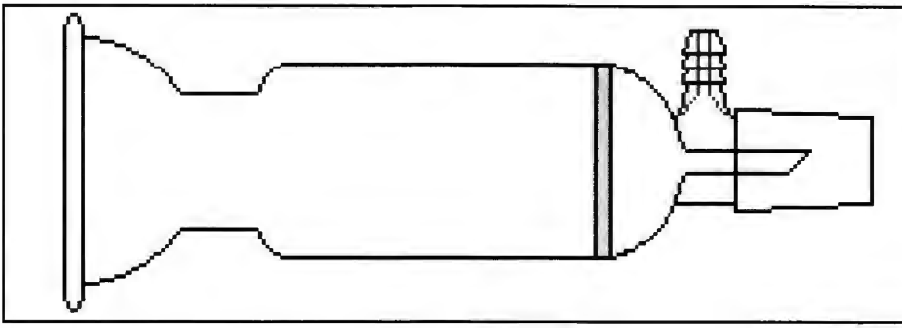

Figure 20

I will now discuss the holder I use to assemble filter funnels. The full technique for building the filter funnels can be seen in my poster. Figure 20 shows a typical filter funnel

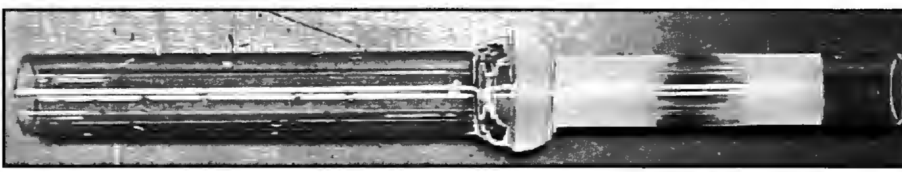

Figure 21

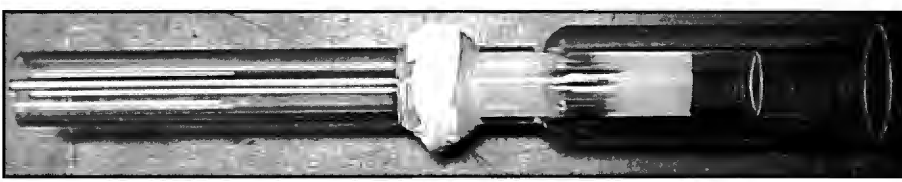

Figure 22

To assemble the holder (Figure 21), I first begin with an inner member of the needed joint, in this case a 50/30. A tube that fits inside the blank tubing of the outer member is selected, in this case a 30mm standard wall. It is then sealed to the bottom of the ball joint. As you can see in the illustration, this holder has been ground on the 30mm tube. This was necessary because the ball joint did not sit flush against the socket joint when they were mated with each other. I had gathered too much glass at the point of seal between the bottom of the ball joint and the top of the 30mm tube. It was easily corrected by simply grinding away the offending glass. The holder is then slid into the prepared socket joint (Figure 22). If your lathe is fitted with sun and planet chucks, a ball and socket clamp can be used to hold the holder into the socket joint. In this case, I was using a lathe that had scroll chucks, so masking tape was used.

Next the frit is slid into the body of the filter funnel, and an additional support tube is brought from the tailstock (Figure 23). Your frit will now be held straight for the seal.

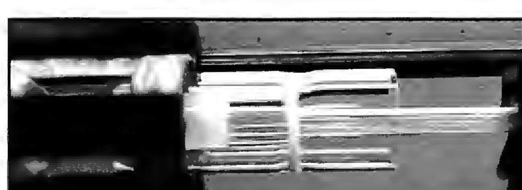

Figure 23

I will conclude this paper by quickly showing a few of the fixtures that I have used in my new glassblowing facility, in order to show a little contrast in holder style.

The first is a mold I had made by the machine shop. It is used to resize tubing to a specific i.d. (Figure 24). With the holder in the headstock,

Figure 24

I chucked a piece of larger than needed tubing into the tailstock (Figure 25).

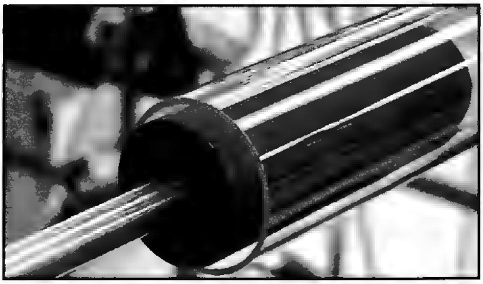

Figure 25

Figure 26

After preheating, I began melting the tubing and paddling it down onto the mold (Figure 26). I also assisted the shrinking by sucking in on my blow hose. I simply worked my way down the graphite until I reached the end (Figure 27). After the graphite begins to cool, it can be removed (Figure 28). It works well when short pieces of tubing are required.

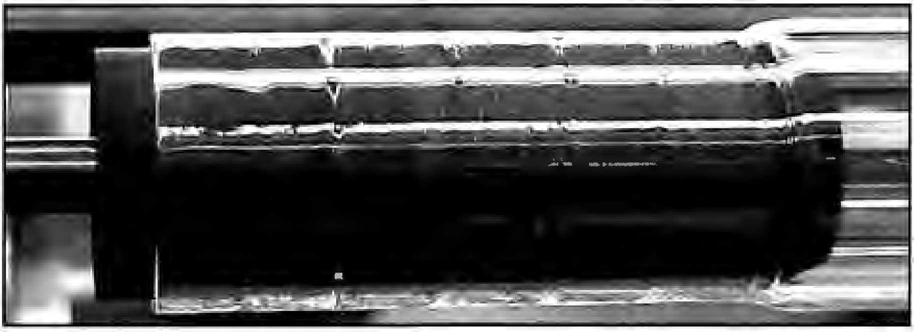

Figure 27

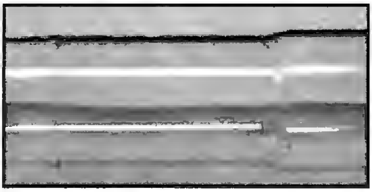

Figure 28

Figure 29

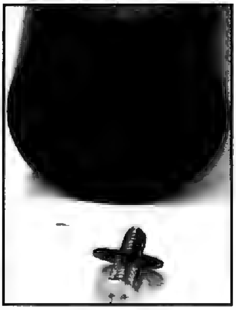

Figure 30

The last fixture I have to show is for resizing the o.d. of tubing. For this, I simply had the machine shop cut the top of a piece of graphite rod very smooth and tap the bottom (Figures 29 & 30). The rod is mounted upright in the lathe and is screwed in where the carriage fires would normally go. By setting the gap between the tubing and the graphite to a determined amount (in this case 12mm standard wall tubing with a 3mm gap), you can achieve consistent results (in this case 18mm thin wall) (Figure 31).

By slowly moving the carriage tray along the tubing, you can resize the tubing as long as you need (Figure 32). As with the mold, this works best on short lengths. Here you see a cut away of the tubing (Figure 33).

As we all know, every glass-blower has unique and individual methods of doing their work. It is up each and every one of us to share that information without reservation. Even your most trivial shortcut or technique could be invaluable to your colleagues, and we should never pre-judge our little nuances as trifle. Even the most obvious thing to me, might never occur to someone else. This is especially important as we younger glassblowers are coming into our own. The ASGS was founded on this principle, and it is our tireless effort in sharing that can keep this Society vibrant well into the 21st century.

Figure 31

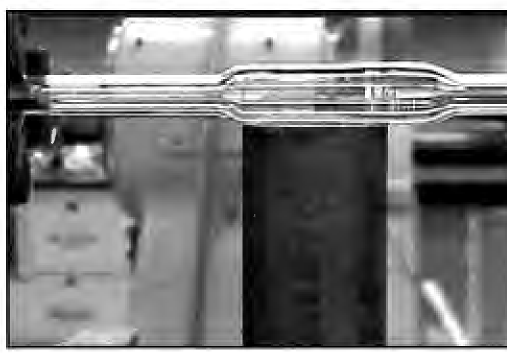

Figure 32

Figure 33

Liquid/Liquid Extraction

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ABSTRACT

Purifying a product is an important chemical process. Often the desired compound must be separated from the starting materials, impurities, side products and solvents used during the reaction. Liquid/Liquid extraction is one method of achieving this. The basic process of liquid/liquid extraction and the functioning of different styles of liquid/liquid extractors will be addressed. This will range from historical to present-day usage.

INTRODUCTION

Each year when filling out my annual evaluation form there is a space for goals. One of my most consistent goals has been to understand what scientific glassware is used for and how it functions. Without understanding what we as scientific glassblowers are fabricating, we cannot offer meaningful suggestions which might improve the design or help the researcher realize his goals more effectively. Besides, it is just more fun to know how things work.

As a member of the Education Committee of the ASGS, I feel that this understanding of "how things work" is a key to the enjoyment of our profession and our success in our chosen careers. Today I would like to share with you a particularly fascinating piece of glassware, the liquid/liquid continuous extractor: what it is used for and how it functions.

Before we get to liquid/liquid extractors let us backtrack to discuss some basic chemistry which will help us better understand liquid/liquid extraction.

BACKGROUND

A large portion of chemistry is involved with separations. After completing a reaction, you do not "all of a sudden" have the pure material which you desire. You usually must separate it from your starting materials, impurities, side products and any solvents you have used to run the reaction.

There are many different methods used to separate desirable compounds from other material. You are probably familiar with some of these such as,

- recrystallization (for purification of solids)
- distillation (for purification of liquids)
- sublimation
- lyophilization (freeze drying)
- column chromatography
- extraction

and many more.

Since liquid/liquid continuous extractors are the subject of this paper, let us take a closer look at extraction and define some terms which will be useful in understanding our apparatus.

Extraction – a method of isolating a desired compound from other material by contact of the mixture with a second phase. In our case, the phases are both liquid and the two liquids must be immiscible.

Immiscible – not capable of mixing together, substances such as oil and water are said to be immiscible.

Solvent – a substance in which another substance (solute) is dissolved.

Solute – a substance which is dissolved in another substance (solvent).

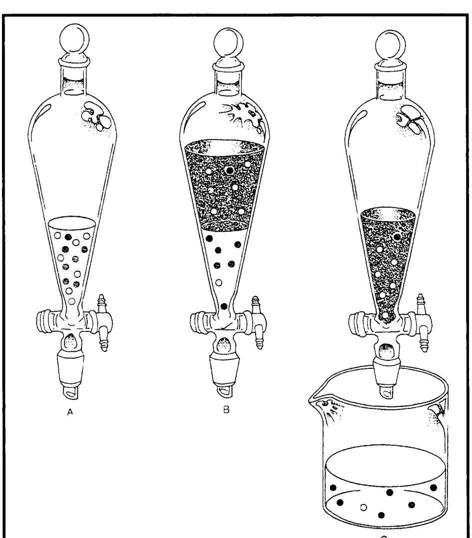
Aqueous – relating to water.

Partition or Distribution Coefficient - the ratio of the concentration of solute dissolved in one solvent to the concentration of solute dissolved in another solvent when the two immiscible solvents are mixed together and are at equilibrium. This is often denoted as:

$$K = \frac{\text{concentration of solute in solvent A}}{\text{concentration of solute in solvent B}}$$

Often liquid/liquid extraction is used to separate an organic compound from an aqueous solution using an organic solvent as the extracting media. Several criteria should be met.

- The extraction solvent should be immiscible with the aqueous solution.
- The material to be extracted should be soluble in the extraction solvent, preferably more soluble than in the aqueous solution.
- The extraction solvent should not react chemically with the compound or other material in the aqueous solution.
- After the extraction is complete, the extraction solvent should be easily separated from the desired compound.



The simplest form of liquid/liquid extraction is a batch extraction with a separatory funnel. In a highly simplistic example, suppose there were two different organic compounds (black and white) dissolved in water (an aqueous solution) and we desire to extract the white (Figure 1). Ideally we would select an immiscible organic solvent in which only the white compound was soluble.

Figure 1. *The extraction process. A. Solvent 1 contains a mixture of molecules (black and white). It is desired to separate the white molecules by extraction. A second solvent (shaded), which is immiscible with the first solvent, is added, and the two solvents are shaken together, B. After the separation of layers, most white molecules, but not all, have been extracted into the new solvent. C, With separation of the two layers, the black and white molecules have been partially separated.*[1]

[1]From *Introduction to Organic Laboratory Techniques,* 3rd edition by Pavia. ©1988. Reprinted with permission of Brooks/Cole, a division of Thomson Learning: www.thomsonrights.com.

Pour your aqueous solution into the separatory funnel, add your organic solvent, shake them together, allow them to separate, drain off the bottom layer and pour off the organic layer which has extracted a portion of your desired compound. Repeat as necessary. Evaporate off your organic solvent and claim your compound. If only it were so simple. The basic process is fairly elementary, but the devil is in the details. As you can see from the following overhead it is not a simple matter at all. This is merely one page of references out of three from a single article on extraction in 1958 (Figure 2).[2]

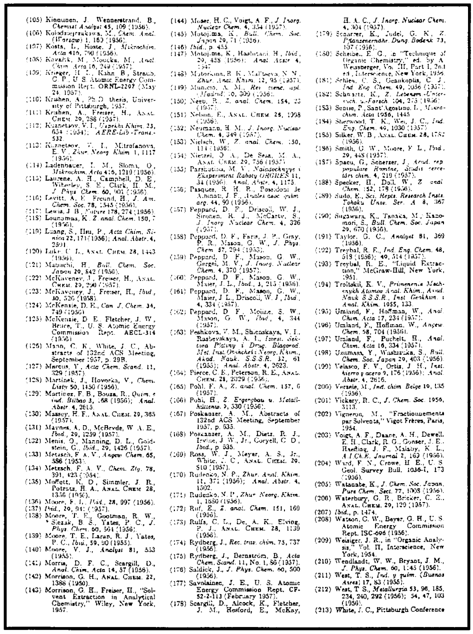


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Figure 2

A batch extraction using a separatory funnel works well with the proper selection of solvents and if the distribution coefficient is large. But even in an ideal case you will often hear that "Several small extractions are more efficient than one large extraction." If the distribution coefficient is small, then those "several" small extractions can multiply into "many" small extractions and you may end up spending all your time shaking a funnel and using large amounts of solvent which then have to be disposed of.

CONTINUOUS LIQUID/LIQUID EXTRACTORS

The continuous liquid/liquid extractor is the answer to these problems. It offers four main advantages.

- It uses relatively little solvent.
- It works for relatively poor distribution coefficients.
- It does not require the physical presence of an operator.
- It can be configured for "more" or "less" dense operation.

Examination of different styles of continuous liquid/liquid extractors will illustrate these advantages.

In this early (1937) extractor (Figure 3)[3] by S.

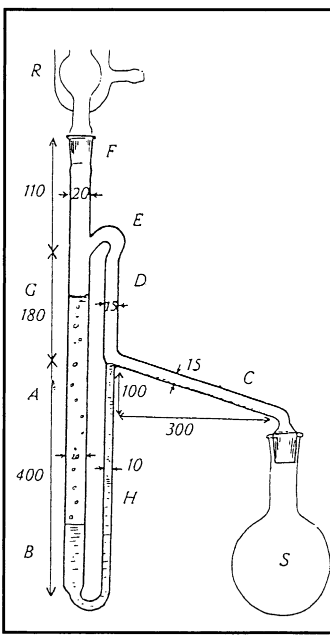


Figure 3

[2]Reprinted in part with permission from G. H. Morrison and H. Freiser, *Analytical Chemistry,* 30 (1958): 632-640. Copyright 1958 American Chemical Society.

[3]Reprinted in part with permission from S. Wehrli, "Ein Apparat zur Extraction von Lösungen mit Schweren Lösungsmitteln," *Helvetica Chimica Acta*, 20 (1937): 927-931. Copyright 1937 Schweizerische Chemische Gesellschaft, Basel. Verlag Helvetica Chimica Acta AG.

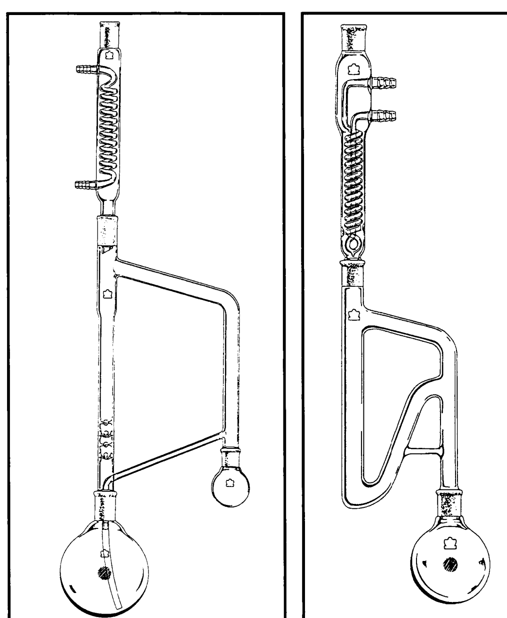
Wehrli, the extracting solvent is placed in flask *S*. The less dense material being extracted is located in portion *A* of the long column. When the round bottom flask is heated, the extracting solvent is continuously distilled, condenses in the Allihn condenser *R* and, because it is more dense, drips through area *A*, extracting the solute as it goes and collecting at the bottom of the apparatus. Eventually it will fill arm *H* and overflow back into the round bottom flask. From here it is redistilled; the fresh solvent repeats the cycle and the extracted solute becomes more and more concentrated in the round bottom flask. As you can see, this operation illustrates the four main advantages:

- Through constant redistillation and recycling of the extracting solvent the total amount of solvent is kept relatively small.
- Even if the distribution coefficient is small and the extraction is inefficient, because of the continuous nature of the process, the extractor overcomes these drawbacks. In effect it is many, many small extractions.
- It is easy to see that no operator is needed to shake the apparatus, drain off the appropriate layer and add fresh solvent as in the case of a batch distillation using a separatory funnel.
- This extractor, by design, is permanently configured for "more" dense operation. That is, the extracting solvent is more dense than the solvent containing the material being extracted. These are also sometimes referred to as "heavier than water" extractors because many extractions use an organic solvent to extract the solute from the aqueous solution. In this case, the extracting solvent, chloroform is "heavier" or "more dense" than the water.

The following figures are of the "heavier than water" style. These are currently available from laboratory glassware suppliers and function on the same basic principle seen in the S. Werhli extractor:

- There is a way to distill the extracting solvent.
- The extracting solvent is condensed and passes through the material to be extracted.
- The extracting solvent with dissolved solute returns to the distillation pot for reuse.

In this example (Figure 4), the flask containing the aqueous solution can be varied in size to accommodate different amounts of material. A small portion of the extracting solvent is placed in the larger flask. The upper assembly is put in place and the extractor is filled with the material to be extracted. The smaller flask is filled with the extracting solvent and the extraction process can begin.



Figures 4 and 5. Apparatus, Continuous Liquid/Liquid, Heavier than Water, Courtesy of Kimble/Kontes.

This smaller apparatus (Figure 5) is designed to hold 5-10ml of the material being extracted. Again, a small portion of the more dense extracting solvent is placed in the main body tube, the less dense solution to be extracted is added, the round bottom flask is filled with the extracting solvent, and the extraction process can proceed until completion.

If the material to be extracted is in a solution which is more dense than the extracting solution, obviously a problem arises. The less dense solution will just sit on top of the more dense solution and no extraction will occur. In this case, an extractor of "less" dense or "lighter than water" style will be necessary.

The "lighter than water" extractor works by conducting the less dense extracting solution to the bottom of the extracting chamber. This is most often achieved through the use of a solvent guide tube which gives the extracting solvent enough hydrostatic head so that it can force its way out the bottom of the guide tube. From there, being less dense, it bubbles up through the material to be extracted and returns to the distillation flask for reuse.

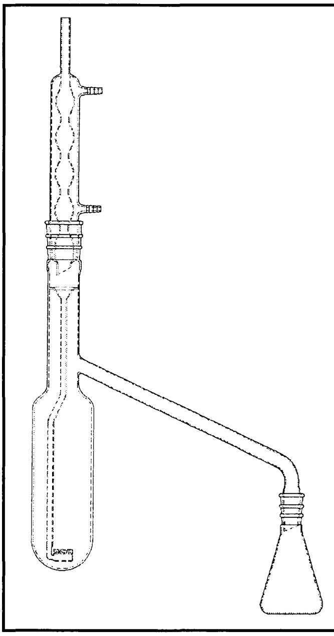


Figure 6. Extraction Apparatus, Courtesy of Ace Glass.

In this first example (Figure 6), it can be readily seen that processes of distillation, condensation, and extraction, followed by redistillation are the same as in the "heavier than water" extractor. The solvent guide tube is the main difference. The body of the extraction apparatus is filled with the material to be extracted. The flask is filled with the extracting solvent. Initially the extracting solvent will not pass through the material being extracted, but as the guide tube starts to fill, enough hydrostatic pressure will be developed to eventually force the extracting solvent out the bottom. The solvent extracts as it rises and returns to the distillation flask via the return arm.

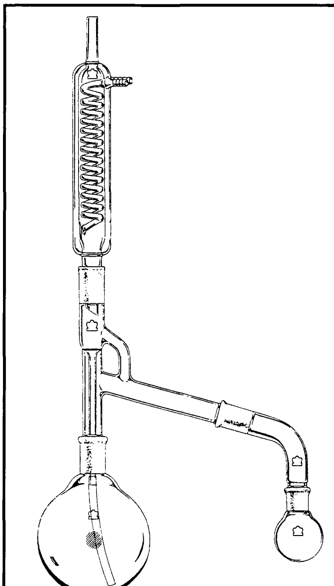


Figure 7. Apparatus, Continuous Liquid/Liquid, Lighter than Water, Courtesy of Kimble/Kontes.

This second example (Figure 7) functions in exactly the same way. The aqueous solution is placed in the large flask. The "less dense" extracting solvent is placed in the smaller flask attached to the return arm. The extracting solvent is distilled from smaller flask, condenses and drips into the solvent guide tube, extracts the desired material as it bubbles up through the aqueous solution and overflows into the distillation flask, via the return arm, for reuse.

This Mini-Lab extractor (Figure 8) is a nice compact unit for small amounts of material. Note the solvent guide tube which gives the extracting solvent enough pressure to push down through the more dense material being extracted. This extractor does not have a return arm. The extracting solvent overflows and returns to the outer, distilling chamber down the outside of the inner chamber.

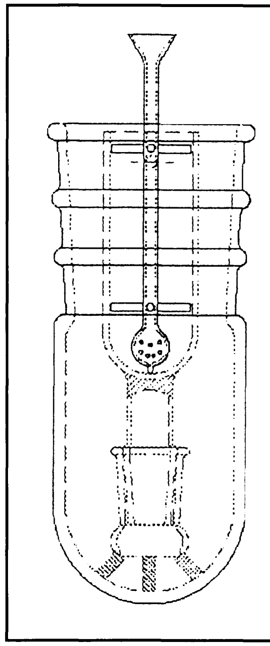


Figure 8. Mini-Lab Extractor, courtesy of Ace Glass.

There are many, many different designs of continuous liquid/liquid extractors. Some extractors, such as the Mini-Lab extractor can be configured to act as both a "heavier than water" and "lighter than water" extractor by changing the solvent guide tube. Other extractors, such as the Combination Solid/ Liquid – Liquid/Liquid extractor (Figure 9)[4] designed by Joe Gregar can be configured to serve multiple

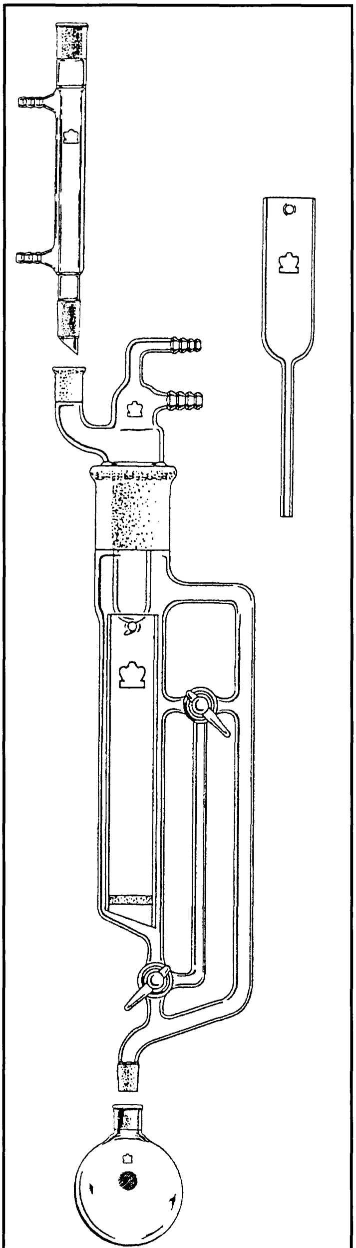


Figure 9. Apparatus, Continuous Solid/Liquid – Liquid/Liquid, Courtesy of Kimble/Kontes.[4]

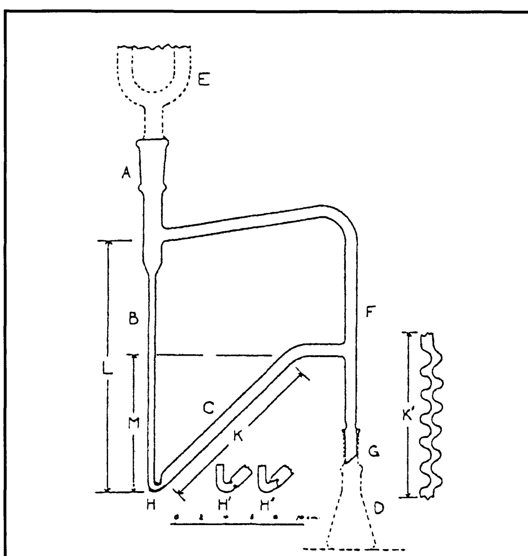


Figure 10[5]

[4]Design at Argonne National Labs by Joseph Gregar & Ken Anderson, Ph.D.

functions through the proper position-ing of stop-cocks and the proper insert.

These historic, complex and fascinating liquid/liquid extractors (Figures 10[5], 11[6], 12[7]) are, as they say, "left as an exercise for the reader." Yet, despite the apparent complexity of some and the simplicity of others, they all follow the same basic principles.

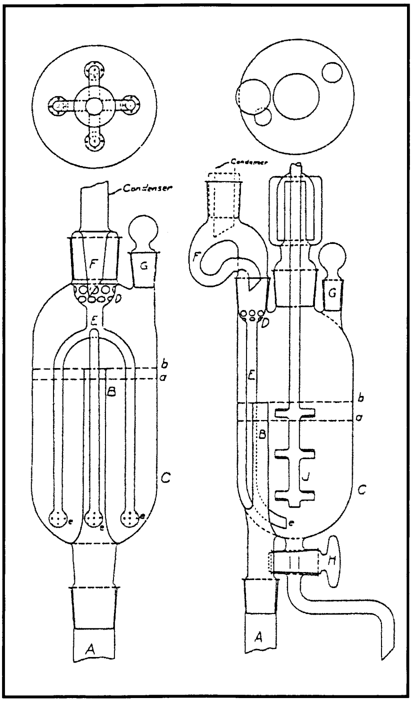


Figure 11[6]

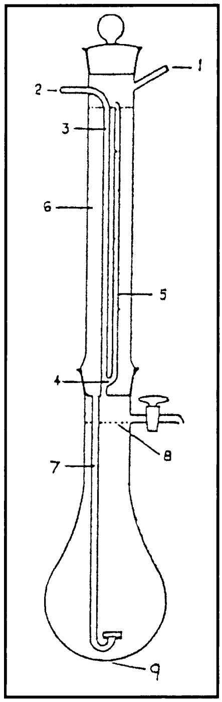


Figure 12[7]

CONCLUSION

Scientific glassblowers are an integral part of the science of chemistry. The knowledge of "how things work" adds to our value as glassblowers and the enjoyment of our profession.

ACKNOWLEDGEMENTS

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

[5]Reprinted in part with permission from M. Wayman and G. F. Wright, *Industrial and Engineering Chemistry, Analytical Edition* 17 (1945): 55-56. Copyright 1945 American Chemical Society.

[6]Reprinted in part with permission from W. A. LaLande Jr. and E. C. Wagner, *Industrial and Engineering Chemistry, Analytical Edition* 6 (1934): 300. Copyright 1934 American Chemical Society.

[7]Reprinted in part with permission from W. D. Long, *Industrial and Engineering Chemistry, Analytical Edition* 16 (1944): 180. Copyright 1944 American Chemical Society.

NMR Tube Cleaner: A New Design

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ABSTRACT

Over the past 30 years as the University's resident glassblower, I have had a continuous demand to repair NMR tube cleaners. This paper details how, after many experiments to improve upon the conventional design, I had an insight that would lead to a radical change in design and concept. This new innovative type of NMR tube cleaner, which is much more durable than the conventional type, led to the invention of a semi-automatic vial or small flask cleaner.

A LITTLE BACKGROUND

NMR, nuclear magnetic resonance, is to chemists what MRI, magnetic resonance imaging, is to medicine. Both will give precious information in a non-destructive manner (a rather nice touch for human beings). This technology is so useful that today it would be unthinkable to do research in chemistry without the aid of NMR. Many atomic nuclei behave as though they are spinning, and, as a result of their spin, each nucleus possesses an angular momentum and a magnetic moment. Although these two properties were observed in the 1920's, it took 20 years of studies to become a tool for research.

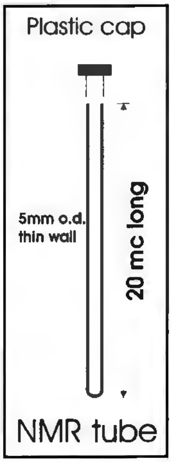
In 1938, Dr. Rabi and his team at Columbia University made some major improvements in beam experiment, which culminated in the first observation of nuclear magnetic resonance. This work earned Rabi a Nobel Prize in 1944. In 1946, two other scientists working at M.I.T., Bloch and Purcel, coined the term 'nuclear magnetic resonance;' both earned a Nobel Prize in 1952, but it still took another 20 years for the technique to be widely used in research. Another Nobel Prize in the same field was later awarded to Richard Enrst in 1991. Since the mid 1970's, many techniques have been developed to obtain two-dimensional and then three-dimensional images of macroscopic objects.

THE THEORY

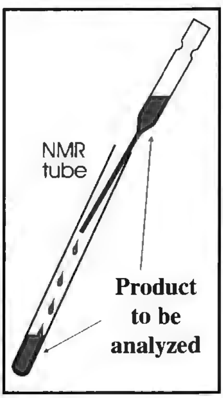
Nuclear Magnetic Resonance (NMR) is a technique to detect the chemical environment of a nuclei in a molecule. When a molecule is placed in a strong magnetic field, nuclei of different chemical environments will move at different and characteristic frequencies about the magnetic field. These frequencies can be detected with NMR by sending a radiofrequency to the sample and measuring its response. When the radiofrequency matches the frequency at which the nuclei moves about the magnetic field, "resonance" occurs, which creates an instrument response.

Needing to be inserted into standard cavities inside very powerful magnets being spun at 20 Hz, those NMR tubes with a very thin wall and extremely uniform in diameter, are manufactured in staggering amounts. Several hundred thousands of these tubes are manufactured, sold, and used each year in North America alone. Since those tubes are still rather expensive, most chemists will try to re-use them many times before they finally end up being broken. Hence the need to clean them. This brought to the front the need for a tool and a technique to clean them easily, and the NMR tube cleaner was invented.

This is a regular NMR tube. They come in two standard sizes, 5mm and 10mm in diameter, but the most commonly used one is the small one: 5mm by 18cm long. They are sold with a little plastic cap to close them, this cap once put on the bottom of the tube will hold it in place upside down in the NMR tube cleaner.

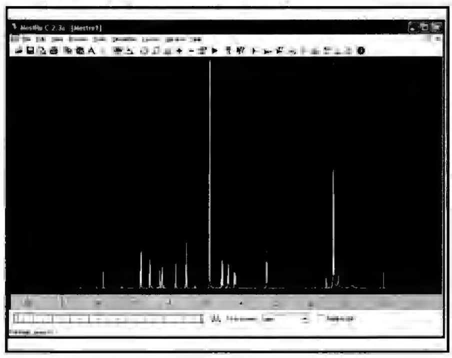


The researcher will introduce the unknown chemical in the NMR tube and will "run" it in the NMR machine. The computer will display a graph and an analysis of the different chemicals contained in the tube.

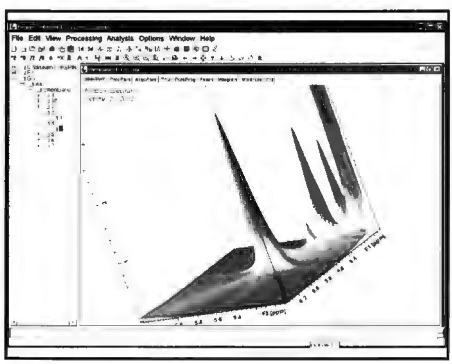


This is one of the several NMR machines in our department; this one is a 500MHz.

Dr. Zhicheng Xia, our NMR specialist doing an analysis on the computer.



A regular one-dimensional view.



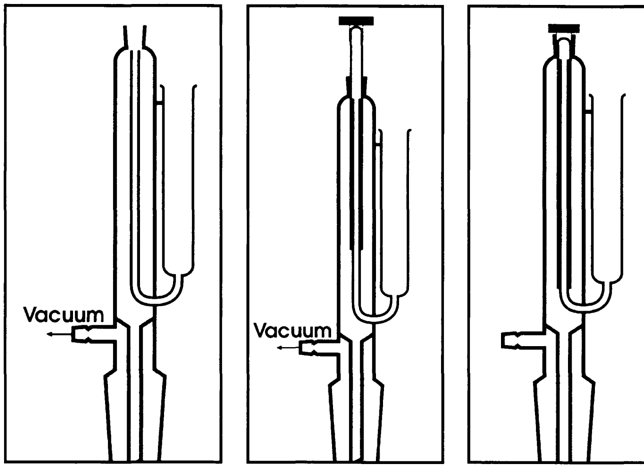
A beautiful two-dimensional view.

After having run the sample, the chemist needs to clean the NMR tube. Since it is very narrow (5mm o.d.), and quite a long tube (usually 18 cm), it is rather difficult to remove the material that seats in the bottom of the tube. This is where the NMR tube cleaner comes into operation.

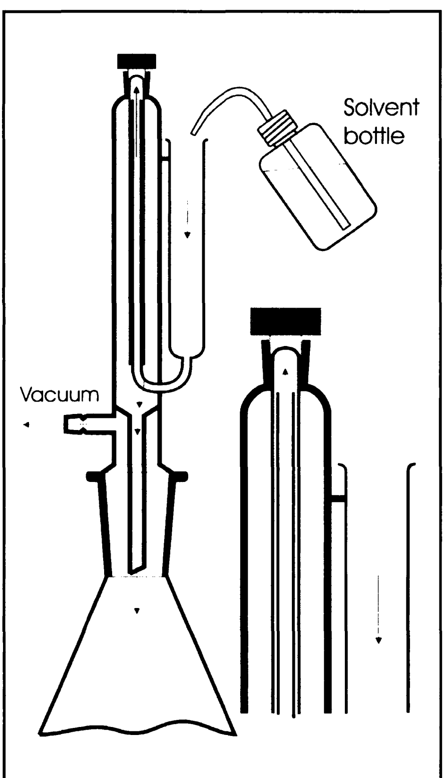
Generic type NMR tube cleaner. (left)

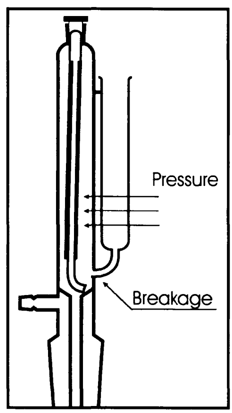
The NMR tube is being inserted in the cleaner. The cap will keep it in place and will seal the opening. (middle)

NMR tube cleaner with NMR in place ready for cleaning. (right)

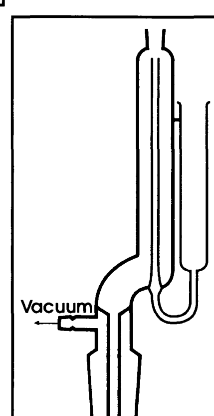


In this conventional design (below), the solvent is introduced in the outside tube; once vacuum is applied, the atmospheric pressure pushes the solvent up the inner tube. Like a hose spray, it dislodges the waste which will drip down along the insides of the tube into the waste flask.



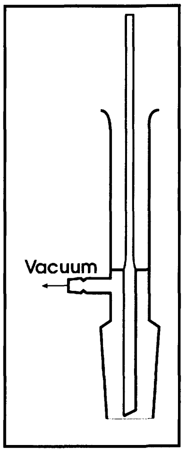


Unfortunately the inner jet (only 3mm o.d.) is extremely fragile, especially at the bend: a slight twist while inserting the NMR tube will break it, resulting in an extremely difficult repair. (left)

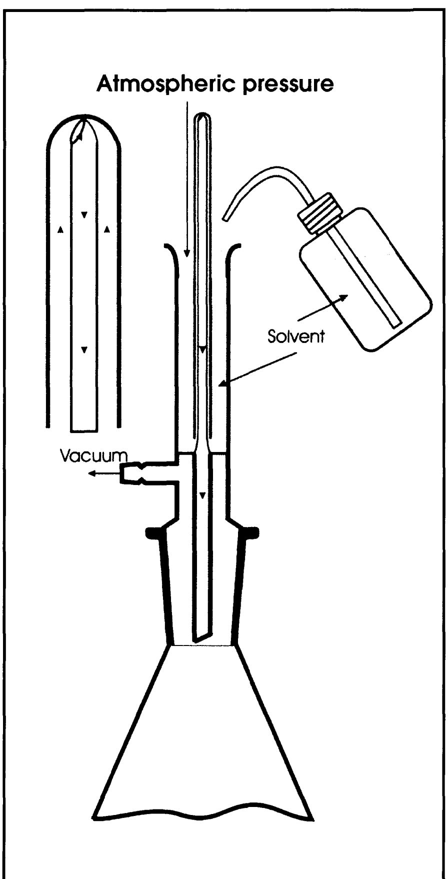


My first attempt at improvement was to attach the cleaning tube to the bottom of the assembly. This resulted in a much stronger joint and a slightly easier repair. But this takes more time to build. Unfortunately in this conventional design, the jet still smears dirty solvent to clean areas. (right)

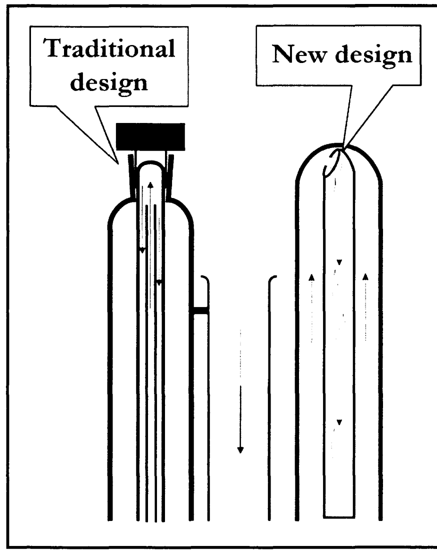
The final design is completely different from the original one. Because of the flexibility of the inner tube, it can bend without breaking (up to an extent). The jet actually sucks the dirty material instead of spraying it with solvent. (right)



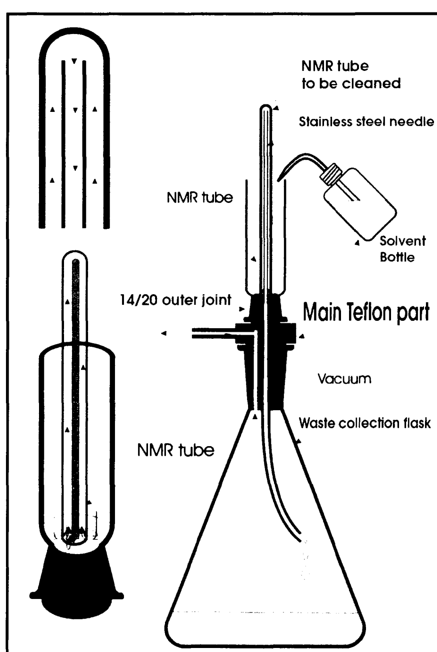
The NMR itself by being inserted in the cleaning solution, closes the inner jet to the air, permitting the vacuum to draw the solvent up the sides. The vacuum directly sucks the dirty material and pulls clean solvent up the wall into the waste flask via the stainless steel needle. (below)



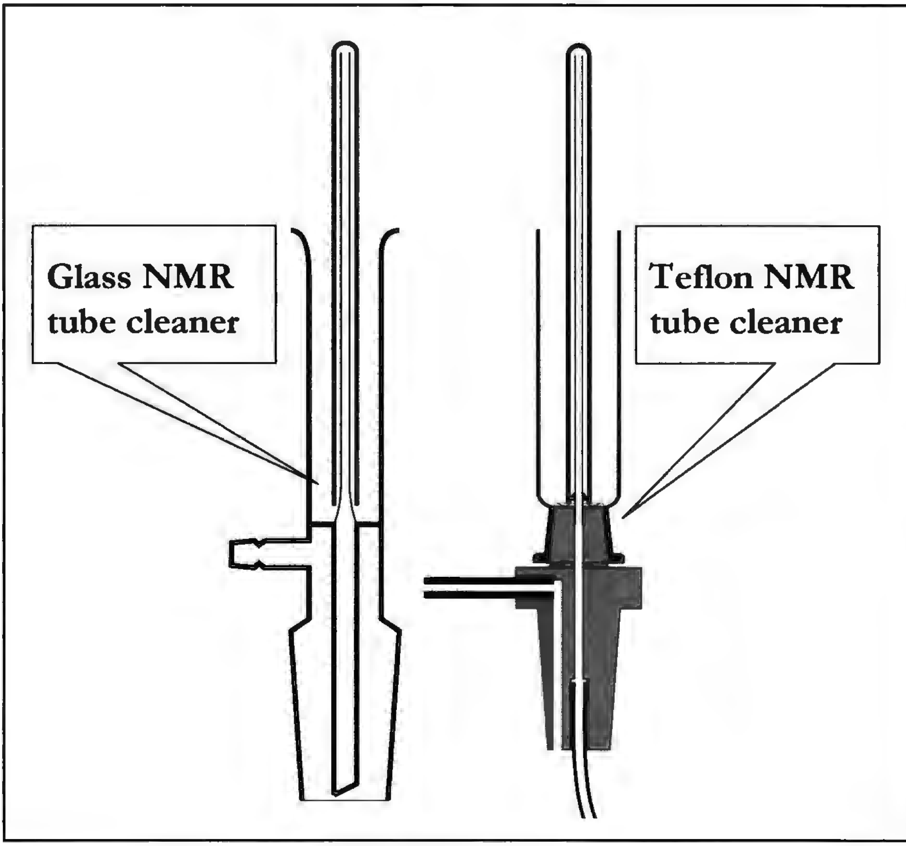
Below, on the right is the radically different design. This is complete opposite in concept and operation: vacuum cleaning versus spray washing.



Below is a design similar to the previous example but the main body is constructed entirely of stainless steel and teflon. It is nearly unbreakable. The only glass parts, a 14/20 outer joint, is easily replaceable.



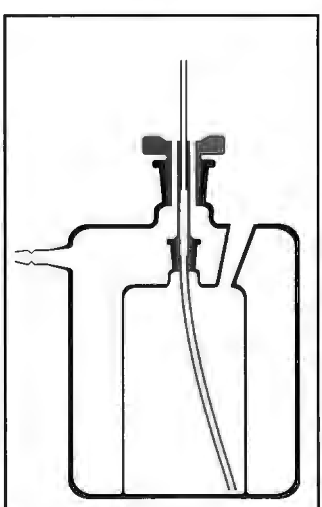
Same concept but different material

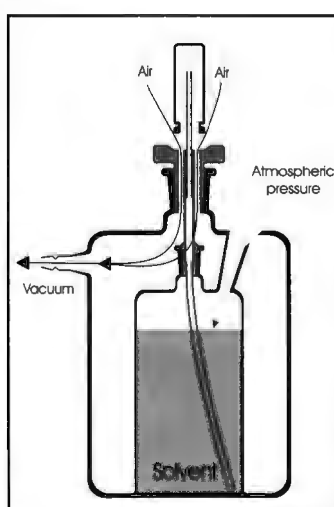


PART 2

Based on the previous idea, I would like to present a vial cleaning flask.

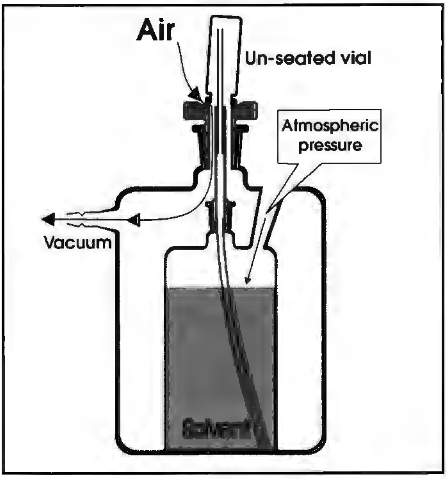
This vial cleaning flask is entirely constructed of glass and teflon, and is semi-automatic in operation. (left)



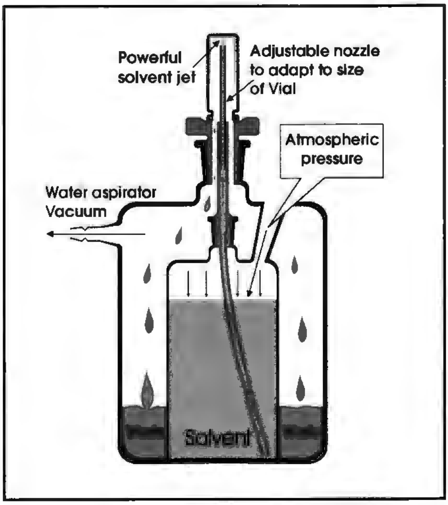


Until a vial is properly seated on the teflon fitting, the vacuum is not created in the chamber; therefore the solvent does not go up the jet, and nothing happens.

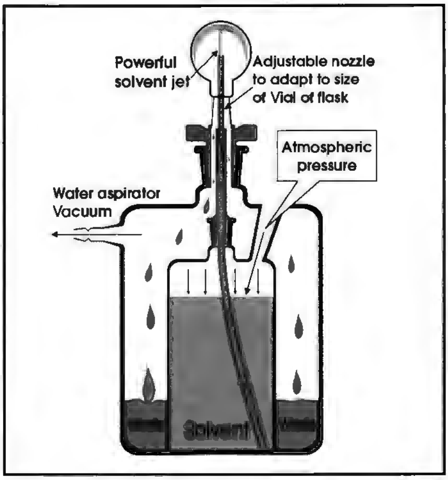
The vial is almost seated properly but not completely; as a result, a vacuum is not yet created, and nothing happens. (right)



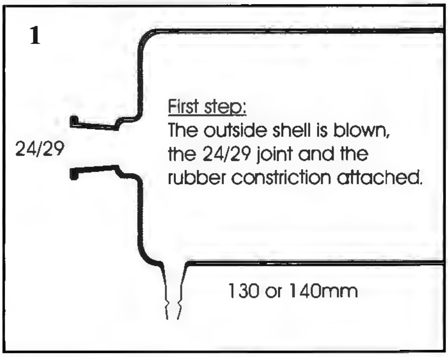
As soon as the vial is properly seated, the vacuum is created in the outer chamber; a powerful jet of solvent is then projected into the vial, thoroughly washing it. As soon as the vial is removed, the vacuum breaks and the washing stops. One just needs to place another vial to repeat the process. (below)

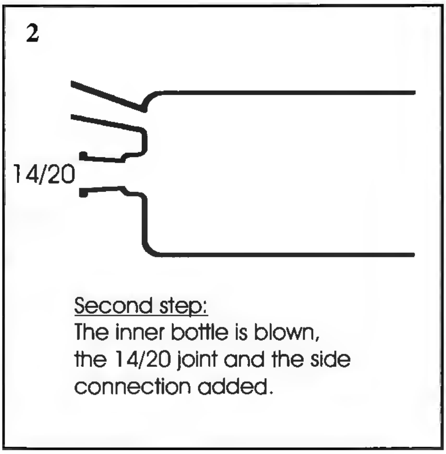


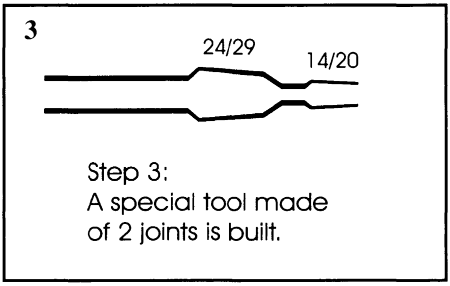
The same vial cleaning flask could be used to wash small flasks as well. (right)

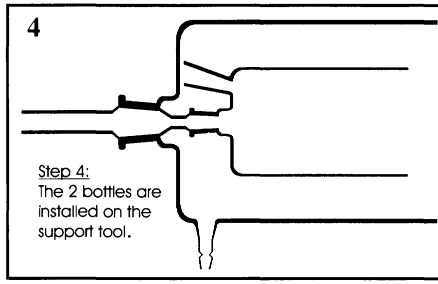


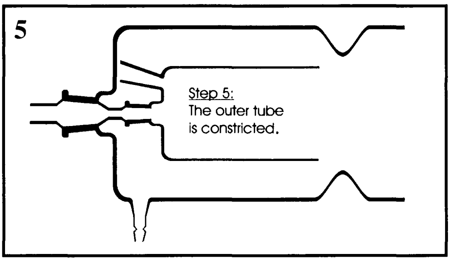
CHAMBER CONSTRUCTION

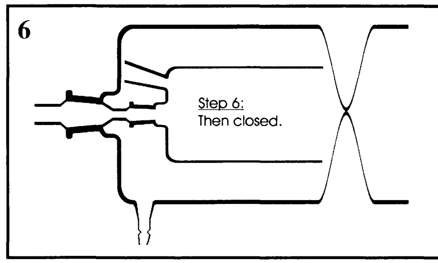


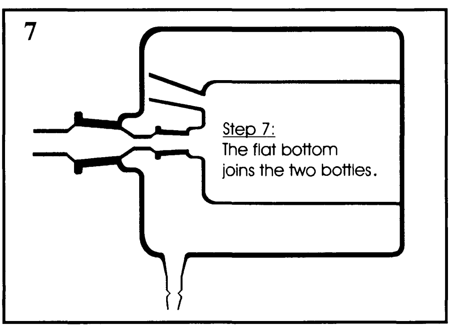


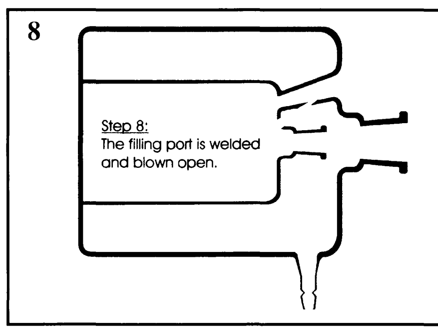




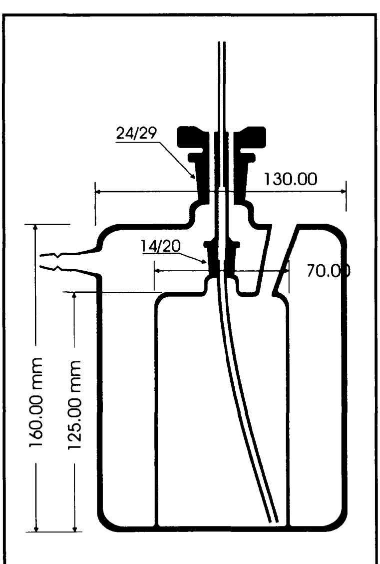




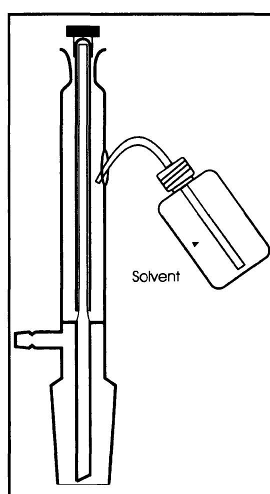




Measurements of cleaning chamber.



Last minute improvements. After several breakages last week in the labs, I came up with a final solution on how to protect the inner tube while keeping the reverse type of cleaning. I made the body full length and blew an opening on the side for the solvent.



I want to express my gratitude to Mr. Trempe, Administrator and to Dr. Lennox, Chairman of the Chemistry Department of McGill University for their support.

A Novel Colon Phantom

by
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ABSTRACT

Our goal was to create an enduring colon phantom that would simulate a cleansed and air-distended colon. This phantom is used to aid the radiologist in assessing the accuracy and dosage needed for the successful detection of polyps in the colon using the technique known as Computerized Tomography (CT) Colonography.

I would like to share with you this project that was a collaboration between the Department of Radiology and the Division of Engineering at the Mayo Clinic in Rochester, MN. Our goal was to create a colon phantom that could be used to evaluate a new imaging technique of the colon known as Computerized Tomography (CT) Colonography. This technique is used to assess different polyps' location, size and morphology.

Before telling you about the phantom we made and the experiments that were conducted using it, I would like to inform you about the importance of colorectal screening of which CT Colonography is one of several tests now available for this purpose. Colorectal cancer is one of the leading causes of cancer-related deaths in the United States. This common disease is preventable because most colon cancers arise from pre-existing benign polyps. The detection of pre-cancerous polyps and their removal can help prevent most cancer related deaths.

CT colonography is a full anatomic examination of the colon and rectum using volumetric data obtained with a helical CT scanner. It is a non-invasive procedure, which requires a single breath hold by the patient, who has been prepared with a cleansed and air-distended colon. The actual examination of the colon is done at a workstation by a radiologist, rather than in the patient. This makes for a safe and highly acceptable examination for patients. (Figure 1)

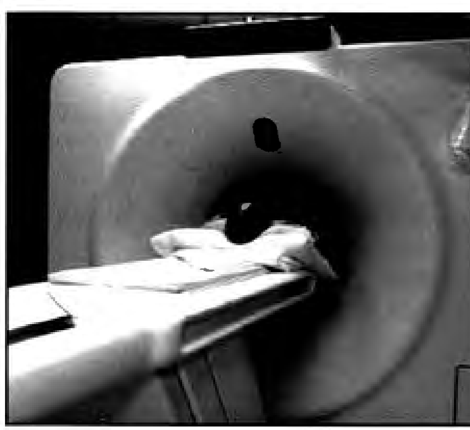

Figure 1

[1]Division of Engineering, Mayo Clinic, Rochester, MN.
[2]Department of Radiology, Mayo Clinic, Rochester, MN.

After the digital images are acquired, they are transferred to a workstation for interpretation. The radiologist uses a combination of axial images, multiplaner images and 3D endoluminal images to examine the colon. The 3D endoluminal views simulate the view at colonoscopy without the use of any internal instruments. (Figure 2)

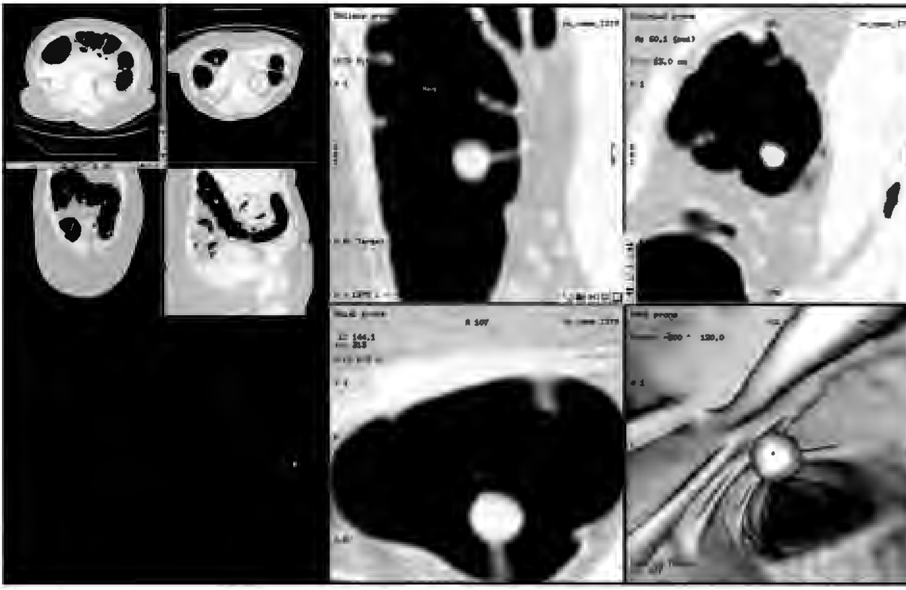

Figure 2

This slide shows the sensitivity of CT colonography for large polyps in two major studies. The sensitivity ranges from 75%- 90%, nearly as good as colonoscopy, the current gold standard for colon examinations.

The current sources of error at CT Colonography can be divided into both technical errors and perceptive errors. Technical errors refer to polyps that cannot be detected even retrospectively. Perceptive errors refer to those polyps that can be seen in retrospect but are prospectively overlooked. (Figure 3)

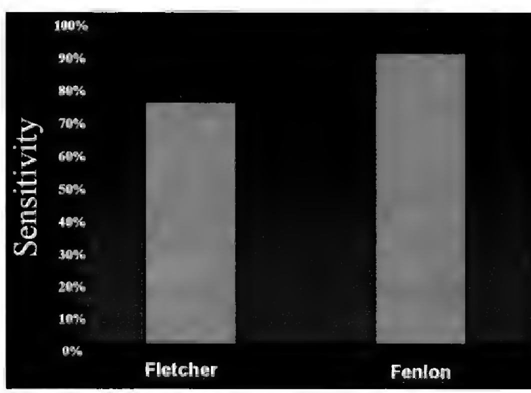

Figure 3

Theoretically, technical errors could be overcome by optimization of CT technique. The purpose of this study is to address the technical sources of error at CT Colonography. The aim was to create an enduring colon phantom with multiple polyps of varying shapes, sizes and locations. This phantom would be used to determine the optimal radiation dose needed to establish polyp size, morphology, and location.

The colon is a tortuous tube with many folds known as haustrations. After investigating several different materials, the decision was made to create a custom phantom using borosilicate glass. A life size anatomically correct model that would simulate an air-distended colon was constructed from 51mm standard wall tubing. The phantom was made up of five sections. These sections were designed with 28 folds, and four bends or flexures. After the polyps were in place, the sections were held together with a silicon adhesive and shrink-wrap. The completed phantom holds 140 polyps and is submersed in a 12" o.d. Plexiglas water bath that simulates the soft tissue attenuation of body organs that surround the colon. (Figure 4)

Figure 4

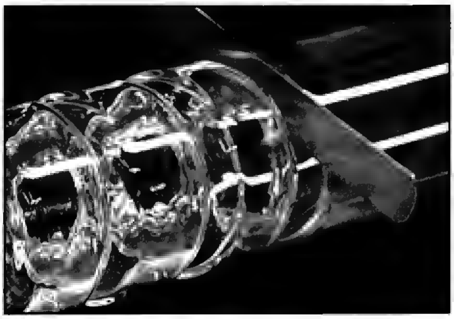

Figure 5a

Figure 5b

The colon's haustrations were formed by heating a small band around the tube and shaping the softened glass with a tool made of 1mm x 15mm stainless steel. After the initial shape of each haustration was achieved, a #1 National torch tip was used to go around the surface at the tip of each fold in order to remove any sharp edges created during the shaping process. As each fold was formed and shaped to the desired diameter, the piece was flame annealed to remove any stress created during the shaping process. The technique used to make glass bellows could have been used here, but the haustrations would have been too perfect. The colon is anything but uniform, so in an attempt to capture the actual shape of the colon this technique was chosen. (Figures 5 a & b)

Polyps that grow in the colon are generally classified into three types: pedunculated polyps have a stalk, sessile polyps are like a 1/2 sphere, and flat polyps are twice as wide as they are high. These various types of polyps were created from a substance called Solid Water. The material, Solid Water, is an epoxy resin that has the absorption and scattering properties within 1.0% of living tissue. (Figure 6)

Figure 6

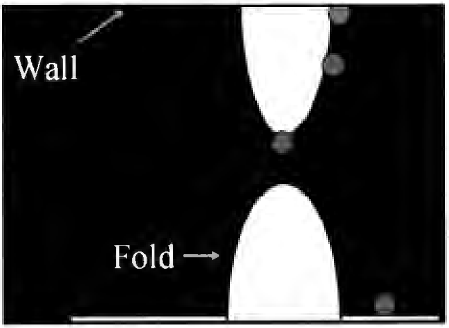

Figure 7a

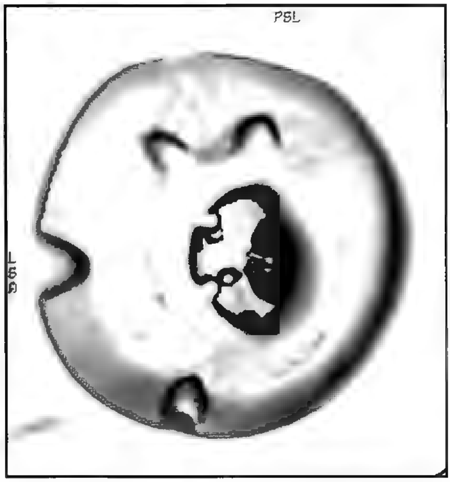

Figure 7b

The simulated polyps were attached with a silicone adhesive to various locations in the phantom – either on the colon wall, at the tip of the fold, on the fold, or at the base of the fold. The location of each polyp was mapped so that the radiologist could assess whether they were visible through CT Colonography or not. (Figure 7a) 3D endoluminal image of the phantom shows polyps on the tip base on the fold. (Figure 7b)

Various CT techniques were used for the 13 experiments performed on the phantom. Notice that three different slice thickness were used as well as radiation doses that ranged from a moderately high dose to an extremely low dose technique. Please note Series 5 since this becomes important in the results of the lowest dose and noisiest data set. (Figure 8)

Series	Slice Thickness	mAS	Dose (mGy)
1	1.25	308	24.43
2	1.25	145	11.5
3	1.25	65	4.87
4	1.25	15	2.25
5	1.25	5	0.37
6	2.5	135	10.71
7	2.5	65	4.87
8	2.5	30	2.25
9	2.5	5	0.37
10	5	65	4.87
11	5	30	2.25
12	5	15	1.12
13	5	5	0.37

Figure 8

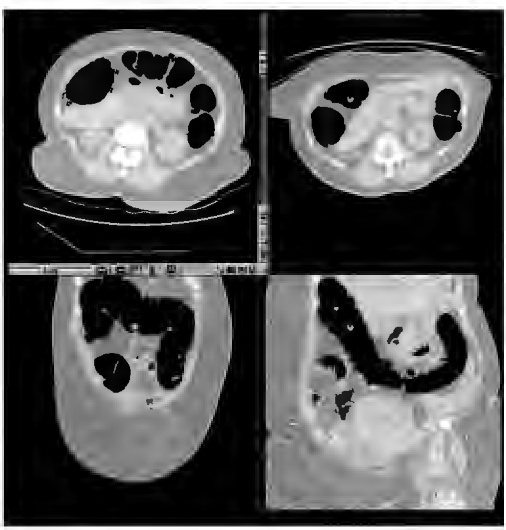

Figure 9

Each of the 13 Series were reviewed by a diagnostic radiologist with experience in CT colonography. Using the polyp map, each of the 140 polyps were assessed if they were visible or not. The radiologist was not blinded to the presence of the polyps since the goal was not to assess the reading accuracy of the physician but rather to assess whether or not the CT technique was adequate for polyp detection. (Figure 9)

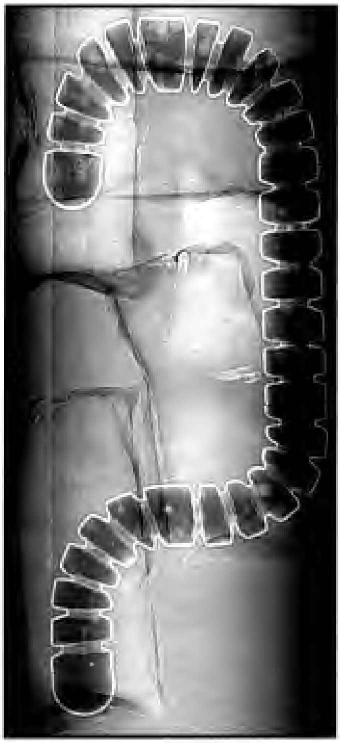

Figure 10a

Figure 10b

Here is the phantom in the water bath being scanned on the CT scanner. Notice how haustral folds were planned so that they were cut by the CT scanner in either perpendicular, oblique or parallel fashion. (Figures 10 a & b)

All polyps in the 13 series for 12, 10 and 7 millimeter sizes were detected regardless of CT technique. The 5 millimeter polyps were all detected with the exception of series 5 where only 87% were detected. Series 5 was the lowest dose technique using a mAS (milliamp second) of 5.

The CT Scanner has the ability to detect polyps of various shapes and sizes. All pedunculated polyps regardless of CT technique were located. Similarly, all the sessile polyps were detected except in series five, the lowest dose technique in which only 96% of the sessile polyps were detected.

All of the flat polyps were detected, except those in series five, the lowest dose technique, here only 94% of the flat polyps were detected.

The location of the polyp on the haustral fold had no effect on polyp detection except in series 5, where two polyps on the wall and three polyps at the base of the fold were not detected. The relationship of the fold to the gantry had no effect on polyp detection except for series 5, which had four missed polyps on folds oriented oblique and one parallel to the CT gantry. All polyps were detectable at all dose settings except when using the CT technique with the highest relative noise (series 5, 1.25 slice thickness and 5 mAS).

LIMITATIONS

- The borosilicate glass wall of the phantom has a higher density than soft tissue causing mild streak artifact. This did not interfere with polyp detection.

- The full spectrum of natural polyp shapes and sizes were not represented. Only the most common shapes and sizes were used, including flat polyps that are considered to be the most difficult to detect.
- The goal was to determine if the technique was adequate for detection. Reader performance and inter observer variability was not assessed.

CONCLUSION

- An Enduring phantom was created that can test various CT techniques used at CT Colonography. This phantom will be useful in the evaluation of new CT scanners.
- All polyps were detected regardless of their size, location on the haustral fold, morphology, or x-ray dose, save the five polyps that were missed using 1.5 millimeter slice thickness at 5 mAS.
- In this model, polyp detection was rarely related to differences in CT technique. Other causes of errors at CT Colonography should be investigated.
- Confirmation in a human population would be helpful in the future.

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Rapid Prototyping of Microfluidic Systems in Glass

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ABSTRACT

High-resolution microchannel flow cells were required for a new hand-held Surface Plasmon Resonance (SPR) detector. A procedure was developed using a commercially available high resolution photolithographic method to fabricate these channels in glass. The samples produced with this method had uniform and consistent lateral definition and depth. In addition, these channels demonstrated good flow characteristics, chemical resistance and biocompatibility. This procedure can be adapted to many applications involving small fluid channels in glass.

INTRODUCTION

A research project in the UW-Madison Chemistry Department required the fabrication of small fluid channels in a chemically-resistant material for use in an analytical instrument. Typically, channels of this type are formed from polydimethylsiloxane (PDMS), but this application required a more durable seal than could be obtained with this material. A procedure was developed using a commercially available high resolution photolithographic method to fabricate these channels in glass. The lateral definition and depth of the channels was uniform and consistent between samples. In addition, these channels demonstrated good flow characteristics, chemical resistance and biocompatibility. Fabrication of a set of channels takes approximately 30 minutes, not including artwork preparation. Much of the work can be paralleled, so that making multiple channels does not take significantly longer than a single unit. The completion of this project used a technique demonstrated at the 2002 Midwest section meeting held at the University of Wisconsin's Chemistry Department Glass Shop.

SURFACE PLASMON RESONANCE (SPR) FUNDAMENTALS

High-resolution microchannel flow cells were required for a new hand-held Surface Plasmon Resonance (SPR) detector. While the techniques discussed below are for a specific application and project, they can be adapted to many applications involving small fluid channels.

SPR is a surface-sensitive optical technique used to check to detect the presence of biological molecules in solutions by exploiting their binding affinity for complementary surface-bound species. SPR imaging detects biomolecular affinity on a chemically modified gold surface by monitoring the change of index of refraction upon absorption of the target molecules to the surface. Light is passed through a prism at an angle such that the light is totaly reflected from the gold surface back to a detector.

Fabrication of SPR samples begins with a thin gold film vacuum deposited on one face of a triangular prism. A polydimethylsiloxane (PDMS) mask is attached to the gold surface. Channels in the PDMS are filled with solutions used to attach an array of bio-molecules to the gold. The mask is removed and a glass microchannel cell is adhered to the gold over the array. Glass channels were selected for this application because PDMS does not adhere adequately to the prism to be used as a flow cell in applications outside the laboratory. Channels fabricated in glass can be attached to the prism using adhesives, forming a reliable, permanent seal.

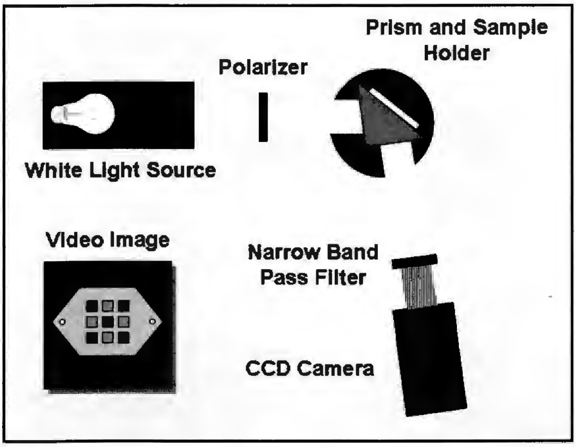

Figure 1. *Principle of Surface Plasmon Resonance (SPR).*

In order to perform an analysis, a sample containing unknown bio-molecules is passed through the cell as a beam of monochromatic light is directed at the gold surface through the prism at a specific angle. At a fixed angle, the reflectivity of the gold surface changes as molecules flowing through the cell attach to the base array molecules. SPR uses the measurement of this difference in reflectivity to indicate binding of the target molecules to the array. This concept is illustrated in Figure 1.

The hand held SPR unit is shown in figure 2. The prisms are machined from polystyrene and the required flow cell dimensions are 500um (20 mils) wide and 100um (4 mils) deep.

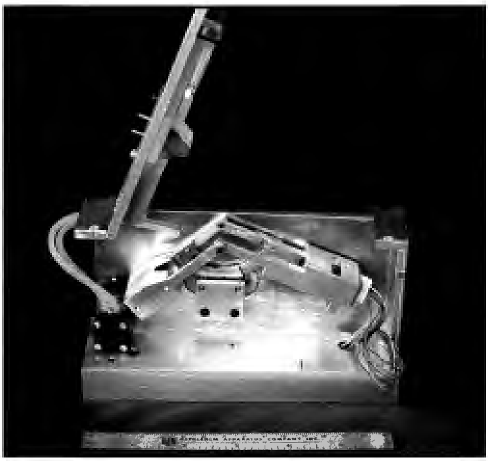

Figure 2. *Hand held SPR Detector.*

MICROCHANNEL CONSTRUCTION

High resolution and quick production times for prototyping and production were required. PhotoBrasive™ Systems has one of the most comprehensive lines of photolithographic resist films in the industry. Based on the microchannel dimensions and speed requirements, the use of RapidMask™ film was chosen.

RapidMask™ is a green, UV-curing, self-adhesive, 3-layered photolithographic resist developed by DuPont. Sheets of the product come in 3 and 6 mil thickness. According to their literature, the 3 mil thickness has a 3 mil resolution to a 1/16" depth. The three layers include: the self-adhesive UV-curing photo resist layer, a shiny protective covering film and a dull release backing sheet. Because the resist is UV curing, it requires no washing out or drying stages which are typical with other photo resist methods.

CREATING THE ARTWORK

There were two flow cell configurations: a worm array and a hexagonal cell. These are shown in Figure 3. The configurations were produced with Adobe Illustrator and printed on transparency film from a Xerox Phaser 1325 printer. The artwork must be photonegative. Exposure of the RapidMask film to UV light through the clear areas of the mask hardens the film, turning it brittle and blue. These areas are will be blasted away. The unexposed film will form the resist.

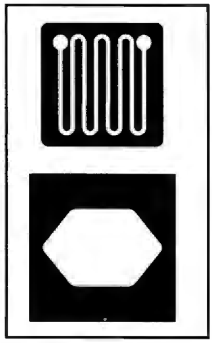

Figure 3. *Flow cell: worm array (top), hex cell (bottom).*

EXPOSING UNDER LONG-WAVE UV

This process consists of six steps:

- The dull (emulsion) side of the artwork transparency is placed against the dull side of the RapidMask™. The photo resist process system includes a Letralite™ UV exposure unit and is shown in Figure 4.

Figure 4. *Letralite™ UV exposure unit.*

- The transparency is placed between the UV light source and the resist mask, and exposed for 90 seconds. Figure 5 represents the resist after exposure.
- The dull release backing film is removed and the film is applied to the cleaned glass slide.
- A rubber roller is used to improve the adhesion.
- The piece is allowed to sit for five minutes before blasting.
- The shiny cover sheet is removed before sandblasting.

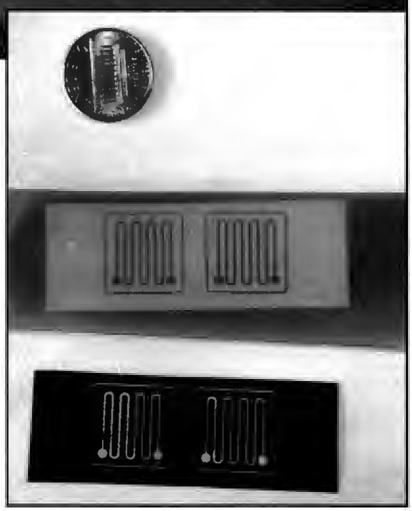

Figure 5. *RapidMark™ after 90 second exposure to UV.*

SANDBLASTING

The sandblaster was a siphon type sandblaster with 220 grit silicon carbide blasting medium. Operating conditions were: 60 PSI with a constant nozzle height of 150mm above and perpendicular to the glass. The set-up is shown in Figure 6.

Figure 6. *Sandblaster set-up.*

Prior to making the cells, a number of tests were made to determine blasting time versus depth. A dial indicator on a granite surface plate was used for depth measurements and proper fluid flow in the cell was verified. Based on the results of these tests, a blasting time of ten seconds was used to achieve the desired 100um (4 mil) depth.

REMOVING THE MASK

After blasting, it is important to remove the mask without scratching the glass. The glass was put in an ultrasonic cleaner with hot water and sodium carbonate (1/4 C to a gallon of water) until the mask was removed. See Figure 7.

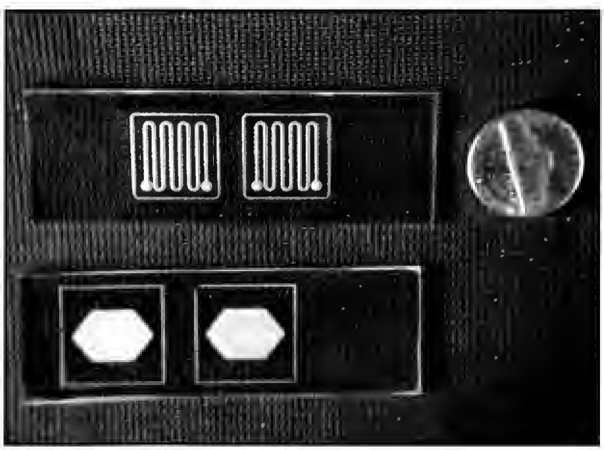

Figure 7. *Glass slide after removing the mask material.*

An alternative method is to brush the slide with soapy water under hot running water to remove the grit particles and mask. After the mask is removed, the cell is cut to final dimensions and the edges are sanded.

DRILLING HOLES

The holes through the plate allow fluid flow from the instrument into and out of the cell. The openings in the cell are sealed to the instrumentation with 1/16" rubber O-rings. A small drill press was used with a 1.3mm diameter diamond-coated twist drill bit. The glass slide (blasted surface up) is adhered to a glass backing plate, using Crystal Bond 509®(Aremco product). A Sharpie® marker is used to mark the location for the holes. The holes are drilled in a shallow tray of water. Acetone is used to soften the Crystal Bond® and remove the cell from the backing plate. Alternatively, the cell plus backing plate, can be placed in the annealing oven and ashed at 300°C for 2 hours. A completed cell is shown in figure 8.

Figure 8. *Completed hexagonal cell.*

ADHESION OF CELL TO PRISM

For the hex cell, a drop of superglue is applied in each corner of the cell. Then, it is pressed onto the prism.

This technique did not work for the worm array cells. Fluid short-circuted the channels. For the worm array, a photo-positive was used to generate a silk screen of the worm array. The screen was registered over the glass cell and a screen printable adhesive (tra-bond 2450) was squee-geed on. The adhesive is uniformly applied to all spaces around the channels.

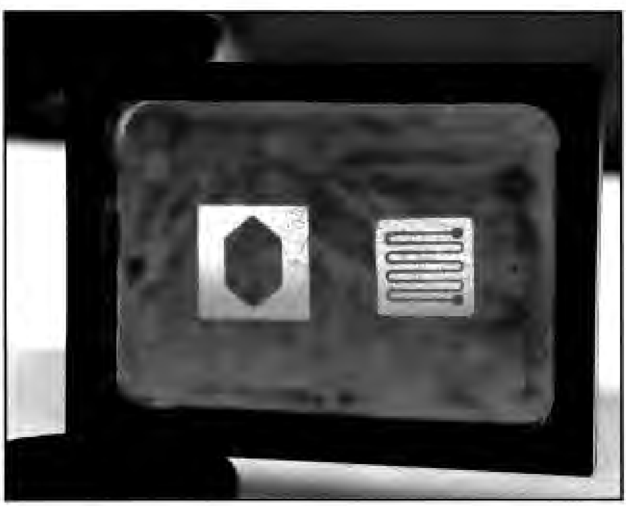

Figure 9. *Silk screen for adhesive application.*

The glass was then applied to the prism. The silk screen is shown in Figure 9 with the final worm array cell adhered onto the prism shown in Figure 10.

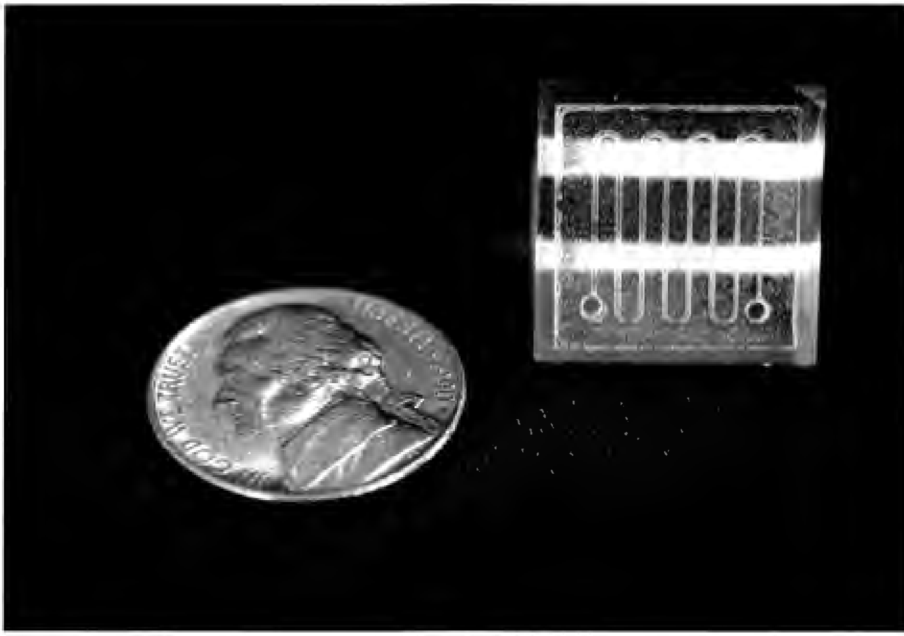

Figure 10. *Completed worm array cell adhered to polycarbonate prism.*

CONCLUSION

Fluid microchannels on glass microscope slides have been successfully constructed for an analytical instrument in the Department of Chemistry at the University of Wisconsin in Madison. This process is consistent, repeatable and fast. It is useful for any application requiring high resolution and detail definition.

Acknowledgements

I would like to thank the Department of Chemistry for their support and encouragement. I would also like to thank Professor Corn and his group for their collaboration on this project.

Sample Vessel for Measuring Hydrogen Production on Irradiated Molecular Sieves

by
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ABSTRACT

This paper will cover the design and fabrication of a simple sample vial. It will also include a step-by-step description of the sample preparation leading to the irradiation of the molecular sieves in a quest to generate free hydrogen;

Sometimes we scientific glassblowers are asked to do more than just blow glass. This was one of those jobs when the glassware was just the beginning. The researcher needed sample vials fabricated to contain molecular sieves, which had to be heated, evacuated and purged with argon. Then a measured amount of degassed water was to be added to the sieves. After this, the vials would be irradiated in our cobalt source. What the researcher was looking for was the production of free hydrogen over the sieve bed, so we needed to do a gas analysis of the vial.

Figure 1. *Sample Vial #1 after irradiation. There is a brownish tint to the glass.*

Figure 2

My first task was to come up with an acceptable vial design. The vial had to be greaseless because, unknown to me, Apiezon grease gives off hydrogen when irradiated. I also needed to minimize the use of Teflon as it does not hold up well to radiation. My first design (Figure 1) used 24/40 O-ring joints with a J. Young glass plug stopcock on top and a #7 Ace Thread with septa on the side. Design two (Figure 2) also used the O-ring 24/40 joints but I used a 0-4mm Chemglass glass plug vacuum stopcock. There was some concern over the use of the O-ring 24/40 joints as there was considerable variation in the fit of the joints. Our final design (Figure 3) used a 0-8mm Chemglass glass plug vacuum stopcock with E.P. O-rings sealed to 1" tubing with the Ace #7 thread on the side.

Figure 3. *The final vial design is made with a Chemglass 0-8mm glass plug vacuum stopcock sealed to 1" medium wall tubing. A #7 Ace Thread with silicone rubber septa is sealed to the side.*

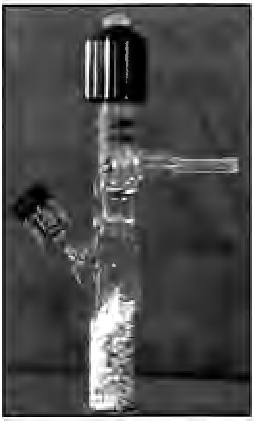

Figure 4. *Sample vial loaded with 10 grams of molecular sieves.*

Figure 5. *Sample vial in heating mantle. One thermocouple is connected to the mantle controller, the other is reference temperature.*

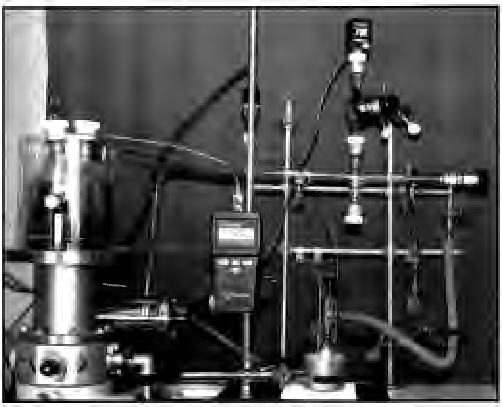

Figure 6. *Vacuum system consisting of Edwards CR100M Diffstak with an Edwards EM5 rotary vacuum pump. Heating mantle with aluminum vial holder is located at the lower right. The reference thermocouple readout is in the center. A Hasting HPM-760 vacuum gauge is located at the top right.*

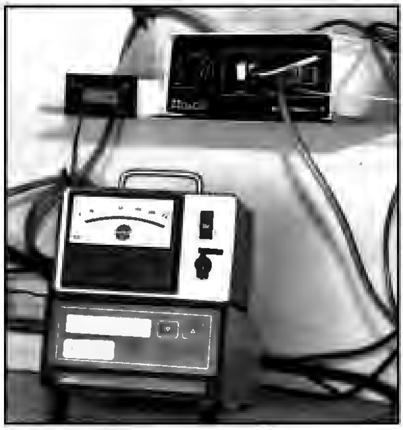

Figure 7. *The heating mantle controller is located at the top right. The power supply/display for the Hasing 760 vacuum transducer is on the top left. At the center is a Hasting vacuum gauge attached to a DV-8 gauge tube. Under this is an Edwards Series 1000 Controller attached to a Penning vacuum gauge.*

The pelletized molecular sieves needed to be baked between 125°C and 130°C while being evacuated. I used a 100ml beaker heating mantle for which I had an aluminum holder machined. The holder was 1.75" in diameter by 2" in height. A 1" hole was drilled in the center to hold the sample vial and two holes were drilled for thermocouples. One thermocouple was the mantle controller, the other was a reference temperature. To come up with the proper controller setting, I placed the controller thermocouple in the holder and the reference thermocouple in the sieves. When the sieves reached the required temperature, the controller setting was noted.

Figure 4 shows a sample vial loaded with sieves ready to be heated and evacuated. Figure 5 shows the vial in the holder being heated and evacuated. The vacuum system used consists of an Edwards EM5 rotary vacuum pump and an Edwards CR100M Diffstak. Figure 6 shows a sample being evacuated and Figure 7 shows the gauges and the heating mantle controller.

After heating and evacuating the sample vial for the required time I needed to back fill the vial with Argon. I closed the valve to the manifold then the valve on the sample vial. Next, I purged the manifold with Argon and then opened the sample vial allowing it to fill with Argon. The pressure in the manifold and vial can be read on the Hasting 760 Transducer.

Figure 8. *Vessel used to degass distilled water. Nitrogen gas was bubbled through the water for about an hour. The dispersion tube was removed and the bubbler sealed with a solid plug. Water was removed with a syringe through the Ace septa port.*

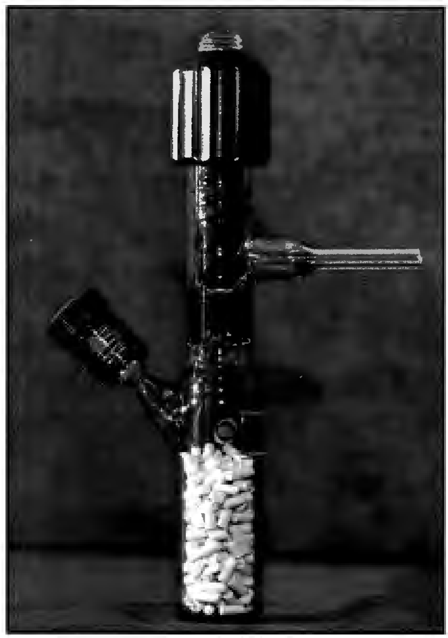

Figure 9. *Sample vial loaded with 10 grams of molecular sieve. Sample was gamma-ray irradiated for one hour. There is a brown tint, which is a characteristic of gamma radiation on borosilicate glass.*

The next step was to inject some degassed distilled water into the sample vial. The degassing apparatus is Figure 8. To degas the water, I bubbled Nitrogen through the water for about an hour. After degassing, I removed the dispersion tube and sealed the vessel with a plug. We used a syringe to remove the required amount of water through the Ace septa port. Then, using the Ace septa port on the sample vial, we injected the degassed water into the vial to achieve about a 15 weight percent per loading. The test vial was then exposed to gamma radiation using a J. F. Shepard Model 109 irradiator for one hour with an absorbed dose of about 9.22×10^5 Rad/hr.

Figure 9 shows a sample vial after it was irradiated. Gas analysis of the over pressure before and after irradiation was by gas chromatography.[1]

Figure 10 takes you through the time line for preparing sample vial #4. The results of this study entitled "Tritiated Water on Molecular Sieves Without Hydrogen Production" by R. Tom Walters, Kevin Sessions, Westinghouse Savannah River Company, LLC Savannah River Technology Center, Aiken, SC 29808 and C. Gentile, L. Ciebiera, Princeton University, Princeton Plasma Physics Laboratory, Princeton, NJ 08543 were published in *Fusion Science and Technology*, Vol. 41 May 2002.

VIAL # 4	7/09/01
Start Roughing Pump	7:08 AM
Start Diffusion Pump gauge pressure 2 microns	8:04 AM
Start Heater gauge pressure 0 microns	1:00 PM
Heater at 128°C gauge pressure 18 microns	1:20 PM
Turn Off Heater gauge pressure 4 microns Temperature 126°C	2:34 PM
Close Sample Vial gauge pressure 2 microns	3:00 PM
Purge line with argon gauge pressure 938.6 Torr Open Vial gauge pressure 874.5 Torr Close Vial	3:06 PM

Figure 10

The opportunity to see a project through from design to published paper does not come that often to the scientific glassblower. I learned a great deal working on this project and the glassware was only a small part of the job. My thanks to Tom Walters for acknowledging me in this paper and for supplying reprints. A special thanks to Jeff Siler for my photos.

[1]R. Tom Walters, Kevin Sessions, C. Gentile, and L. Ciebiera, "Tritiated Water on Molecular Sieves Without Hydrogen Production," *Fusion Science and Technology* 41 (May 2002): 685-689.

Some New Tools

by
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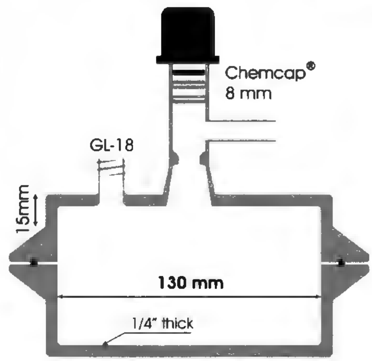
ABSTRACT

This paper presents some tools that I have designed this year. While some of them are not quite new in concept, they are rather improvements or maturations of previous ideas. The desire is always to simplify our work and, at the same time, to make it more efficient and safer.

Some researchers in our Chemistry Department were in need of a very shallow chamber. Since there was no easy way to hold the flange in the lathe using conventional tools, I had to find some new way to support it safely while welding the 1/4" window that was to be used as a flat bottom.



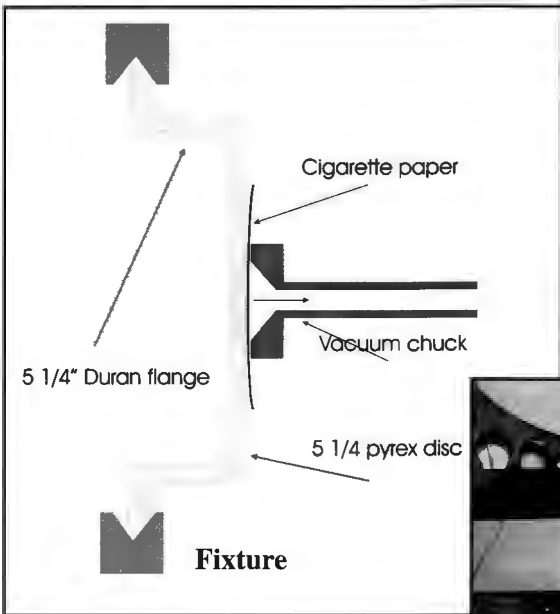
Vacuum chamber
built with 2 Duran® flanges
130 mm I.D.

Flange support tool

I decided to use my regular aluminum inserts. I had the machine shop cut a 90 degree V into one of them.

Very little pressure is used to support the flange in place to avoid distortion.

The flat bottom is welded to the flange without blowing. The 5 1/4" disc is held with a vacuum chuck.



One can see on the drawing that the glass window which is 5" wide and 1/4" thick is held via a vacuum chuck; some cigarette paper protects the glass and will not leave any ashes.

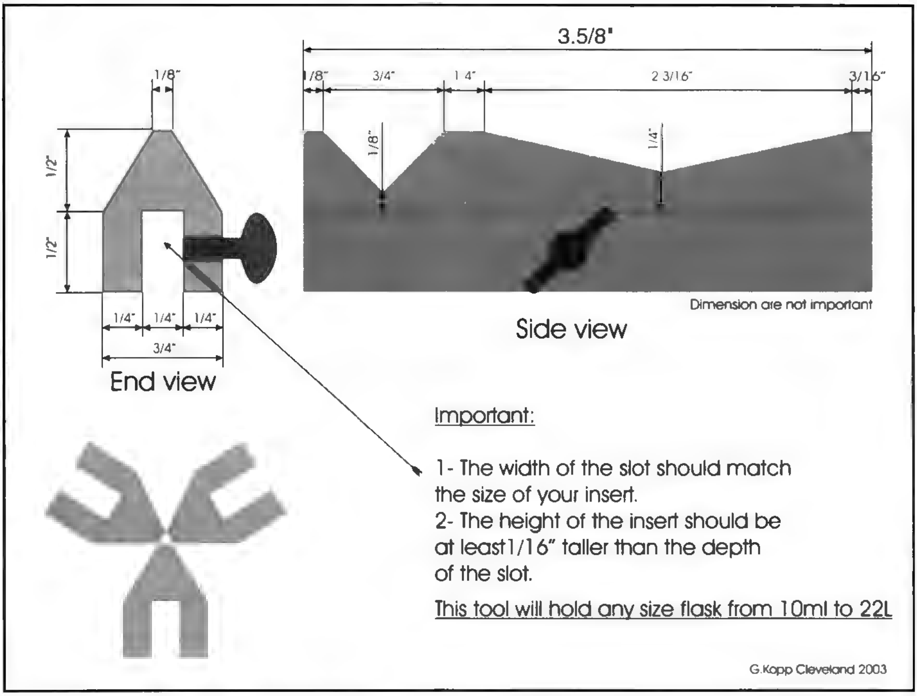
The fixture worked so well, that I later used it to support the foot of a vase.

UNIVERSAL FIXTURE FOR THE CHUCK

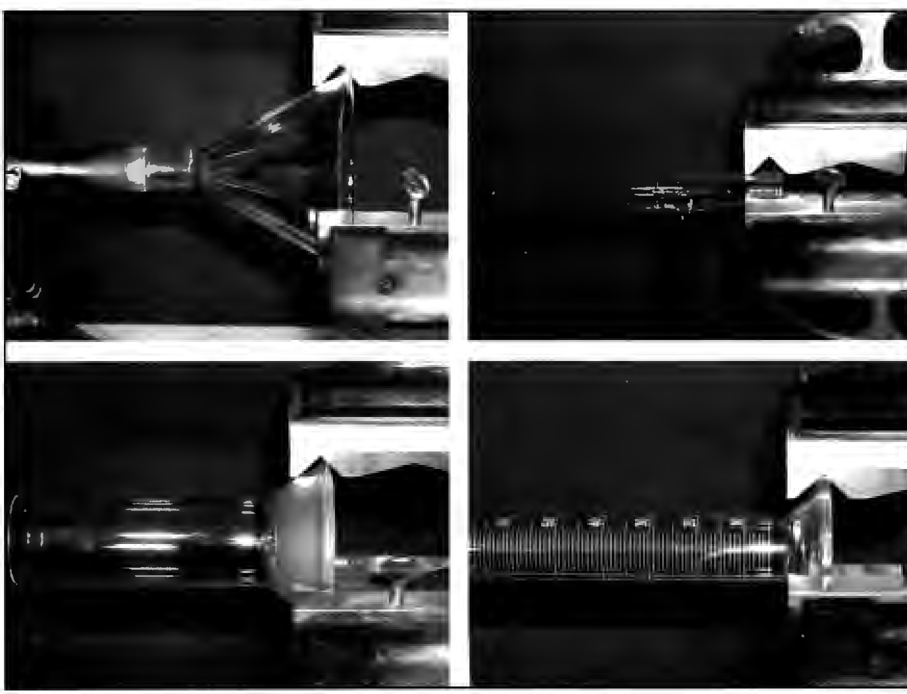
After using the previous fixture for a while, I thought about improving the design to make it even more versatile. I experimented with various configurations until I was satisfied with the following design. This one can safely support nearly any odd shape and I leave it almost permanently on my tail chuck.

To be used mostly on tail stock

A fixture that could hold just about any odd shape.

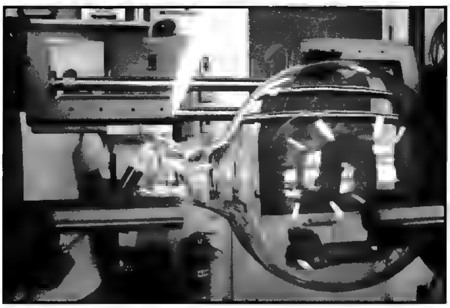


The next pictures will serve to illustrate some examples of odd shapes, as well as regular glass pieces that can be supported in the chuck. The fact that one does not have to change the fixture often improves work efficiency.



Very useful to support large flasks.

Adding a #75 O-ring joint to a 22L flask.



Adding the stopcock to the bottom of the 22L flask.

The top cover of the 22L flask with #75 O-ring joint and 4 GL-18.

Since that fixture is made of three sets of conical shapes, it becomes obvious that they have to be perfectly aligned to make the pieces run true. I designed a special tool that I use when I have to put it back onto the chuck in the event that I had to remove it.

A centering tool for the previous fixture.

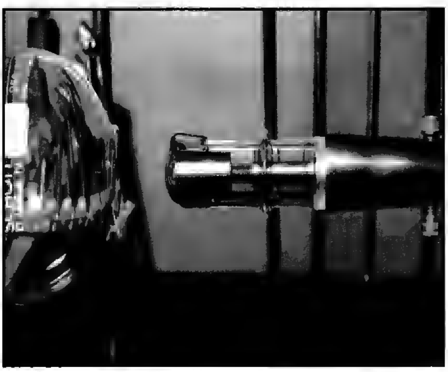
The fixture is tightened over the centering tool for a perfect alignment.

SOME NEW TOOLS PART 2

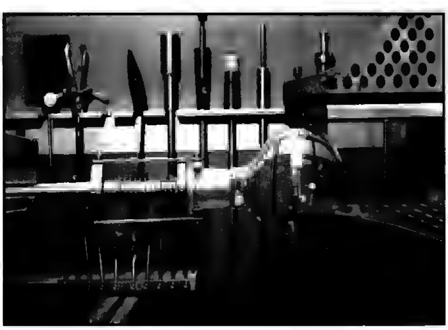
Support tool for making "rotavap" traps.

The tool below is used to support the inner piece of a "rotavaptrap" splash guard. It can be easily adjusted to match the different lengths of the inner piece.

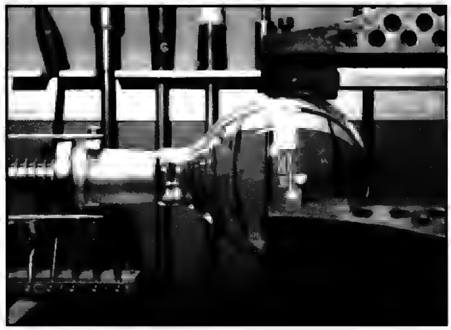
Ready to be welded in place.



I experimented with a wood support shaped exactly like a 250ml flask.



Since wood is softer than metal, I find it more suited to quick closing of the chuck: one can apply more pressure without taking the risk of crushing the flask.

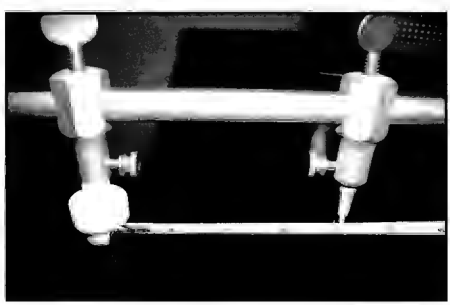


SOME NEW TOOLS PART 3

A simple tool to make repeating cuts easy. This simple-to-build tool will allow one to cut sections of glass fast and accurately without having to measure each time.

Fast adjustment

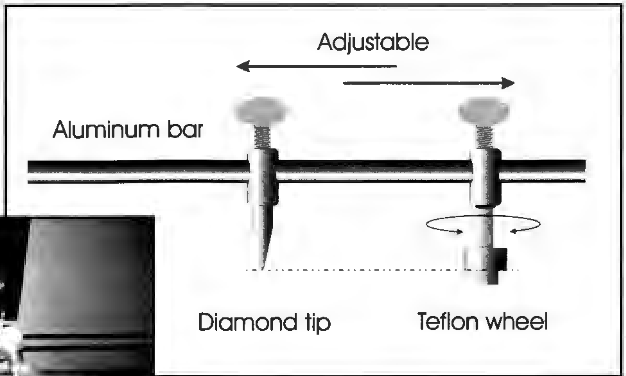
One only needs to rest it against a ruler and set the two screws to adjust the space between the teflon wheel and the diamond tip.



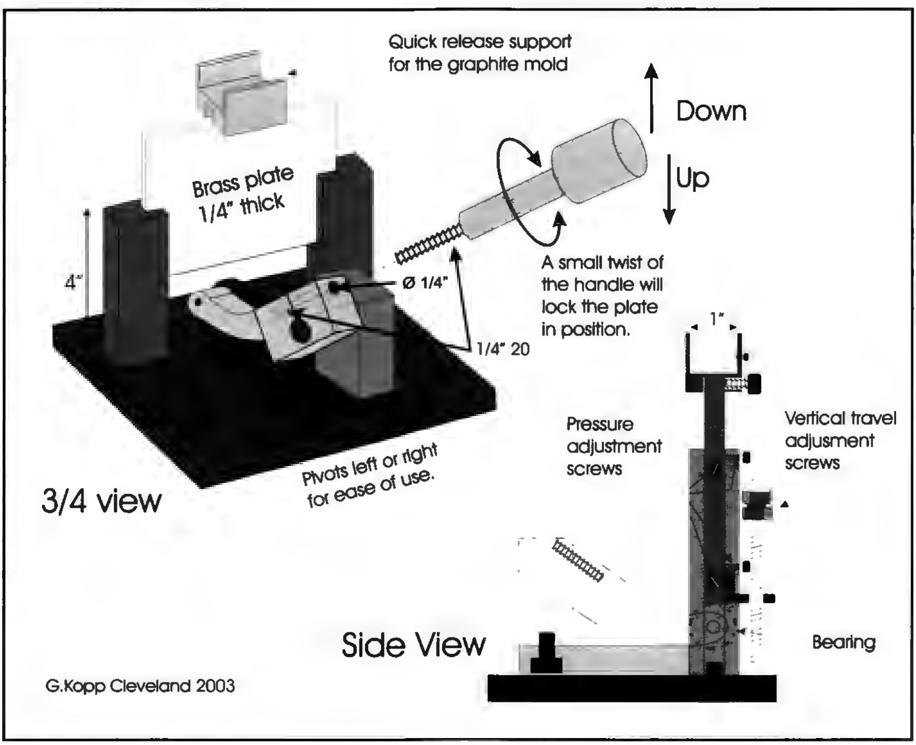
Very easy to use and fast.

The teflon wheel will run against the rotating tube, and a slight pressure on the diamond tip will score the tube at the exact place.

An automatic diamond cutter.



The next drawing illustrates a simple home-made, "up and down" fixture that I use for shaping glass on the lathe. It can be locked in place at any height with one small turn of the handle, and the pivoting mechanism will enable it to be used on the right or the left side of the tool depending on the need. It was entirely made with bits and pieces recycled from previous uses. A removable graphite tool support, makes changing shapes very fast.



2003 Technical Posters

Steve Anderson – "Cardiac Muscle Physiology Tissue Bath"
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David Beaubien – "The Edison Incandescent Lamp Project"
Michigan Glassblowing
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David Beaubien@aol.com

Ronald Bihler – "Comparison of CNC and Sonic Drilling"
Technical Glass, Inc.
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Doni Hatz – "Low Form Constant Temperature Bath"
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hatz.dj@pg.com

James R. Hodgson – "Liquid/Liquid Extractor"
Kansas State University
Department of Chemistry
111 Willard Hall
Manhattan, KS 66506
(785) 532-6676
hodgson@ksu.edu

Richard J. Ponton – "Hey You Built That Filter Funnel Backwards!"
Proctor & Gamble Company
Cincinnati, OH 45253
(513) 627-2313
ponton.rj@pg.com

Kevin Teaford – "Tailstock Flanging Device"
University of Utah
315 South 1400 East Room 2020
Salt Lake City, UT 84112
(801) 581-7592
kteaford@chem.utah.edu

Michael Wheeler – "Modification to the Wheeler 4" Oil Diffusion Pump"
Arizona State University
Department of Chemistry and Biochemistry
Tempe, AZ 85287
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mwheeler@asu.edu

2003 Technical Workshops

Charley Amling – *University of Arizona*
"Bending 2" Opaque Quartz Tubing"

Tracy Drier – *University of Wisconsin-Madison*
"Quick and Easy Thru Seal"

Joseph S. Gregar – *Argonne National Laboratory*
"Resizing Borosilicate Tubing"

Doni Hatz – *Proctor and Gamble Company*
"Klein (bottle) Goblet"

Tim Henthorne – *Ohio State University*
"Cutting Angles"

James Merritt – *University of Southern California*
"Silvering Solutions: A Demonstration"

Kenneth Owens – *University of Missouri-St. Louis*
"The Fundamentals of Giving a Scientific Glassblowing Demonstration"

Kevin Teaford – *University of Utah*
"Use of a Lathe Flanging Device"

The video versions of these Technical Workshops are in the ASGS video library and are available to be loaned out.

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Inge Gunther
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