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Fabricating HD-2(Fe) Type Gas Proportional Counters: A Practical Guide to Techniques, Procedures and Operation

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Fabricating HD-2(Fe) Type Gas Proportional Counters: A Practical Guide to Techniques, Procedures and Operation

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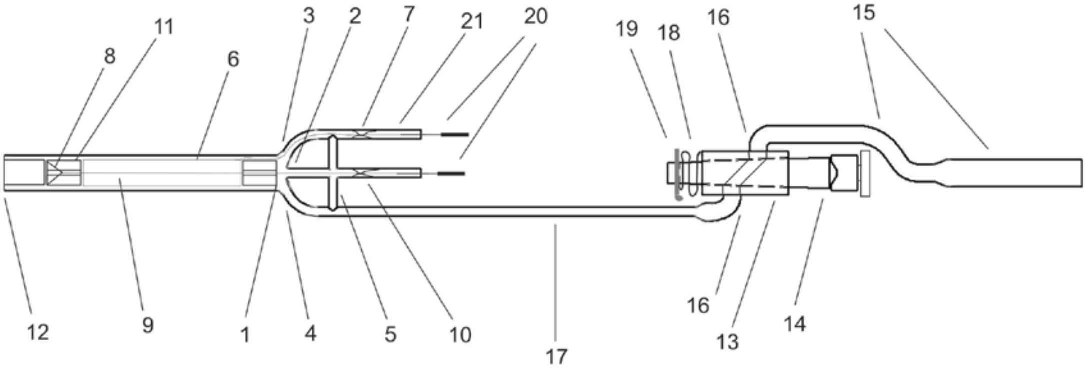
Rebecca Blank, Acting Secretary

National Institute of Standards and Technology
Patrick D. Gallagher, Under Secretary of Commerce for Standards and Technology and Director

EXECUTIVE SUMMARY

Over the last eight years, gas (filled) proportional counters (GPCs) of the HD-2(Fe) type (Davis et al., 1968, 1972; Wink et al., 1993) have been fabricated at NIST through a collaboration of the Materials Measurement Science Division, Facility for Low-Level Counting (*llc*), and the Fabrication Technology Division, Glassblowing and Optical Shops. This type of GPC has been used in our laboratory to detect low levels of (beta) radioactivity by internal gas counting of small atmospheric gas and aerosol samples (Currie, 2008). In the initial phase of the project, reverse engineering was used to develop robust procedures and techniques to fabricate, assemble and test counters. Counters were assembled by a team consisting of an expert technical glassblower to fuse together precision ground synthetic quartz components, an expert optical technician to optically seal tubes to the stopcock and lap the stopcock plug to precisely match the taper and roughness of the barrel, and a research scientist to prepare and assemble the internal components, i.e. inserting the cathode with its connecting electrical lead and stringing the anode wire, both leads requiring a final hermetic seal made by collapsing quartz tubing around each wire. Each GPC was certified for its performance based on an energy calibration as a function of high voltage and its background count rate using a specialized individual pulse digitizing counting system at the NIST *llc* Facility. To date, several GPCs have been fabricated of which few have required restoration because of accidental damages, malfunctions or simple electrical repairs.

The purpose of this document is to describe in detail the process by which miniature GPCs are fabricated and assembled (see photo and diagram on the next page). The process begins by precision grinding four components from high-purity (HP) synthetic fused silica (quartz) rod (Suprasil) to specified dimensions and tolerances: the body (shell), the front plug, the rear plug and the window. These materials are thoroughly cleaned with detergent, HP acids and quartz distilled water before assembled. The counter cathode is made of HP iron (Fe) foil that is acid cleaned and rolled to a cylinder to line the counter inner wall. Quartz components are sealed together with a H_2/



1. Front plug seal
2. Anode tube seal
3. High voltage tube seal
4. Gas inlet seal
5. Support bridge
6. Fe-foil cathode and W-wire lead
7. Quartz on W wire seal
8. Ni/Cr spring (V-shape) anchor
9. W-wire anode
10. Quartz on W wire seal
11. Rear plug
12. Window seal
13. Stopcock barrel
14. Stopcock plug
15. Gas inlet and connector
16. Optical fuse tubes to stopcock
17. Stopcock seal to counter
18. Stopcock spring
19. Spring anchor
20. Electrodes
21. Ga weld W-wire to electrodes

NIST GPC. Photo shows one of several GPCs made by reverse engineering of the HD-2(Fe) proportional counter (Wink et al., 1993). Diagram illustrates the sequential steps involved in the fabrication process.

O_2 flame, optically inspected for any stress or strain, and vacuum tested for their ability to sustain the HP counting gas for periods of weeks. Connectivity between the electrodes and the cathode and anode leads is established through Ga-welds and is immediately examined for continuity. Prior to sealing to the counter, the stopcock is vacuum-leak tested under static conditions for a period of up to one week. Once assembled, the entire counter envelope is evacuated for several days to remove the slightest bit of moisture that may remain. Acceptance is determined by examining the behavior of each counter under operational conditions and confirming a background count rate that is within the established acceptance criteria (Klouda and Filliben, 2013)

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I. INTRODUCTION

A gas (filled) proportional counter (GPC) is a pulse discharge detector designed for internal samples to detect the decay of beta (particle) emitting radionuclides and to quantify their specific radioactivity. In principle, its design is very simply consisting of a tube with a thin metal (cathode) sleeve, a center (anode) wire, electrodes to connect these elements to counting electronics and a stopcock to establish a vacuum envelope for containing the sample gas. Typically, a GPC is filled with a mixture of sample and counting gas at a pressure of 100 kPa. To detect a negative beta particle, negative high voltage is applied to the cathode with the anode at ground potential. Each electron that collides with a neutral Ar molecule (P10 counting gas; Ar/CH_4, 9:1 volume fraction) forms an ion pair. This process occurs for each free electron and thus forms a cascade of ion pairs referred to as the so-called *Townsend avalanche.* Thus, the pulse produced at the anode is detected, amplified, digitized, stored, and processed retrospectively. For a more detailed discussion of the operational characteristics of a GPC, see for example Knoll (2000). Details of the NIST low-level counting (*llc*) system and measurement process are illustrated in several Figures in Section V.

NIST has fabricated miniature (1 mL) GPCs of cylindrical geometry having the following characteristics: a body made of precision ground quartz (synthetically fused, #1 grade, Suprasil); a rolled foil, EDM machined or sintered iron cathode connected to a 13 μm diameter tungsten (W) wire; an anode of 13 μm diameter W wire; and a quartz grease-type stopcock. Quartz tubes extend from the counter body to accommodate the anode and cathode wire leads. To the front of the counter, each wire is anchored by collapsing the tubing around the wire thus proving a high vacuum (0.1 Pa) hermetic seal capable of sustaining vacuum for periods up to several weeks. Two gold coated electrical connectors provide the means of delivering high voltage to the cathode and detecting the electrical charge distribution (output) from the anode. Typically, the GPC is operated at approximately -930 V on the cathode while millivolt changes are sensed at the anode.

This HD-2(Fe) type gas proportional counter has its origin from the Davis GPC designed for measuring few atoms of ^{37}Ar, a product of the reaction of a solar neutrino on a ^{37}Cl atom (Davis et al., 1968, 1972). Wink had since modified the design to meet the specifications of the GALLEX neutrino program (Wink et al., 1993). Since the NIST approach for filling counters was to be by expansion, the Wink et al., (1993) design was changed accordingly for the following reasons: 1) replace the tapered joint with a straight tube for connection to an o-ring fitting, 2) use of capillary tubing between the stopcock and the counter since a Hg Toepler pump would not be used, and 3) carefully match stopcock ground surfaces and coat with vacuum grease to provide a gas tight seal.

Considering our experience and history in constructing Davis type counters, NIST took on the challenge to reverse engineer the HD-2(Fe) type GPC with little more than the details documented in the literature; slightly more detail provided in a design drawing; and, a few real examples that unfortunately had reached their end. Though we were experienced in building GPCs, the learning curve was steep since the literature includes little detail regarding the fabrication and assembly process.

Fabrication and assembly of these detectors requires individuals skilled and experienced in scientific glassblowing and optical grinding techniques; care and patience with manipulating small objects; and, who have a reasonably good knowledge of vacuum technology. The techniques and procedures are summarized sequentially from the selection of materials, to the fabrication of components, and finally to the assembly of counters. Each procedure is outlined in a stepwise fashion and at times includes a discussion. Lastly, the performance of GPCs is measured by their counting behavior and background count rates; results of which are presented considering the design and operational characteristics of the NIST Low-Level Counting (*llc*) Facility.

II. MATERIALS, COMMERCIAL PARTS AND FABRICATION

2.1 Raw Materials

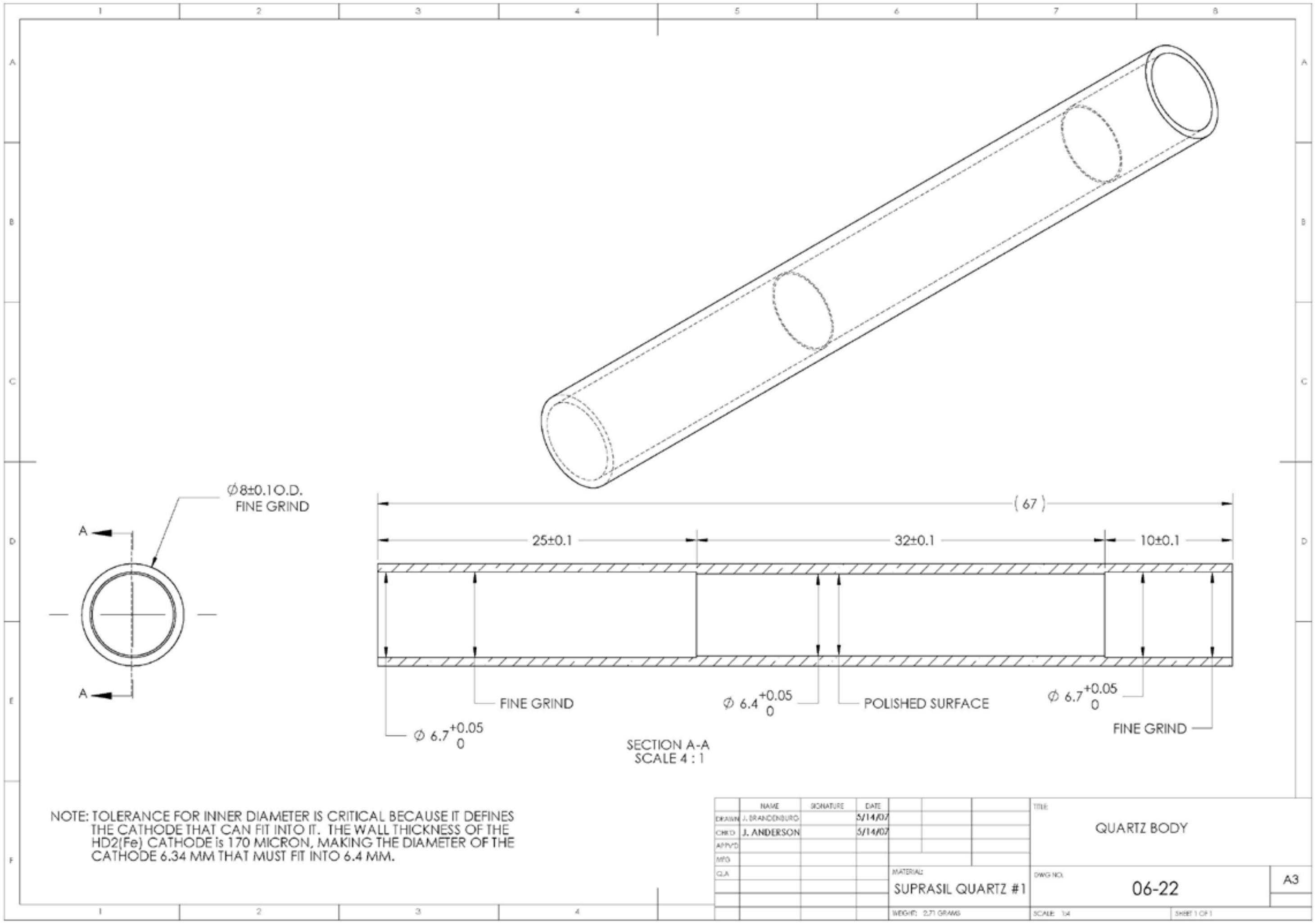
The selection of raw materials is crucial to attaining the lowest possible background count rate of the HD-2(Fe) GPC. Materials of highest purity are naturally sought after; however, chemical and radioactive screening of materials should be an integral part of the process. For the results reported here, materials were identified either by their reported purity or their level of radioactivity as determined by high-purity Ge (HPGe) gamma spectroscopy (Keillor et al, 2009). Since screening materials can be challenging, some materials were considered acceptable simply based on whether or not the ultimate background count rate achieved in the GPCs met program expectations. Wink et al. (1993) has reported on impurity concentrations in various grades of quartz. All counter materials are list below.

- #1 grade Heraeus synthetic quartz rod: 11.5 mm diameter (Wilmad-LabGlass)
- 300 Heraeus synthetic quartz tubing: 0.7 mm inner diameter (ID) x 2.0 mm outer diameter (OD); 0.7 mm ID x 3.2 mm OD; 2.4 mm ID x 4.0 mm OD, and 3.9 mm ID x 6.2 mm OD (Wilmad-LabGlass)
- Gallium (Ga) metal: 7N, 99.99999 % (Rhone-Poulenc); Sigma-Aldrich
- W wire: 13 μm diameter (Midwest Tungsten Service, IL)
- Ni/Cr wire: 0.127 mm diameter (Alfa Aesar)
- Oxygen free high conductivity (OFHC) Cu wire: 0.26 mm dia. (30 gauge)
- High-purity (HP) Fe foil: Purtronic 99.995 %, 0.127 mm thick (Alfa Aesar)
- Low-alpha solder: Kester K100LD, Lead-Free Ultrapure
- Gold-plated electrical connectors: Emerson Connectivity Solutions 40-9856M
- Conductive silver epoxy: EPO-TEK E4110 (EPOXY Technology)
- Epoxy repair putty, FasMetal 10 HVAC 19770 (Devon)
- Cyanoacrylate glue
- HP acids (Seastar Chemicals)
- Quartz distilled water (NIST)

2.2 Quartz Parts and Fabrication

The main components of the HD-2(Fe) GPC are the body, the front plug, the rear plug and the window (slide). These parts are precision ground from HP fused quartz rod to NIST design specifications by Mindrum Precision Inc. (Figures 1-4). The inner section of the body is ground to a smaller diameter than the two outer sections. This internal shelf defines the positioning of the cathode and two plugs. The front plug has a center bore hole for the anode wire and two channels diametrically opposed for accessing the high voltage lead and for providing a gas inlet. The rear plug has a v-channel and center bore hole to position the spring and anchor anode wire. Three bore holes forming an approximate equilateral triangle improve the efficiency for evacuating minute dead volumes and introducing γ-rays for calibration. Since documentation of the Mindrum process is unavailable for reason of protecting intellectual property, Figure 5 illustrates how a rear plug was fabricated at NIST using an ultrasonic mill. The slide is a precision ground tube from which two windows are fabricated. It is cut in half with a diamond grinding wheel where one end of each tube is drawn down to a thin window that allows γ-rays to penetrate yet maintains enough strength to sustain the envelope vacuum. With the rear plug in place, the window should be flush with the rear plug and the open end is sealed to the end of the body (Figure 1, left side). Figure 5 is a display of all the parts prior to assembly.

Quartz 300 grade tubing is used to fabricate three extensions to the front plug and body. One outside tube accommodates a 13 µm diameter high voltage W-wire lead from the cathode to a gold (Au) coated electrical connector. A center tube provides the anchor point on the front of the GPC for a 13 µm diameter W anode wire which is later connected to an Au-coated electrical connector to accept its signal output. The other outside tube connects the GPC body to the stopcock for introducing the counting gas.



NOTE: TOLERANCE FOR INNER DIAMETER IS CRITICAL BECAUSE IT DEFINES THE CATHODE THAT CAN FIT INTO IT. THE WALL THICKNESS OF THE HD2(Fe) CATHODE is 170 MICRON, MAKING THE DIAMETER OF THE CATHODE 6.34 MM THAT MUST FIT INTO 6.4 MM.

	NAME	SIGNATURE	DATE
DRAWN	J. BRANDENBURG		5/14/07
CHK'D	J. ANDERSON		5/14/07
APPV'D			
MFG			
Q.A			

TITLE: QUARTZ BODY

MATERIAL: SUPRASIL QUARTZ #1

DWG NO. 06-22

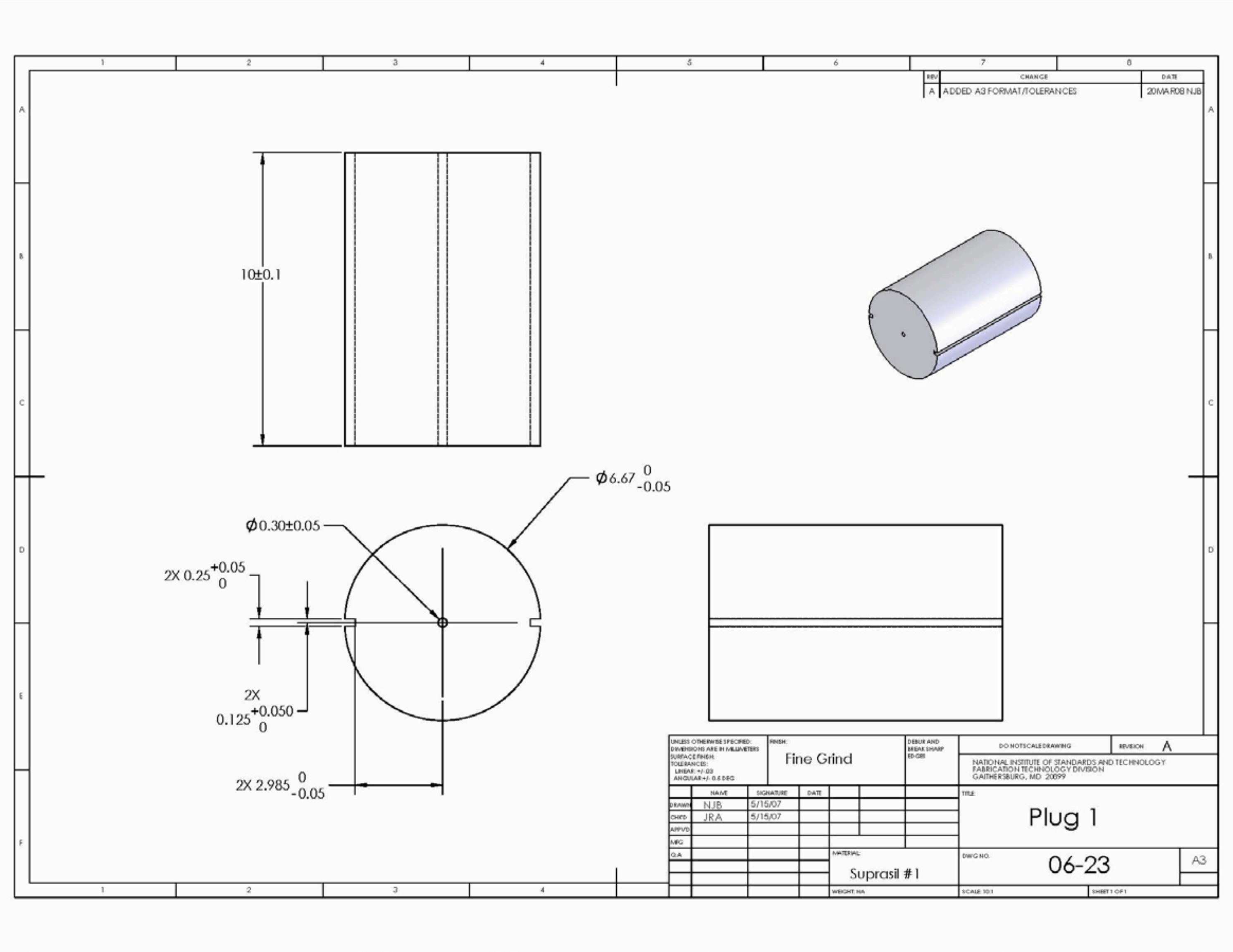
A3

WEIGHT: 2.71 GRAMS

SCALE: 1:4

SHEET 1 OF 1

Figure 1 GPC body. Design specifications to precision grind the body from quartz rod. The internal shelf defines the position of the cathode between the front (right end) and rear (left end) plugs.



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ANGULAR:+/- 0.5 DEG

FINISH: Fine Grind

DEBUR AND BREAK SHARP EDGES

DO NOT SCALE DRAWING

REVISION A

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
FABRICATION TECHNOLOGY DIVISION
GAITHERSBURG, MD 20899

	NAME	SIGNATURE	DATE
DRAWN	NJB	5/15/07	
CHK'D	JRA	5/15/07	
APPV'D			
MFG			
Q.A			

TITLE: Plug 1

MATERIAL: Suprasil #1

DWG NO. 06-23

A3

WEIGHT: NA

SCALE 10:1

SHEET 1 OF 1

Figure 2 GPC front plug. Design specifications to precision grind the front plug from quartz rod. Plug includes a bore hole for the center wire and channels for the high voltage lead and gas inlet.

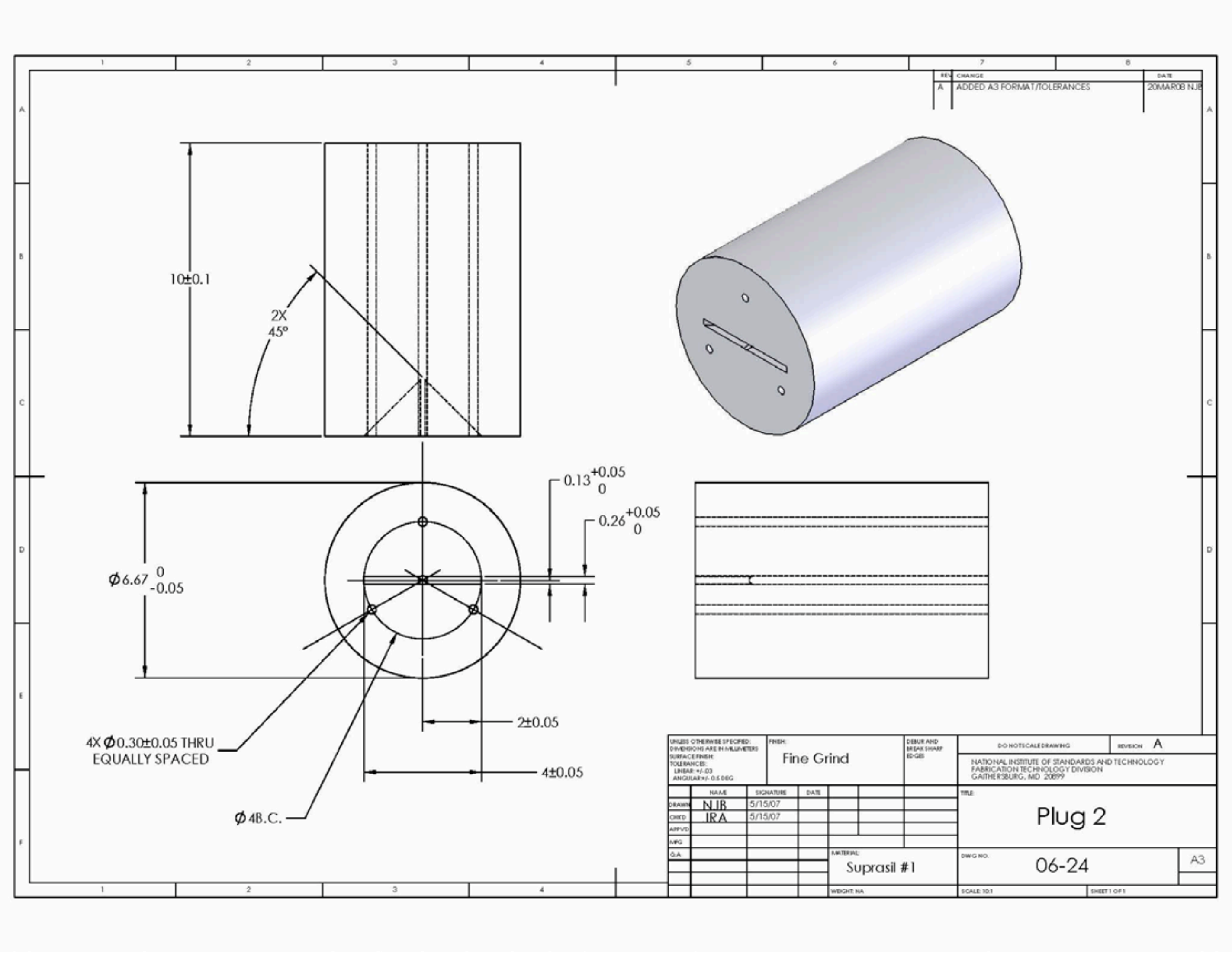


Figure 3 GPC rear plug. Design specifications to precision grind the rear plug from quartz rod. Plug accommodates a spring (in V-channel) to anchor the anode wire and provides bore holes to facilitate evacuation and to calibrate with gamma rays.

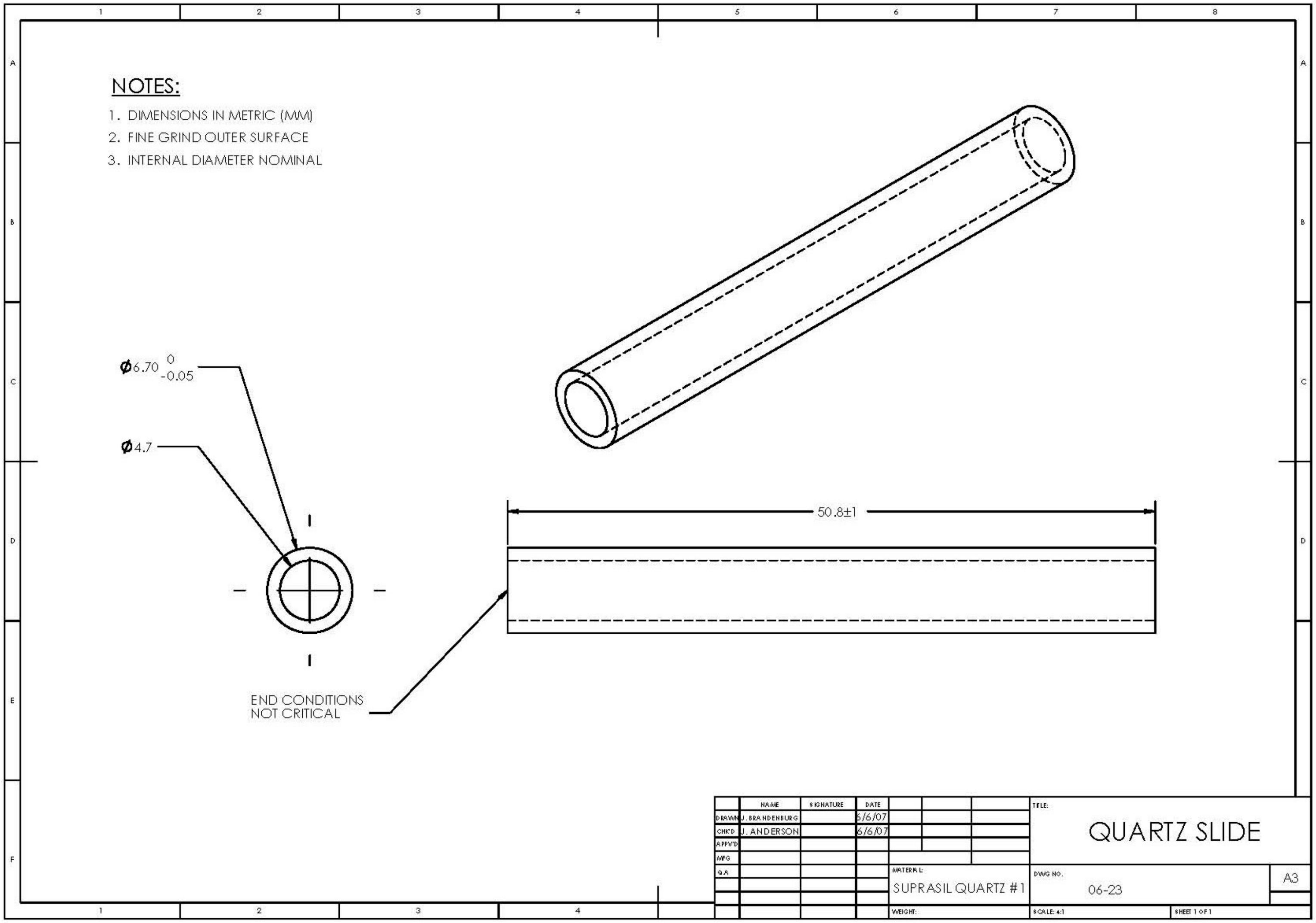


Figure 4 GPC slide for window. Design specifications to precision grind the window slide (cylinder) from quartz rod. Two windows are fabricated from one slide. One end of each half is blown to form a thin window that will sustain vacuum.

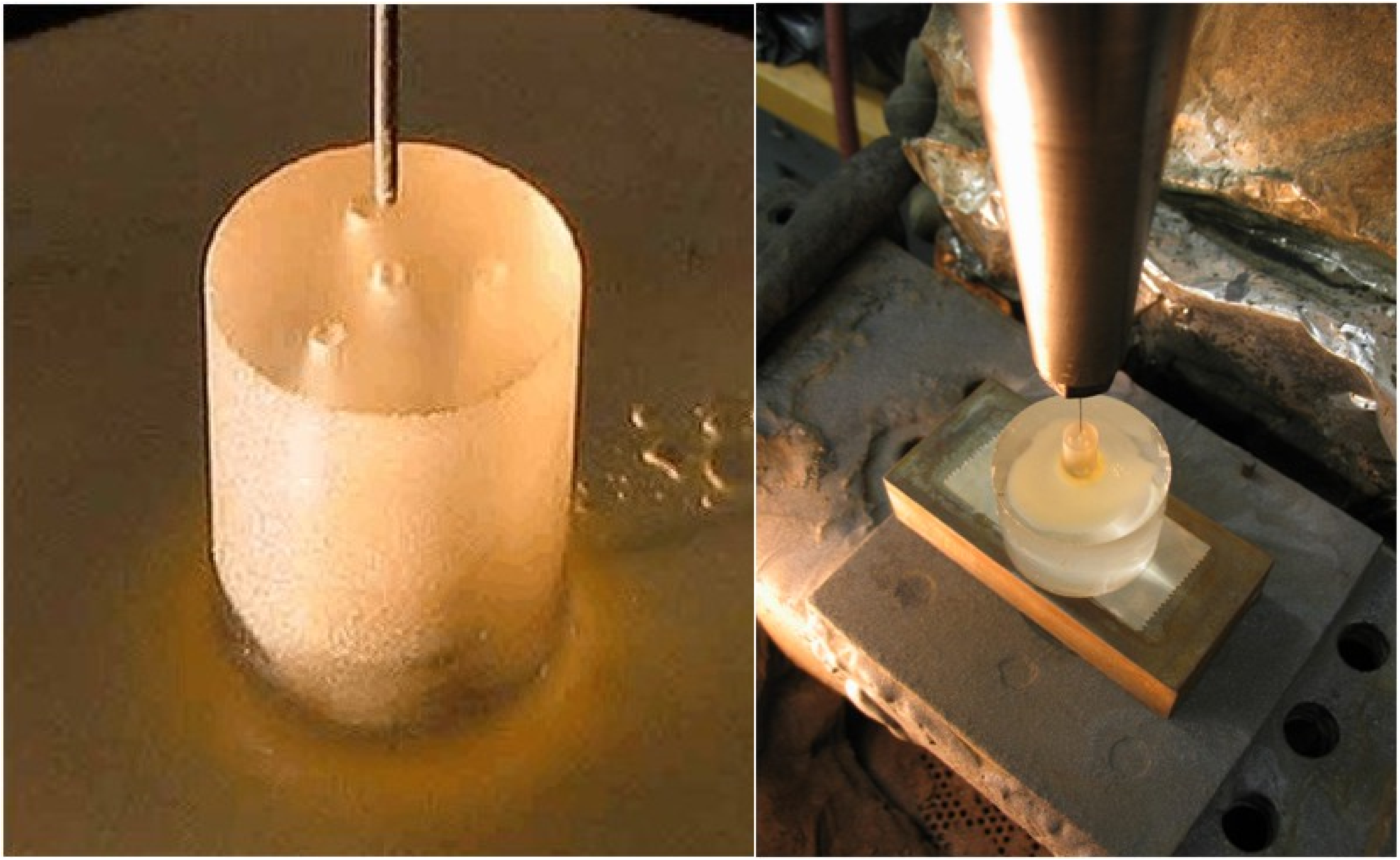


Figure 5 Ultrasonic milling of quartz. A rear plug fabricated at NIST. Bore holes were obtained using an ultrasonic mill.

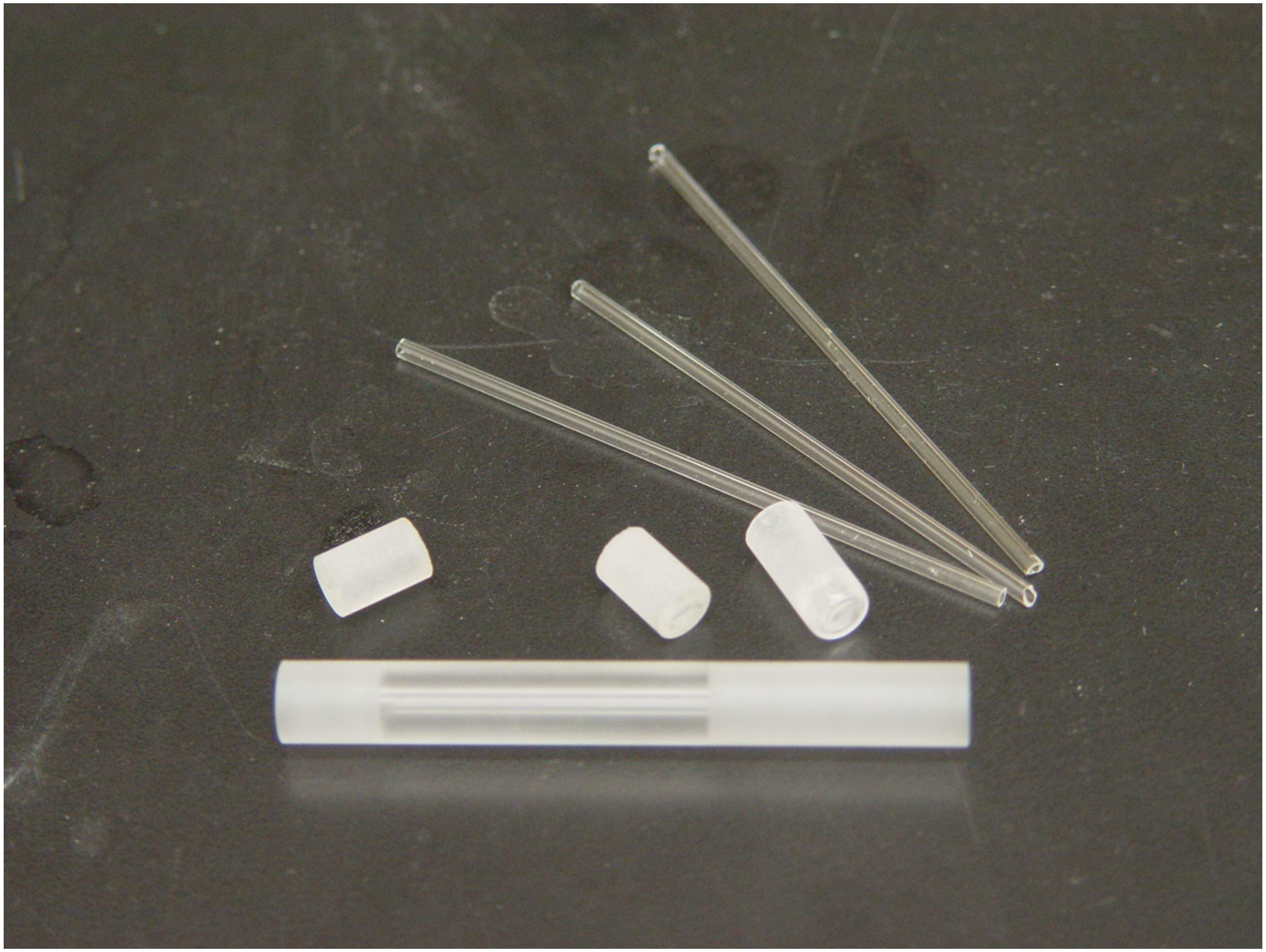


Figure 6 GPC precision-ground quartz components. GPC precision components ground from quartz rod: body (bottom), front plug (left), rear plug (center) and window (right). Window is shown as fabricated from a slide at NIST. Quartz (300) tubing is fused to the front plug and body to accommodate the anode and high voltage electrodes and the gas inlet that connects the stopcock to the GPC body.

2.3 Quartz Stopcock

Quartz stopcocks are purchased from Holm or Mindrum and modified to provide an acceptable seal against gas leakage and diffusion (Figures 7-8). First, each barrel is ground and polished to an optical finish on all four faces in preparation of fusing quartz side-tubes and scribing an identification number. Figure 9 shows how four barrels are simultaneous polished by blocking them on a glass plate using a wax blend. Similarly, tubes are mounted in plaster to grind ends to a polished finish (Figure 10).

Next, the barrel and plug surfaces are lapped to match using 9.5 µm diameter silicon-carbide particles (Figure 11). Extensive lapping of the plug and barrel may require a reduction in the plug diameter at the larger end to allow for a lateral drop in the plug. The degree of roughness is determined empirically through an iterative process of lapping and static-vacuum testing. Care must be taken to avoid over grinding to the extent that the bore-holes can become misaligned or surfaces ground so smooth that grease is forced out after only a few turns of the stopcock. To prevent the former, in the future the barrel specifications will omit the side holes. The holes will be ground at NIST only after the plug and barrel surfaces are matched to meet the vacuum specification.

Tubes are then optically fused to the barrel ports using a natural gas flame (Figure 12-13). Each one is bent to run parallel to the barrel length but in opposite directions while minimizing heat to the barrel (Figure 14). Excessive heat close to the barrel surface may cause a distortion of the inner tapper. A handle is fused to the plug and oriented parallel to the direction of its oblique bore hole.

To test the matting surfaces of the plug and barrel, graphite rod is used to mark the ground surface of the plug and the plug is exercised (rotated) against the surface of the barrel (Figure 14). Any potential path for leakage will appear as a striation on both surfaces. If striations occur, the plug and barrel are then lapped using a somewhat finer grit to make small changes in the surface yet coarse enough to maintain lubrication of the stopcock after several turns of the plug. It may require a few iterations of lapping and testing before the stopcock can provide a vacuum seal against atmospheric pressure (101 kPa). A vacuum tight seal is obtained by coating the plug with Apeizon N vacuum grease.

The final step in preparing the stopcock is to test it for its ability to maintain a hermetic seal for several days at < 0.1 Pa. First, a 6.3 mm outer diameter straight section of quartz tubing is sealed to the outlet tube and configured to follow the inner contour of the cradle as discussed later in the text (Figure 53). Next, a section of 0.7 mm ID quartz is fused to the inlet side of the stopcock and to a 1 mL containment volume similar in size and shape to the GPC body. This assembly allows for testing the stopcock seal under vacuum and also conforms to the cradle cavity (Figure 15). To remove moisture, the assembly is placed in a glass chamber and connected to the cryo-pump for evacuation. The moisture level is periodically checked using a residual gas analyzer (RGA). Once moisture is no longer detected or reaches a steady state, the assembly is removed from the vacuum chamber; the plug is then greased with Apiezon N and fitted with a spring to maintain a good seal.

A static vacuum test is performed by evacuating the volume, closing the stopcock, removing it from the vacuum line and storing it overnight. After a day offline, the assembly is attached back on the vacuum manifold and the intermediate dead volume is pumped out. The stopcock is then opened to a known volume that includes a high accuracy capacitance monometer and the pressure is observed relative to the background vacuum pressure. Any increase in pressure suggests leakage of air into the volume through the stopcock seal. This test is performed at least three times with no sign of leakage before the stopcock is certified for use. Note that the ability to maintain a static vacuum may be a function of the amount of grease present. Too much is likely to plug the oblique bore in the plug and too little is likely to lead to a leaky stopcock and (or) a plug that is difficult to rotate; the latter can easily lead to a broken stopcock.

1. Polish barrel sides to optically fuse inlet and outlet tubes
2. Number the barrel
3. Prepare 2.4 mm ID x 4.0 mm OD flared-ended quartz tubes (20 mm length) to be optically fused to each port on the barrel
4. Dress (lap) barrel with special tool at same angle as plug
5. Grind handle end of plug to a slightly smaller diameter than the barrel-head ID and about 2 mm from the end of the plug
6. Seal handle on plug; handle should be oriented the same for all stopcocks; important to make sure that the plug/handle can be inserted into the barrel when configured to fit the cradle (Figure 15)
7. Fuse 2.4 mm ID x 4.0 mm OD quartz tubing to inlet and outlet ports

8. On the counter side, bend tube at 45 ° with respect to barrel on counter side; seal tube off within a few millimeters of the barrel and far enough away to avoid distorting the barrel
9. Seal a pre-cleaned section of 0.7 mm ID x 3.2 mm OD quartz tubing to the bubble on the counter side; bend tube at barrel and align it parallel to the barrel to conform to cradle configuration. Tube length should extend to the pre-amplifier card edge on the counter side as shown later (Figure 44); its end flared to connect to a containment volume and later to the counter pump-out tube.
10. On the connector side, bend tube parallel to barrel in opposite direction from the inlet and conform to cradle configuration; make sure that the plug/handle can be inserted into the barrel without being obstructed by the tubing
11. Seal a section of pre-cleaned 3.9 mm ID x 6.2 mm OD tubing of the appropriate length to the outlet port side for connection to a 6.2 mm (1/4 inch) o-ring fitting
12. Check stopcock tolerances by checking the fit in a cradle; adjust if necessary to yield the most flexibility fore and aft
13. Ultrasonically clean barrel and plug to remove any grit left behind from the lapping process
14. Seal a quartz container (1 mL) to counter side for static vacuum leak testing
15. Place in chamber to evacuate entire assembly and remove moisture
16. Grease stopcock lightly with Apeizon N
17. Anchor the plug with the spring and cotter pin. Size spring to correct length and tension; cut to fit and bend on cut end to provide a horizontal (flush) contact with the barrel. (The spring anchor is cut from a paper clip to an appropriate length to provide slight tension.) Note: too much tension tends to squeeze vacuum grease out and too little makes it difficult to turn the plug
18. Leak test by pulling vacuum on container, note pressure, close and remove from vacuum manifold for at least one day, and then check vacuum in container
19. Repeat leak test until it passes three consecutive times
20. Cut off container volume at specified length
21. Degrease stopcock using trichloroethylene, ethanol, and distilled water
22. Detergent and acid clean after procedure in Section 2.4

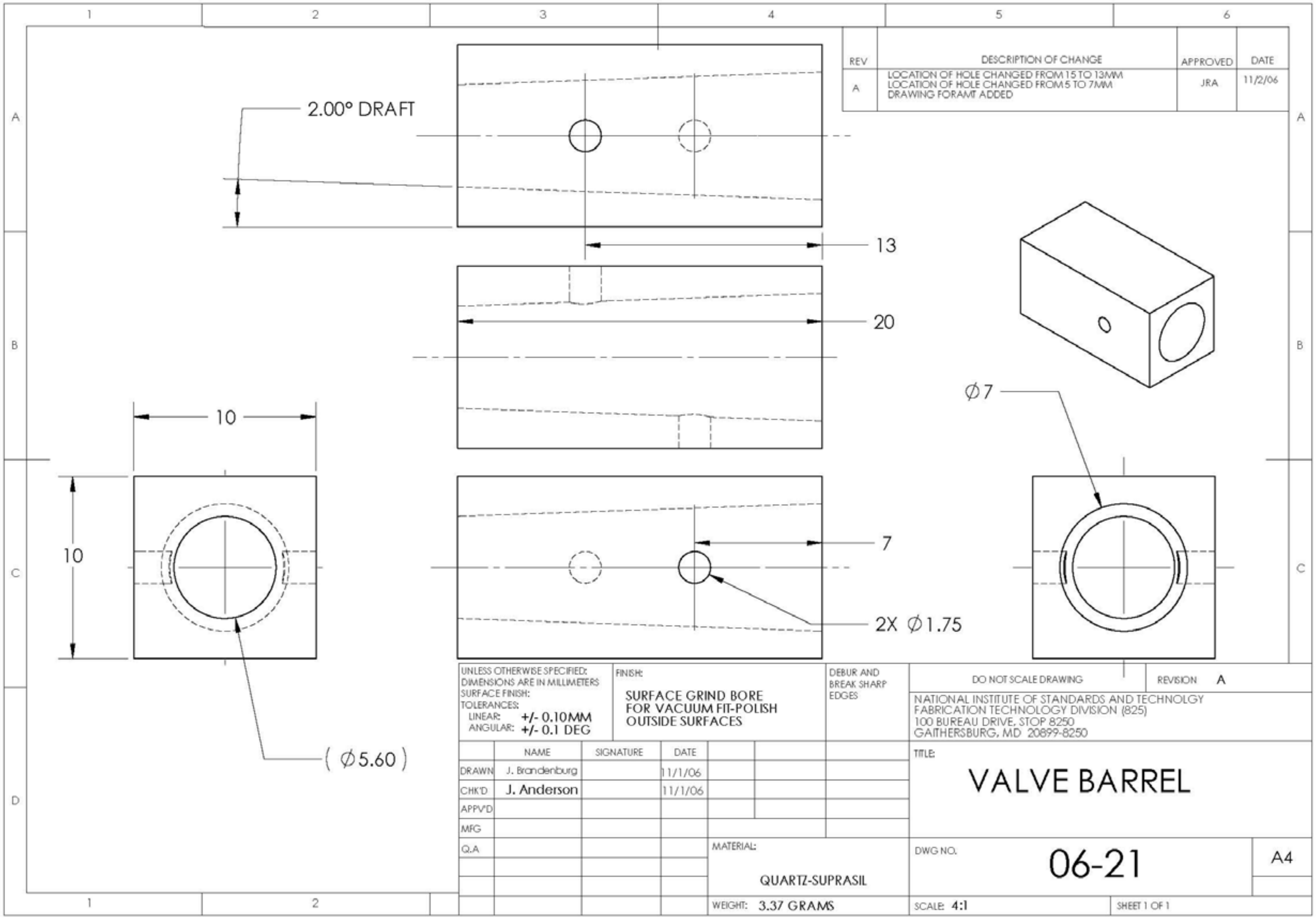
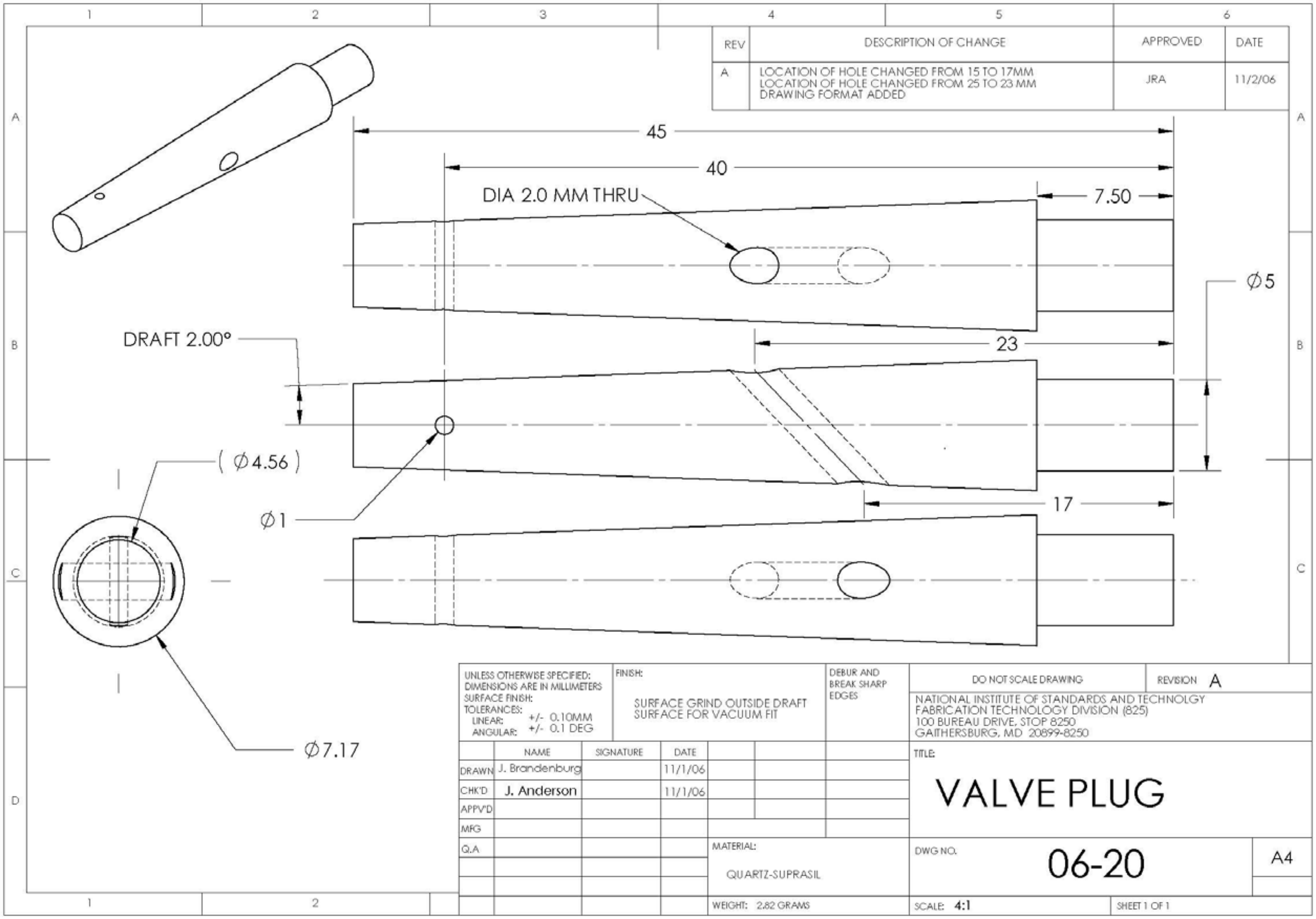


Figure 7 Stopcock barrel. Design specification to precision grind a stopcock barrel from quartz rod.



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DEBUR AND BREAK SHARP EDGES

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CHK'D	J. Anderson		11/1/06
APPV'D			
MFG			
Q.A			

MATERIAL: QUARTZ-SUPRASIL
WEIGHT: 2.82 GRAMS

DO NOT SCALE DRAWING
REVISION A

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLGY
FABRICATION TECHNOLOGY DIVISION (825)
100 BUREAU DRIVE, STOP 8250
GAITHERSBURG, MD 20899-8250

TITLE: VALVE PLUG

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A4
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SHEET 1 OF 1

Figure 8 Stopcock plug. Design specification to precision grind stopcock plug from quartz rod.

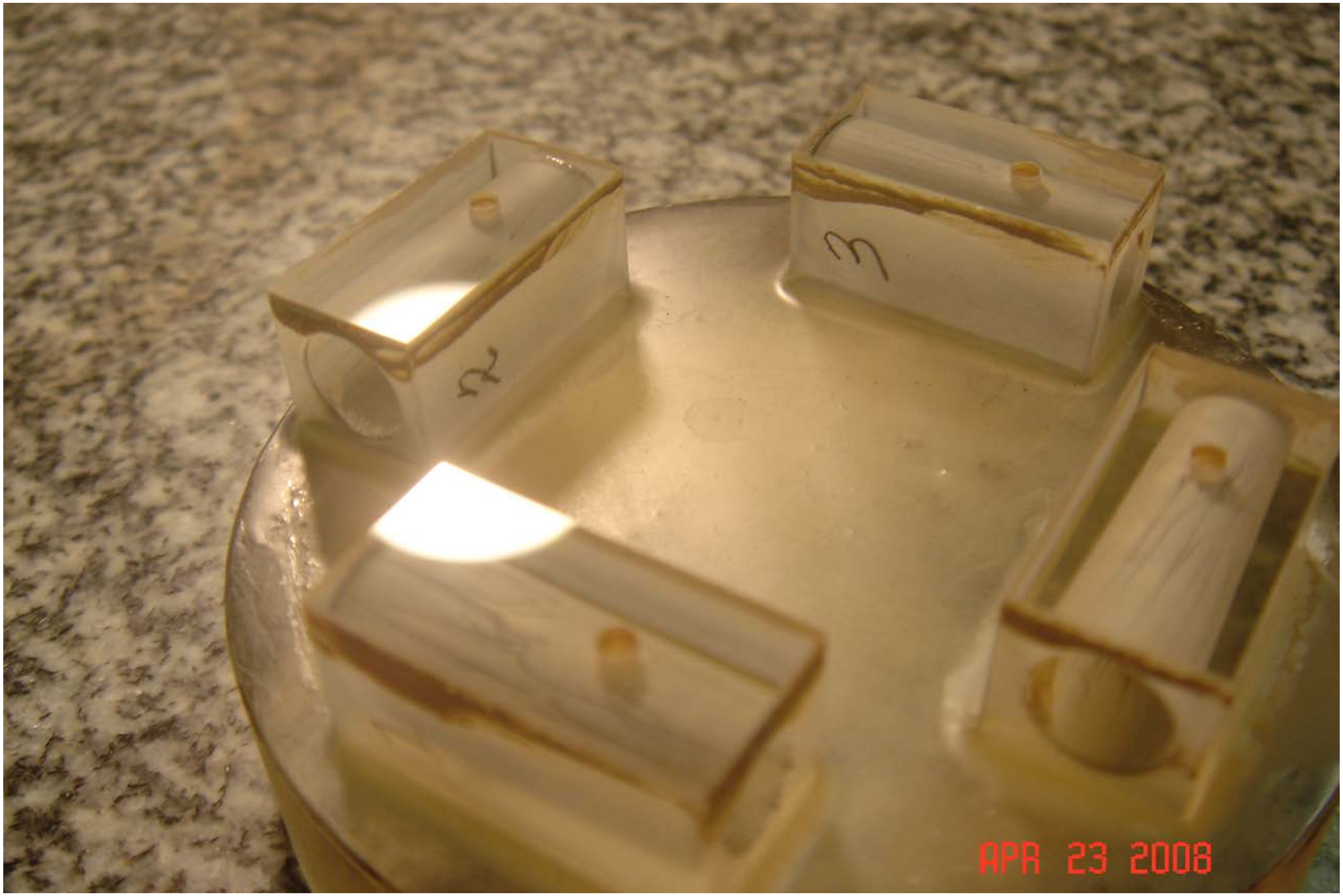


Figure 9 Polishing stopcock barrel faces. Four barrels are mounted to a grinding disc using a mixture of optical rosin and bees wax. Surfaces are ground to an optical finish. All sides are done similarly.


APR 23 2008

Figure 10 Stopcock tubes mounted for grinding and polishing. **S**everal quartz tubes are held in a glass ring using gauging plaster. Tube ends are ground and polished to an optical finish for later fusing to stopcock barrels. The photo shows the tubes being demounted.

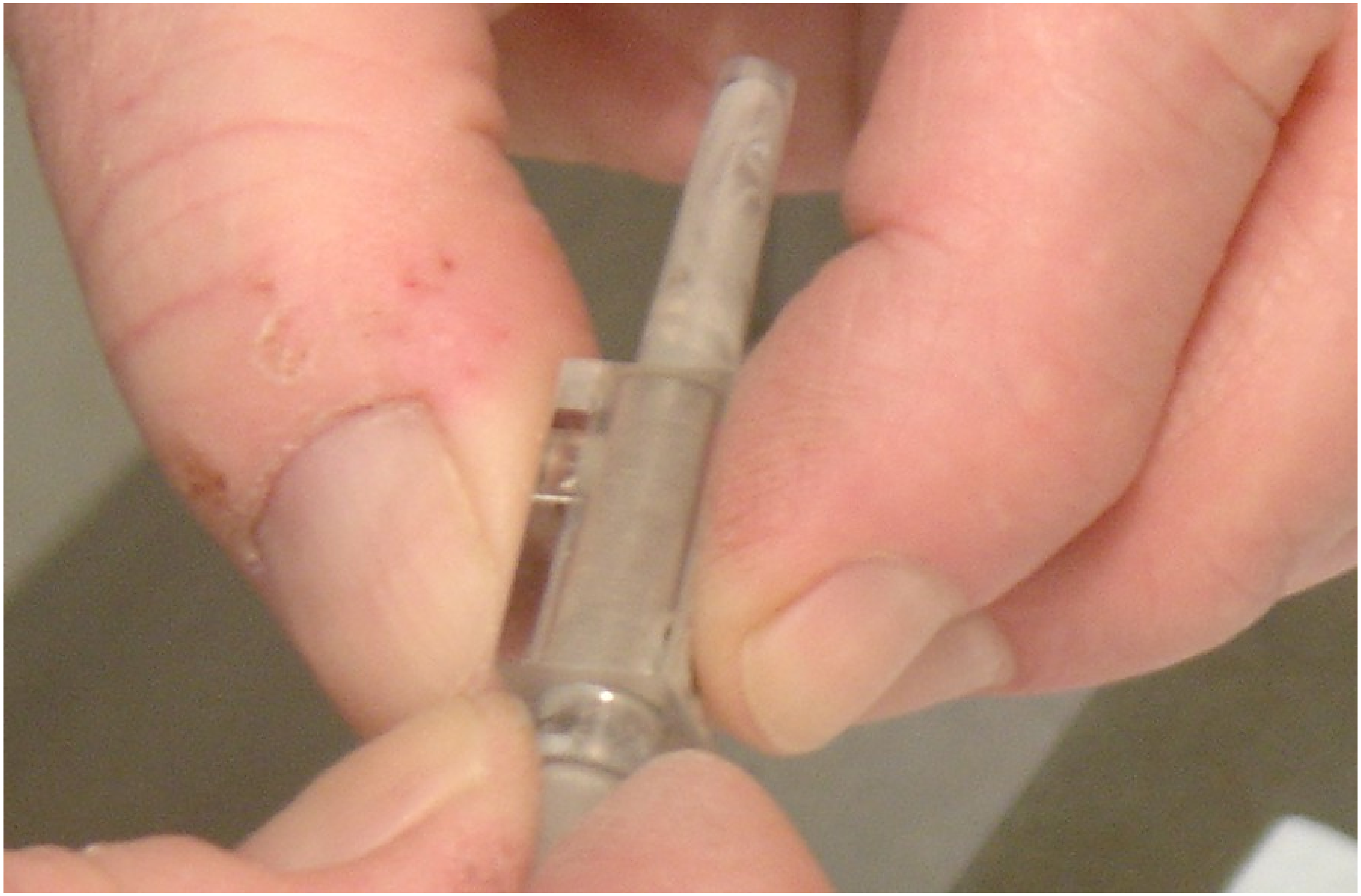


Figure 11 Lapping the stopcock plug and barrel. Each plug and barrel pair is lapped with a 9.5 µm diameter silicon-carbide grinding compound to obtain a surface roughness that provides a vacuum tight (grease) seal against the leakage or diffusion of gases over periods of several weeks. As one laps the two surfaces, any change in taper laterally will need to be offset by grinding the diameter of the handle end of the plug (bottom).

Figure 12 Optical fusing of tubes to a stopcock barrel. Tubes are optically fused to the barrel ports at a much lower temperature than typically used to fuse quartz to minimize distortion. Photo shows a quartz tube aligned with a bore hole on the barrel and held in place. A natural gas flame is uniformly applied to the transition between the tube and barrel.

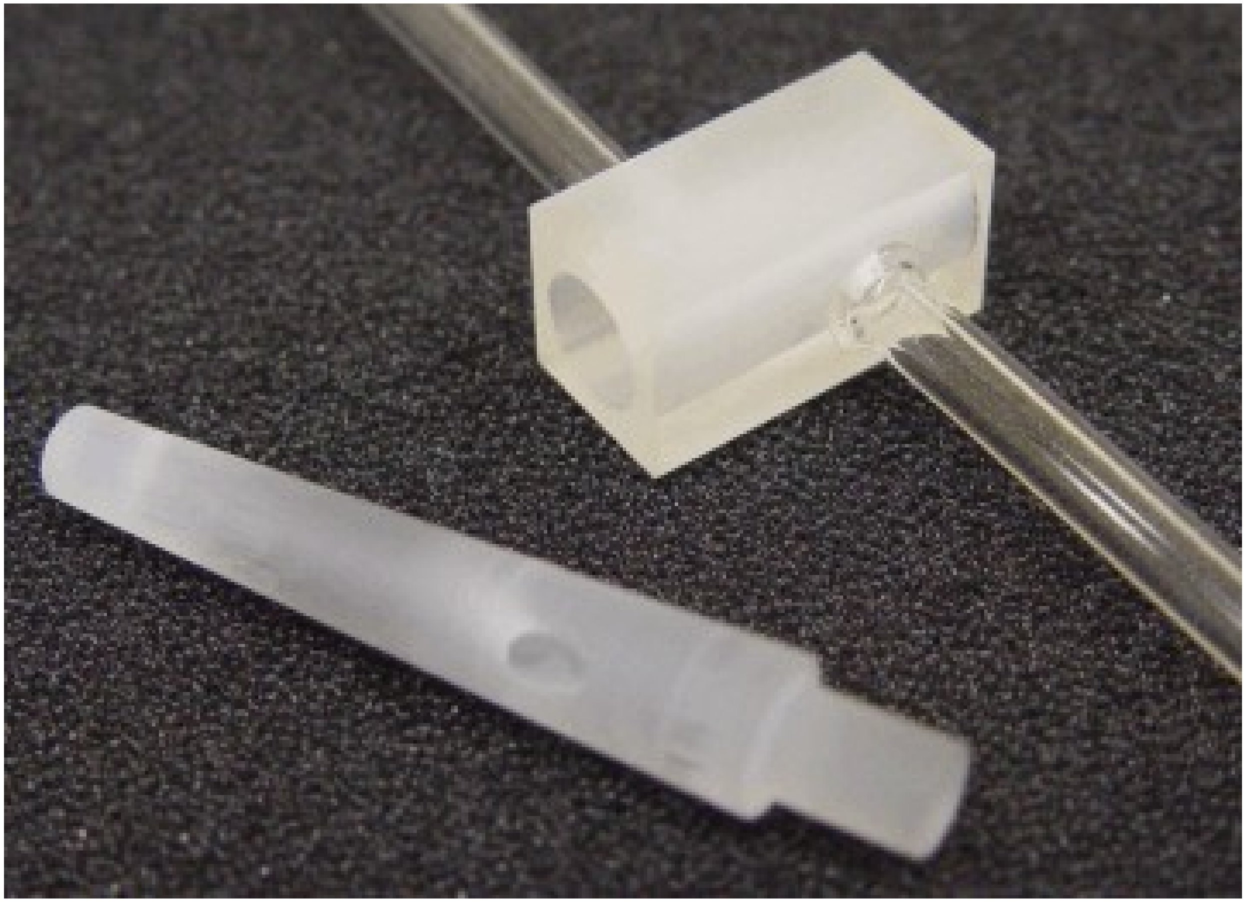


Figure 13 Stopcock plug, barrel and tubes. Apart from the inlet and outlet tubes, the photo shows the stopcock plug (left) and barrel (right) as purchased. Additional preparations are required for the stopcock to be functional, to meet cradle specifications, and to sustain a high vacuum (< 0.1 Pa) over weeks at a time. Modifications performed at NIST include: lapping sealing surfaces, attaching tubes (as shown), grinding back the plug shoulder diameter (right side), sealing a handle (not shown), and bending tubes to fit the GPC/cradle configuration.

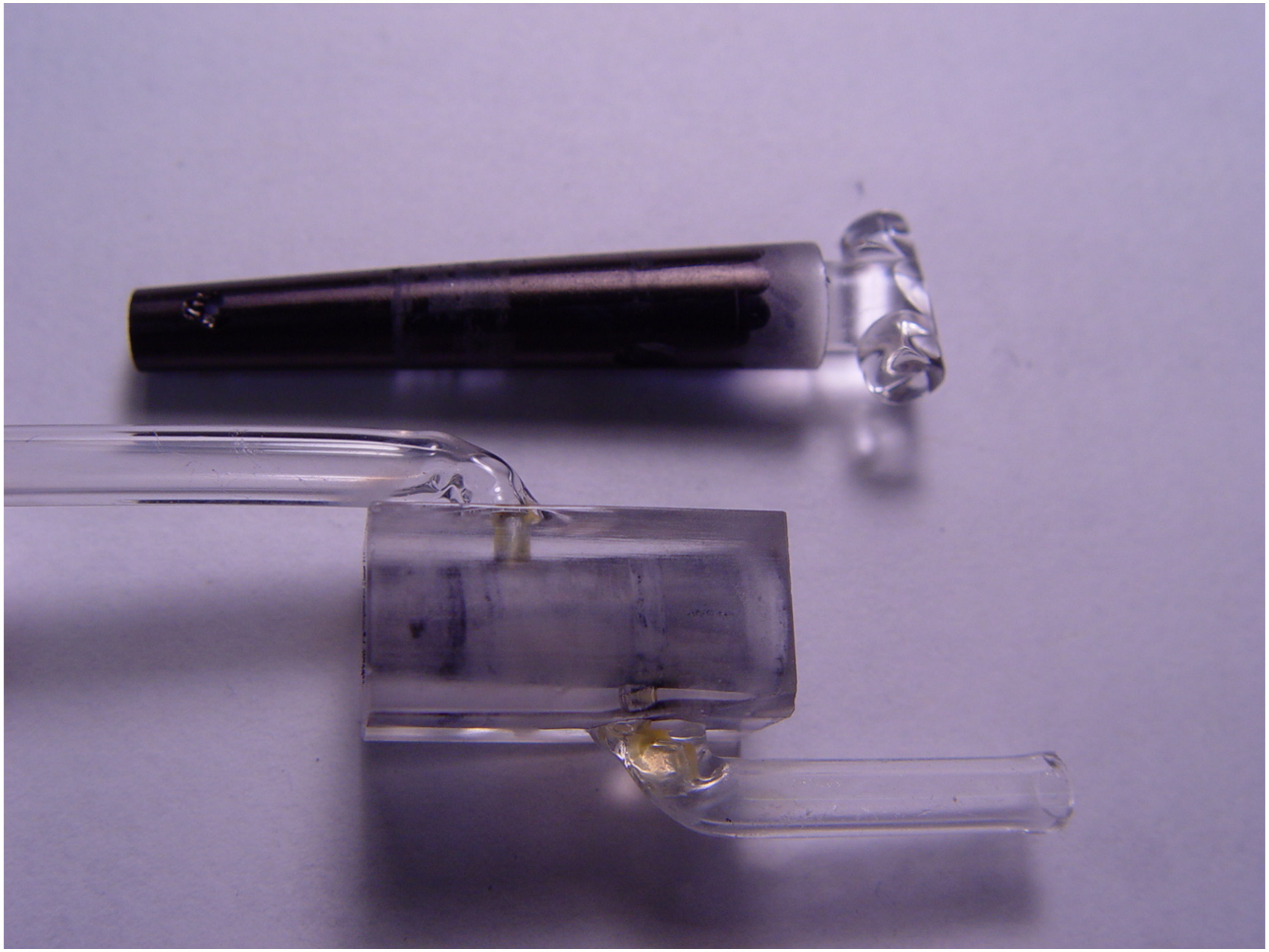


Figure 14 Inspecting stopcock lapped surfaces. Now the tubes are bent and the plug has a handle. The plug and barrel are tested by applying graphite to their surfaces and rotating the surfaces while in contact with each other. Any striations that appear suggest a potential path for gas leakage.

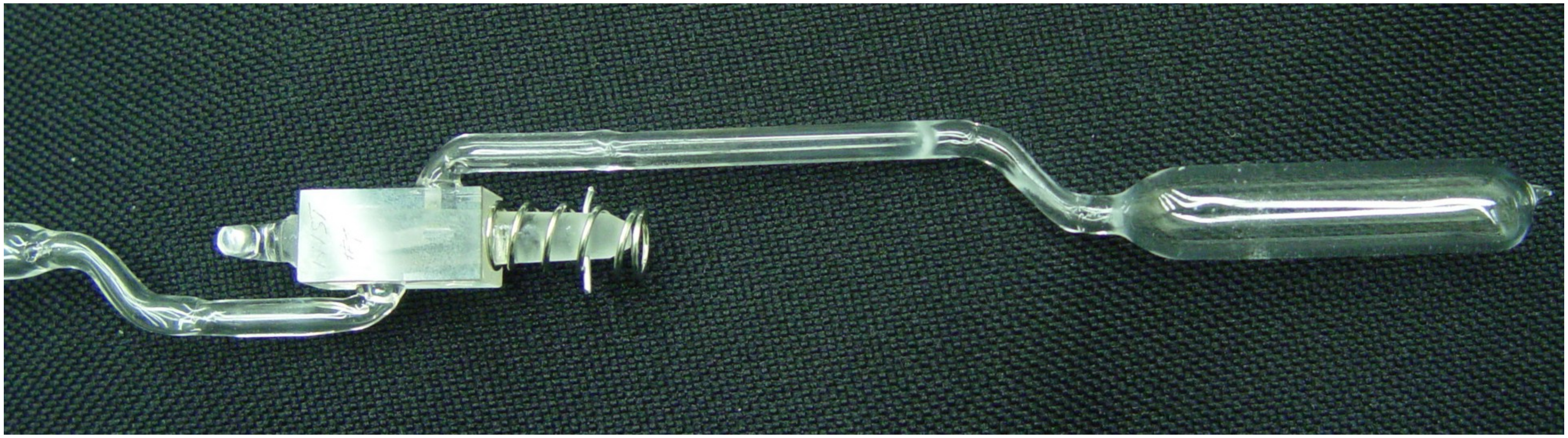


Figure 15 Stopcock static vacuum test. The stopcock is now configured to conform to the cradle and sealed to a 6.2 mm OD straight tube for connecting to an o-ring vacuum fitting (left, only slightly shown). A 1 mL volume is fused to the other tube (right) in a configuration that simulates the position of the GPC when connected. The stopcock is greased with Apiezon N. The plug is anchored with a spring that provides just enough tension to prevent the plug from moving laterally while turning. The static leak test of the system is performed under vacuum and stored off line for periods of several days to a week with no significant change in vacuum.

2.4 Cleaning Quartz

Prior to GPC assembly, quartz parts and stopcock are cleaned to remove surface contaminants that may otherwise contribute to a high background and (or) reduction in counting efficiency (Grootes, 1977). First, parts are soaked overnight in Alconox detergent dissolved in de-ionized water (DW). Next, a regiment of acid soaks each for 10 min in the following order: 4% HF, aqua regia (3:1 volume fraction, 35% HCl and 68% HNO_3, respectively), 10% HNO_3 and 1% HNO_3 with rinses of quartz distilled water (QDW) in between each step. The final rinse with QDW is extremely critical especially since any residue of electronegative species may reduce counting efficiency. To maintain the purity of these acids and QDW, solutions are contained in PTFE (Teflon) bottles and beakers. Parts are then baked out at 170 °C overnight and stored in plastic containers until assembly.

When rebuilding a counter, the window is cut off using a diamond saw blade and the body is reconstructed with new tubes for re-assembly (Section 3.8). In this case, the sub-assembly made up of the counter body, front plug and replacement tubes is rinsed several times only with QDW. Acid rinsing is to be avoided since electronegative chemical residues may remain within the close tolerances of the front plug and body and lower the counting efficiency. Since the presence of trace amounts of water can also affect the counting efficiency, extensive cryopumping must be repeated.

1. Soak in detergent (Alconox) overnight; rinse with QDW
2. High temperature oxidation (700 °C) for 15 min
3. Soak in 4% HF for 10 min, rinse with QDW; rinse with QDW. CAUTION – one must have knowledge of and experience with handling HF. An appropriate ointment such as sodium glueconate must be available in case of contact with skin (see the MSDS for HF)
4. Soak in aqua rega (3:1 volume fraction, HCl and HNO_3, respectively) for 20 min; rinse with QDW. CAUTION – must have knowledge of and experience with the vigorous reaction that takes place when mixing these two highly concentrated acids to form aqua regia
5. Soak in 10% HNO_3 acid for 10 min; rinse with QDW
6. Soak in 1% HNO_3 acid for 10 min; rinse with QDW
7. Dry in oven overnight at 170 °C

2.5 GPC Body Preparations

Before proceeding to assemble a counter, make sure that the front and rear plugs meet tolerance specifications by fitting them in their corresponding ends of the counter body; they should enter with relative ease. Any time clean (internal) counter parts are handled, one must wear particle-free gloves. One source of contamination can be tiny particles and sebaceous material containing ^{14}C. (Also, contaminates from foods rich in potassium will contain ^{40}K.) Check that the front plug is flush or slightly below the body edge. This is the optimum position to make the seal. The plug should slide right out without any assistance other than tilting the body at an angle; however, tolerance between the plug and body should be such that the plug does not rattle.

The GPC body is prepared for assembly by integrating the front plug to the body and attaching three tubes to accommodate the high-voltage (electrical) lead to the cathode, the electrode to the anode wire and the gas inlet (Figure 16). First, mount the body in the chuck of a glassblowing lathe (Figure 17). Place the front plug in the body at the end with the shorter section of larger internal diameter. With a H_2/O_2, seal the edge of the body to the plug about halfway down the plug (Figure 18).

At the high voltage (HV) channel, form a bubble that is slightly to the side of the body edge. The bubble should be as small as possible and positioned on the edge of the plug/body seal, to the side of the body, and in line with the channel from the plug. By creating this bubble, a gradual curved (‘S’ shape) path will be formed once the tube is sealed to ease the threading of a 50 µm diameter W-wire tether. Using a graphite tool, create an indent where the tube is to be sealed. This will be repeated at the other channel to provide a point to connect a tube for the gas inlet. If the path of one channel is not suitable for threading the W-wire tether that will be connected to the cathode lead, then the other opposing channel can be used.

The next step is to seal a quartz capillary tube to the center of the plug to anchor the anode (center) wire. In the other chuck, insert a clean section of quartz tubing (0.7 mm ID, 2.0 mm OD) and check its position relative to the center of the front plug. With the lathe rotating, seal the tube to the front plug (Figure 19).

Next, blow out a channel bubble and seal a tube (0.7 mm ID x 2.0 mm OD) to it offset at a small angle (Figure 20). Diametrically opposed to this tube, seal a third tube of the same dimensions to the other channel for the gas inlet in a similar fashion (Figure 20). Now bend the two outer tubes parallel to the center tube and seal two bridges to give the tubes support. Figure 21 is an example of a completed front section of a GPC. The quartz seals are leak tested by establishing a vacuum in the sealed counter envelope and exposing the outside of the seals one-at-a-time to a Tesla-coil discharge. (Note: the voltage on the coil may be enough to fracture any portion of the seal that is relatively thin.) A leak path is identified when the discharge is direct to a discrete point on the seal (Figure 22). Lay the counter body within the cradle to test for adequate clearance between the two outer tubes and the cradle cavity. An example of the GPC fit within a cradle is shown later (Figure 44).

1. Mount body with plug 1 in the left lathe chuck; attach surgical tubing to rear of body to provide back pressure when necessary
2. Seal front plug to edge of counter body; encapsulate plug except at center bore
3. Create a bubble that is slightly to the side of each channel at the body edge
4. Mount the anode (center) tube in the right lathe chuck; seal the two together; also, seal the end of the tube closed
5. Blow out an outside channel for the HV and seal the tube to it; also, seal the end of the tube
6. Blow out the opposite channel and seal the gas-inlet tube to it; also, seal the end of the tube
7. Loosen the right lathe chuck; back it away from the subassembly
8. Bend one outside tube at the distance from the GPC body specified by the design drawings; repeat for the other outer tube
9. Using a small diameter (1.5 mm) rod fabricated from quartz tubing, seal bridges from the anode tube to the gas inlet and HV tubes for support
10. For quality control purposes, vacuum leak check the sealed plug and tubes by drawing a vacuum on the rear of the GPC body using a mechanical pump with a liquid N_2 (LN_2) trap between it and the GPC while probing the seal with a Tesla-coil; the presence of a leak is apparent if the coil arc (discharge) follows a path through the seal (Figure 22)

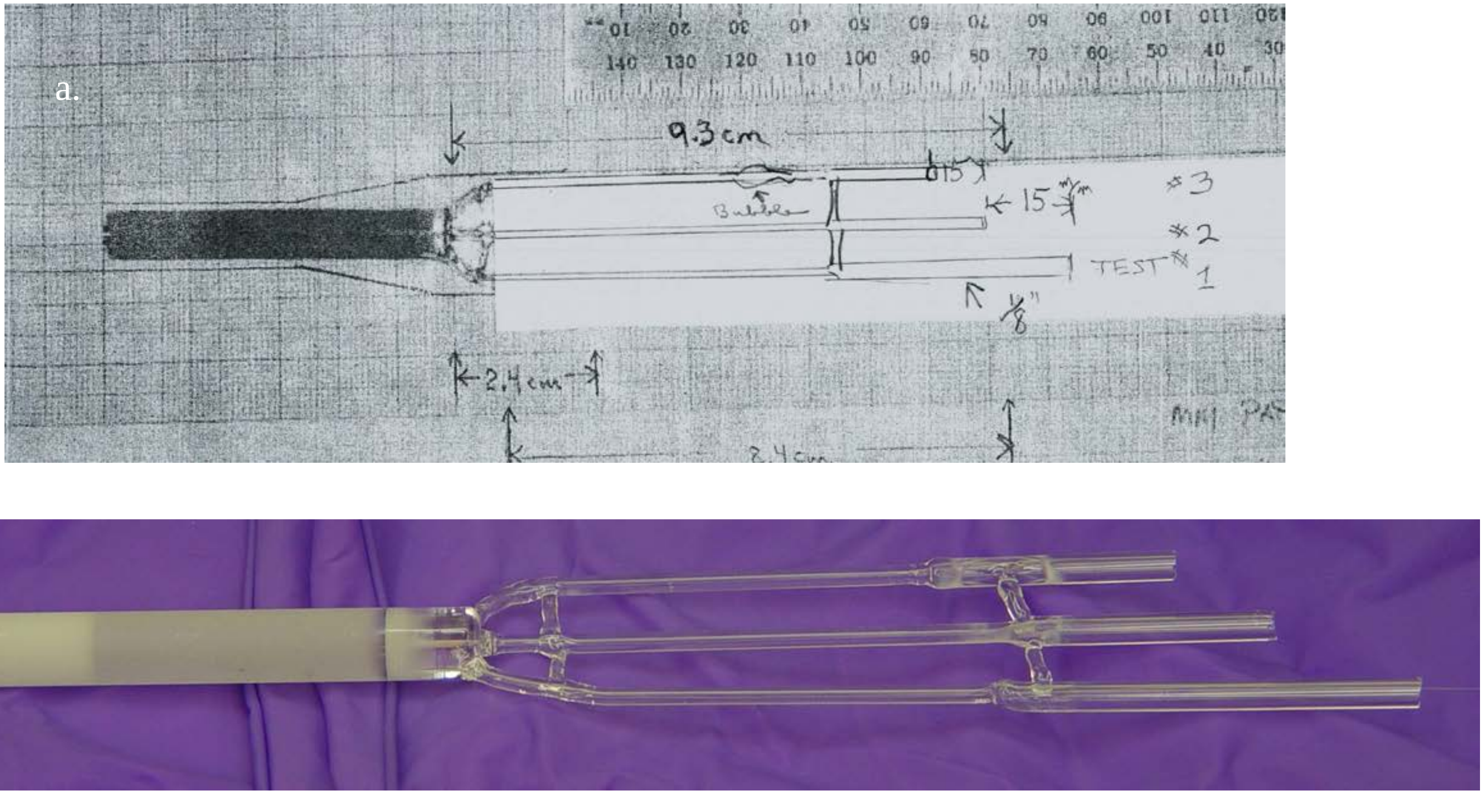


Figure 16 GPC configuration for vacuum leak testing seals. a. GPC dimensions (top) are shown for the counter to conform to the cradle cavity. The top tube (#3) shows a bubble for the seal to connect the stopcock. The three tubes terminate with 3.2 mm outer diameter tubing to connect to an o-ring fitting for vacuum leak testing of each quartz-to-W seal. Tubes are tested in the order stated. **b.** The photo is of an assembled body, front plug, and tubes. Note that the body has been sealed against the plug to minimize virtual leaks. Side channels for the gas inlet and HV have been blown out to the side at the edge of the body. Especially important is a gradual transition from the HV channel to the capillary tube. This facilitates the threading of a 50 µm diameter tether wire through the HV channel and tube that will be used to pull the cathode W-wire lead through the assembly.

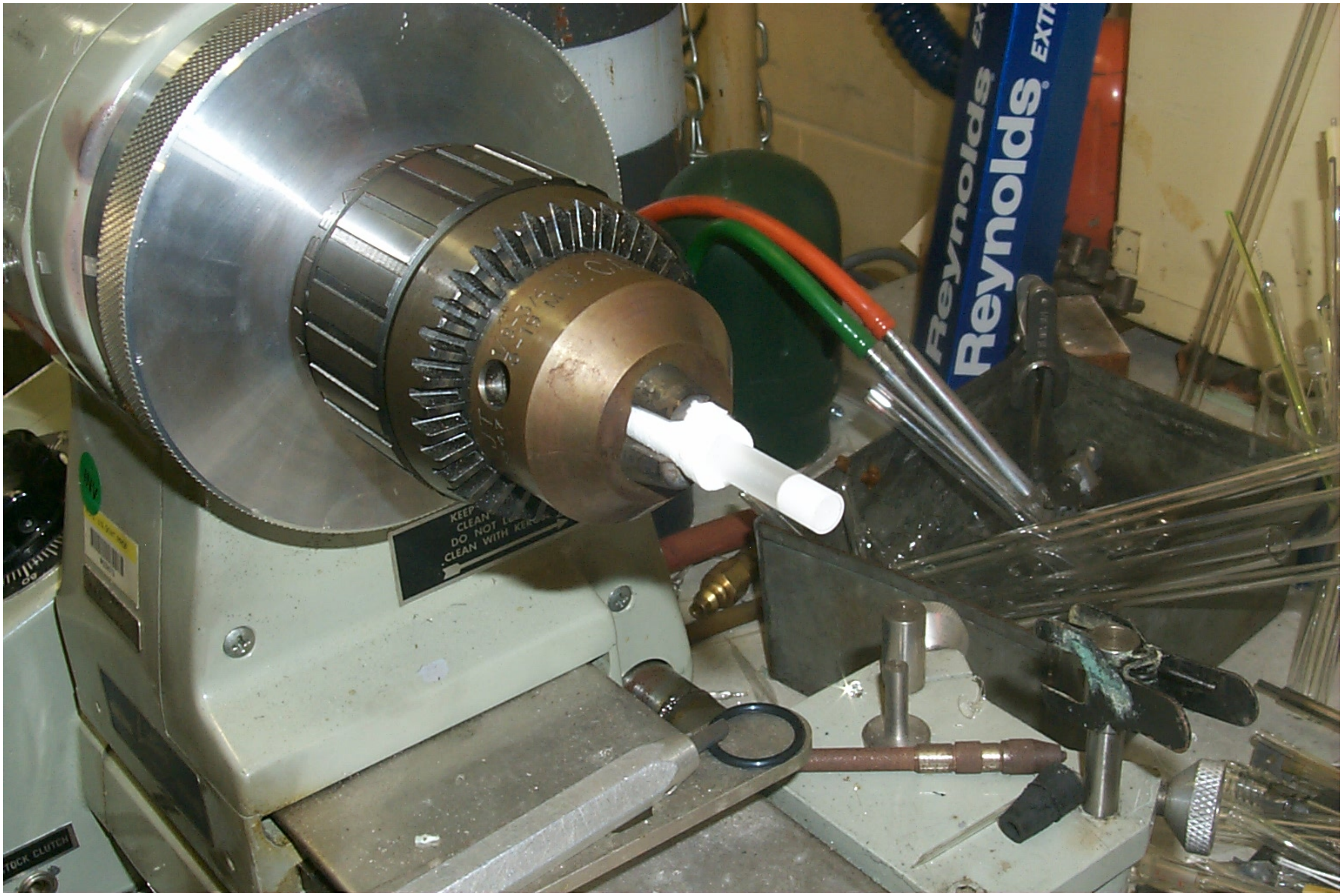


Figure 17 GPC body mounted in a glassblowing lathe. Assembly of a GPC begins with the sealing of the front plug to the end of the body with the short offset (see Figure 1, right end of body).

Figure 18 Sealing the front plug to the GPC body. The glass blower is shown sealing the front plug to the edge of the GPC body using a H_2/O_2 flame.

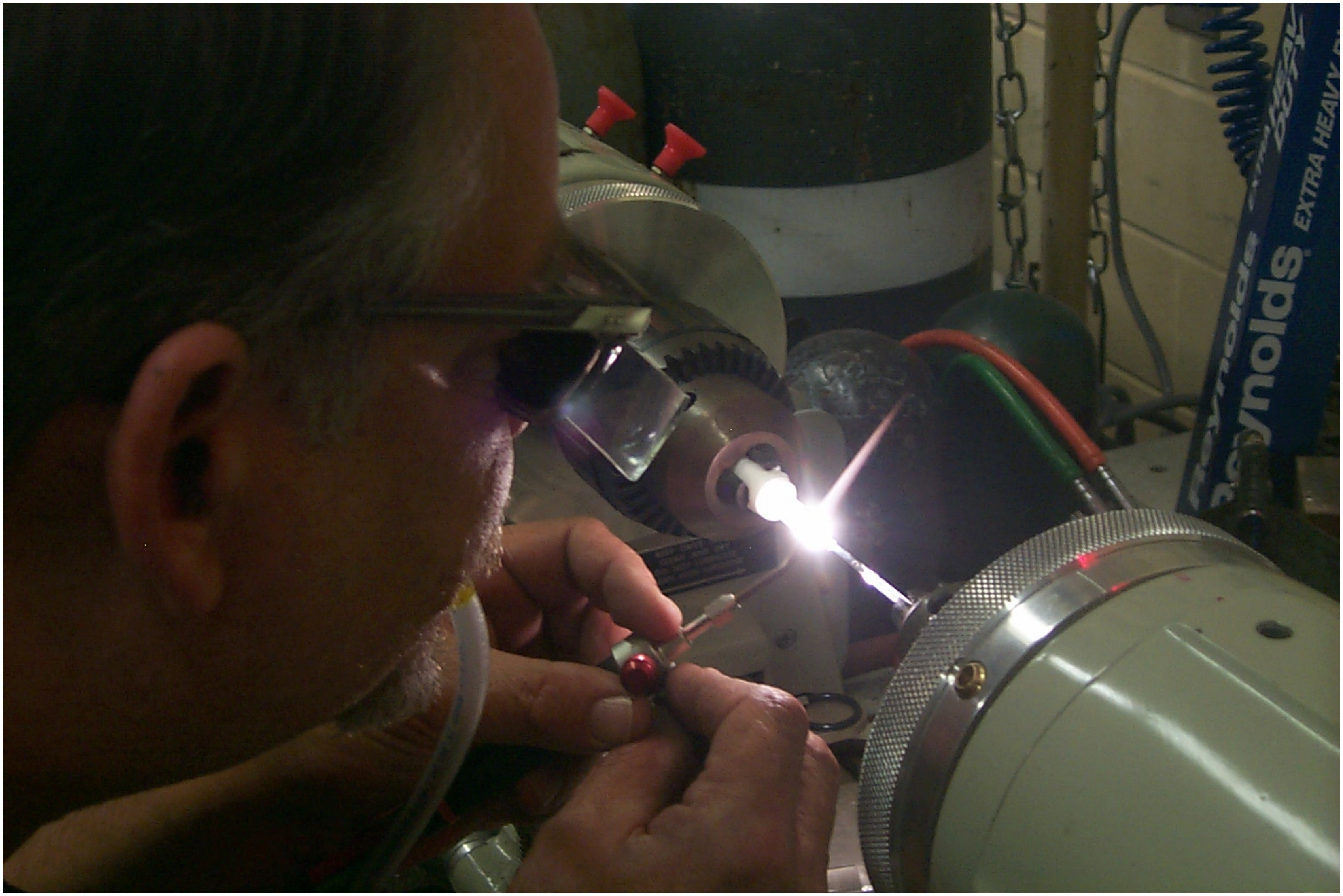


Figure 19 Sealing the center capillary tube to the front plug. The glass blower is shown sealing the center (anode) tube to the front plug. The tube has a 2.0 mm OD and a 0.7 mm ID.

Figure 20 Sealing the outside capillary tubes to the GPC body. The passage way is blown open to accommodate the high voltage lead. A capillary tube of the same dimension as the center tube is sealed at a small angle and in line with this outside channel of the plug (left). The other passage way is blown open to accommodate the gas inlet. A third capillary (same diameter) is sealed to the front plug over the remaining channel for the gas inlet via the stopcock.

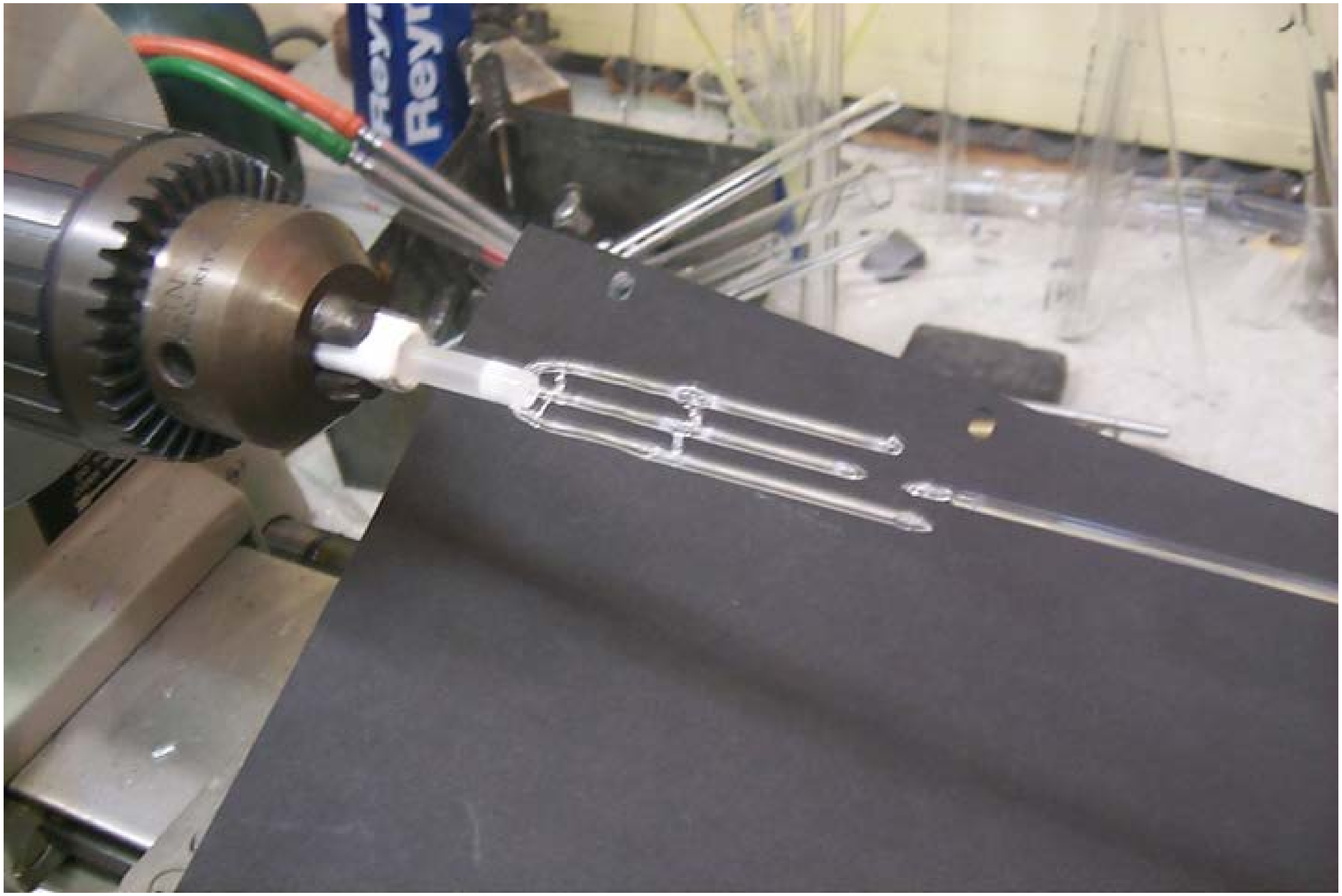


Figure 21 Completed front section of a GPC. The image shows the completed front section of the GPC. A permanent bridge is positioned close to the body. A second bridge to the right is temporary to secure the HV and anode tubes while the quartz-to-W seals are made. Seals are made just to the right of the left bridge by collapsing the tubing around the W-wire anode and cathode leads (shown later; Figures 33 and 38). The top tube has a small bubble just to the left of this bridge to make connection with the capillary tube from the stopcock. This image lacks the 3.8 mm outer diameter tube extensions for vacuum testing.

Figure 22 Leak testing the GPC envelope. A vacuum system and Tesla coil apparatus is used to leak test the seals that connect the body, front plug and tubes together in preparation for counter assembly. The photo shows a cold trap, a glass valve and rubber stopper used to establish a vacuum in the GPC envelope; here simulated by the vertical tube above the stopcock. The GPC is inserted through a hole bored in the center of the rubber stopper. While drawing a rough vacuum (1 Pa) on the GPC, a Tesla coil is positioned near a seal. If a hole exists in the quartz, then the arc discharge will be directed through it from the tip of the Tesla coil (purple arc).

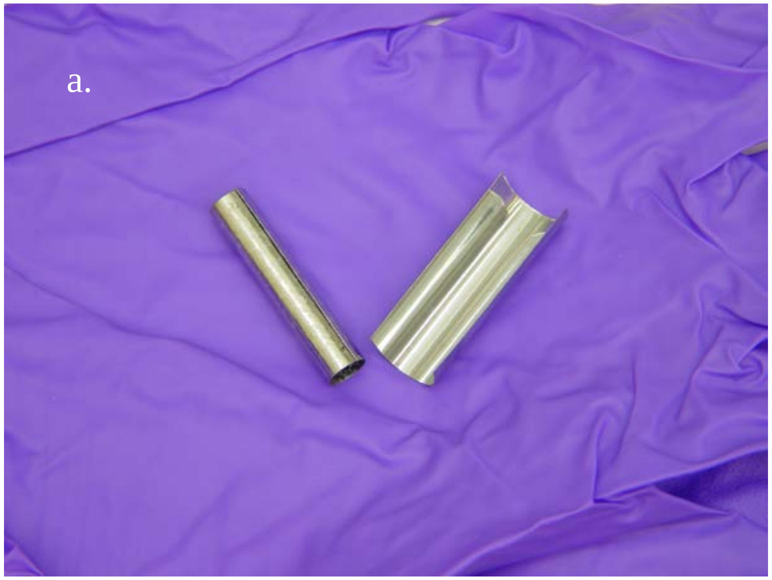
2.6 Cathode Preparation

The GPC cathode is a thin metal liner made of HP Fe foil (99.995 %) that occupies the center (polished) section of the counter body. The foil is 0.127 mm thick and cut to a rectangle nominally 19.75 mm wide and 32.0 mm long. The foil is cleaned with methanol, detergent and 10 % HCl. Using a clean glass rod of appropriate size, roll the foil by hand to a cylinder with a slightly overlapping seam. The seam will naturally spring open until the cylinder is annealed and rolled again. Anneal the Fe cylinder for one hour at 700 °C in H_2. Using a clean glass rod, re-roll the Fe foil with a slight overlap of the edges. The annealing process reduces the spring that normally exists in the Fe foil and thus conforms to the rolled shape. Insert the cylinder into a counter body and roll the cathode against the body using an undersized rod with pressure against the counter body to obtain a friction fit cathode (liner). Remove the cathode and arc weld a 4 mm piece of 50 µm diameter W wire at two points (one third and two thirds) along the wire that is positioned parallel to and close to the cathode seam on one end. Be sure the burr does not extend beyond the end of the cathode (Figures 23-27).

1. Cut Fe foil to specified geometry
2. Rinse foil with QDW
3. Rub Fe foil with a wipe dampened with methanol
4. Dip Fe foil in 10 % HCl; rinse with QDW
5. Rinse with methanol then QDW and wipe dry
6. Roll foil to specified diameter
7. Anneal Fe foil at 1000 °C in H_2
8. Roll Fe foil to a cylinder using specified diameter of clean glass tubing
9. Spot-weld a 4 mm length of 100 µm W wire (burr) near the seam of the Fe foil at one end.
10. Re-roll Fe foil to specification; necessary again since spot welding likely changed the concentricity of the foil cylinder
11. Check Fe cathode fit within precision bore section of body

When reusing a cathode:

12. If needed, spot weld new 50 µm W wire (burr) to cathode adjacent to seam
13. Reduce Fe cathode in H_2 flow at 1000 °C to brighten cathode and weld
14. Allow cathode to cool under H_2 flow
15. If necessary, re-roll cathode for concentricity
16. Store cathode in dry box or container with Ar atmosphere



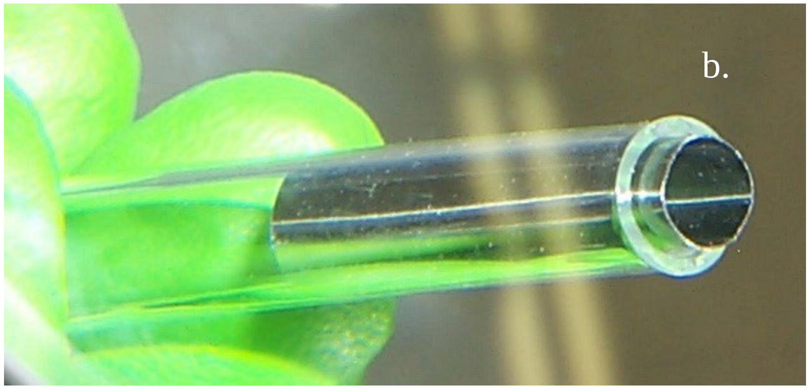


Figure 23 Fabricating cathodes. An Fe-foil cathode is shown here at three stages of shaping: **a.** initial rolled foil (right) with cylinder formed after annealing (left); **b.** rolled cathode friction tight within a tube to simulate a GPC; and,

c. Different materials and methods of forming cathodes have been explored by Wink et al. (1993) and NIST. Examples of these cathodes are shown in the photo. The cathodes at each end are made of silicon and were provided by Max Plank Institute. The second from the left is a Fe-sintered cathode. The center cathode is made of Fe foil treated and rolled as described in the text and photos above. The cathode to its immediate right was cut from solid HP Fe rod using electrical discharge machining (EDM).

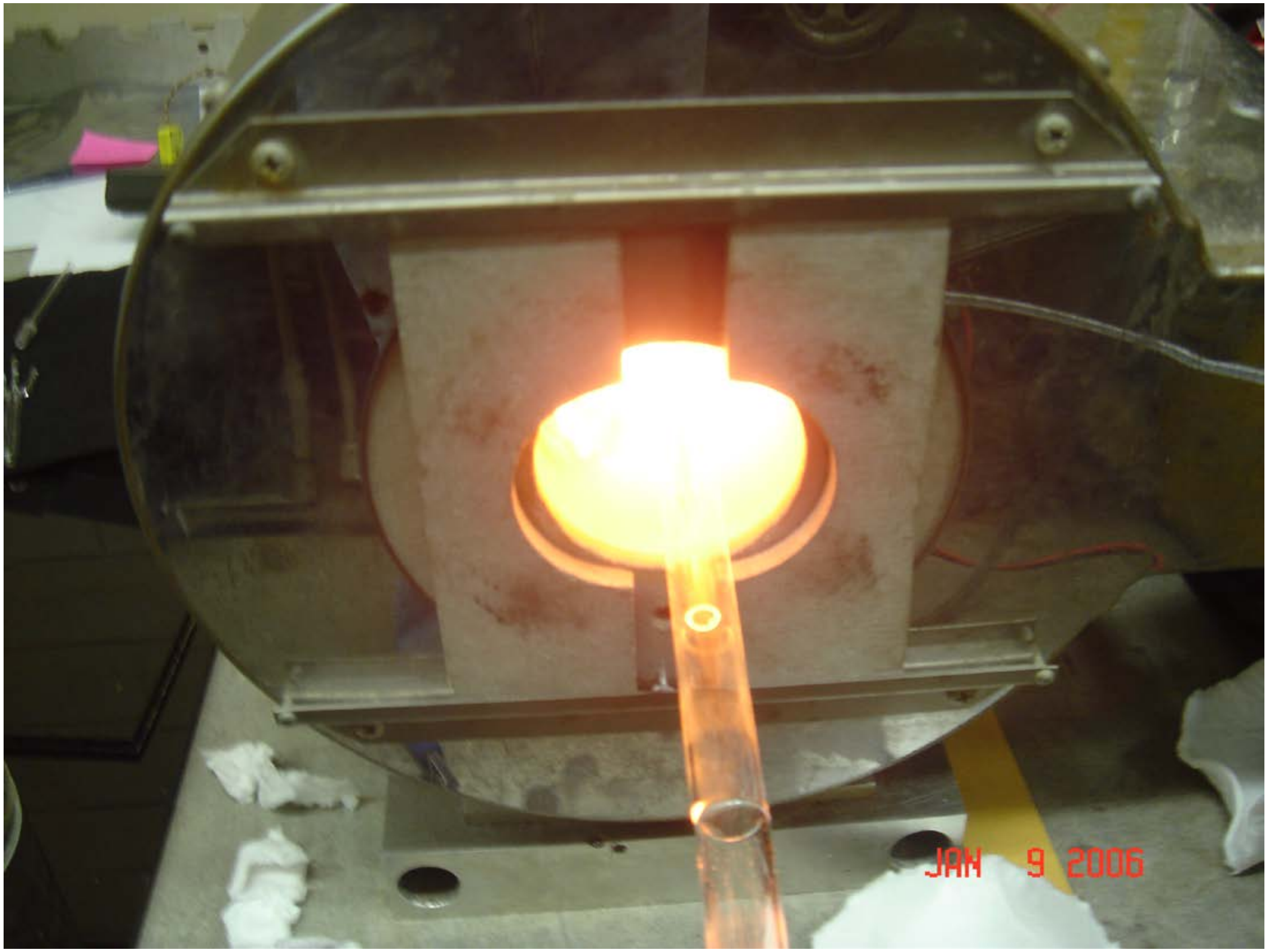


Figure 24 Annealing a Fe-foil cathode. Photo of a rolled HP Fe foil cathode inserted in a quartz tube, purged with H_2 gas, and heated for 1 h at 1050 °C – the annealing temperature of Fe.

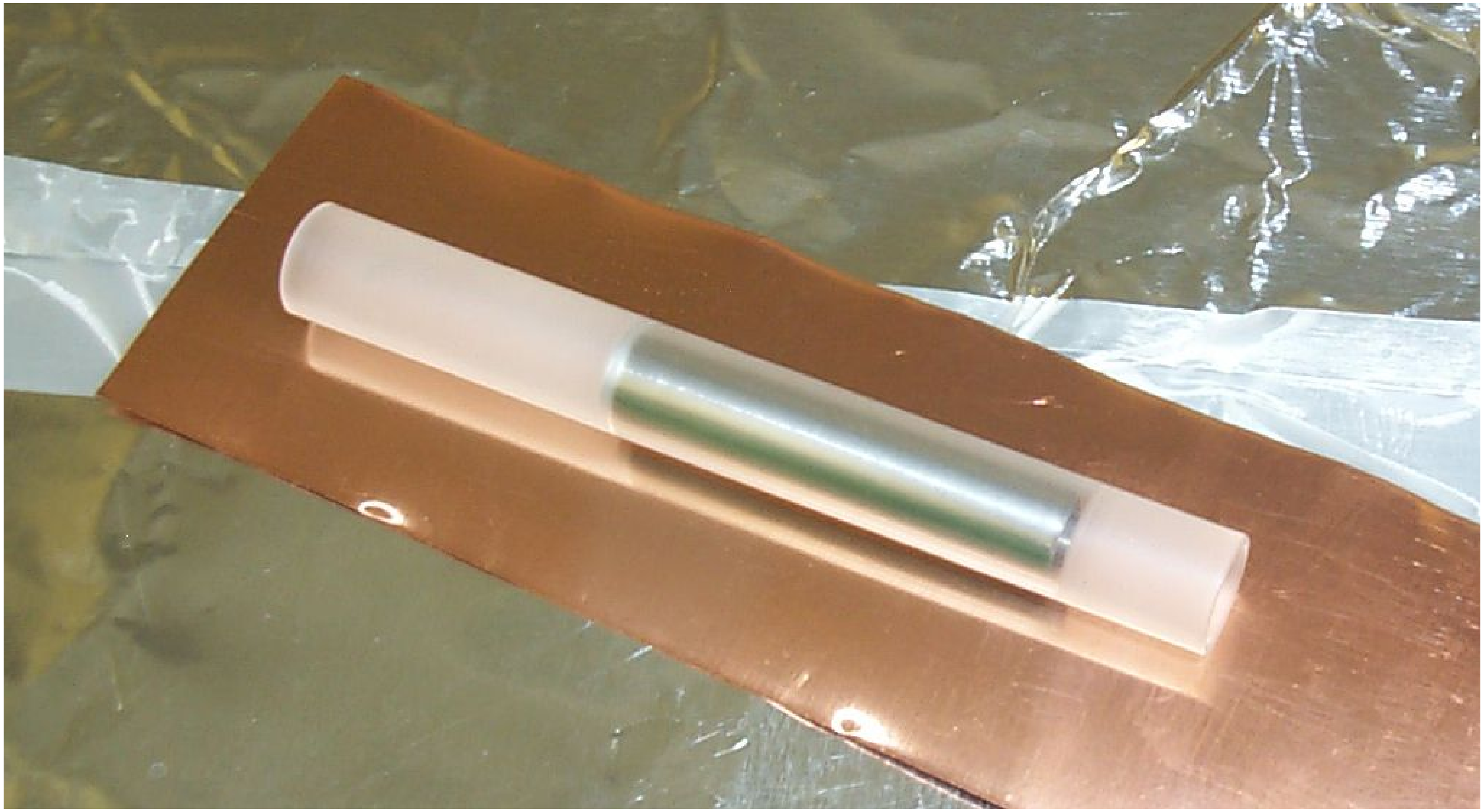


Figure 25 Annealed Fe-foil cathode fit to a GPC body. A freshly annealed Fe foil cathode is shown in a GPC body. A series of quartz tubes and rods of appropriate diameters are used to roll (shape and expand) the cathode after annealing to better conform to the shell dimensions. The cathode is inspected for its tolerance within the GPC body. Its length must match the ground section of the envelope. Its diameter and concentricity should result in a close fit to avoid any movement within the counter.

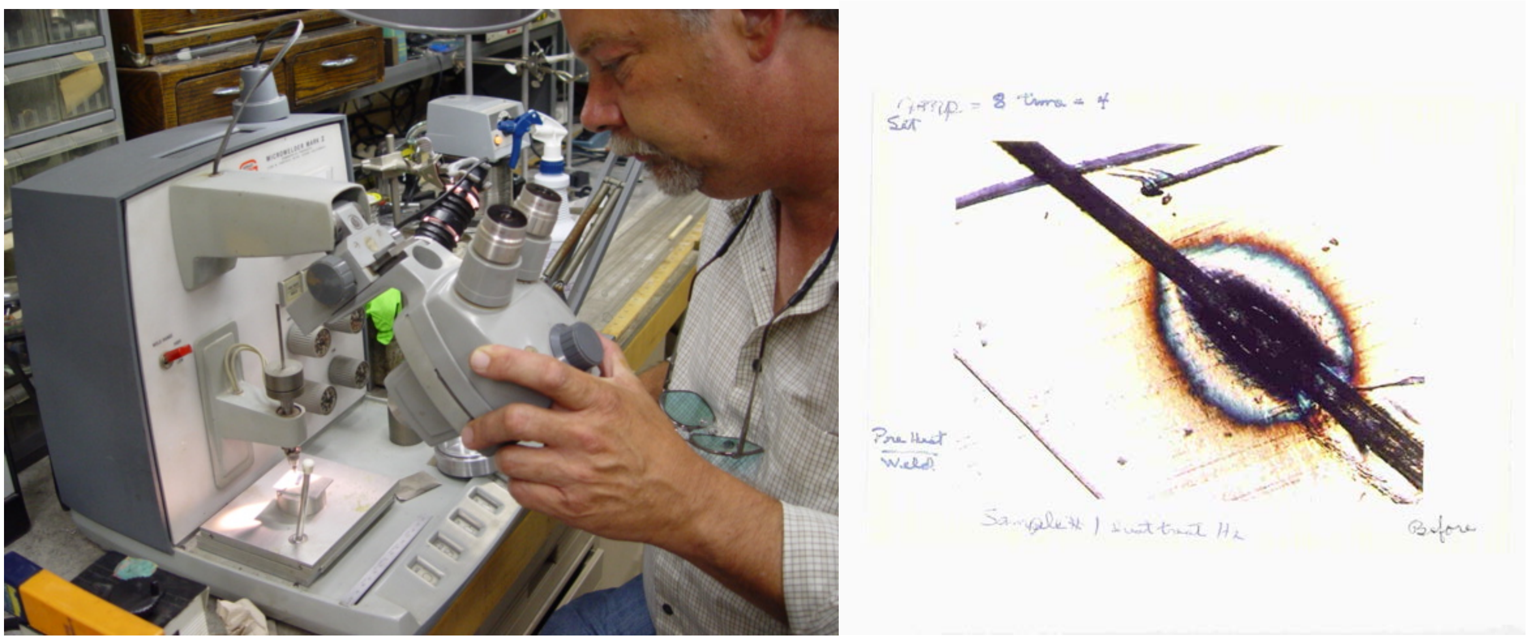


Figure 26 Spot welding a W-wire burr to a Fe cathode. A 50 µm diameter W wire is spot welded to the Fe foil cathode near its seam and at one end. The left photo shows the micro welder with microscope. The right image shows the W wire spot welded to the Fe foil before it is brightened by annealing.

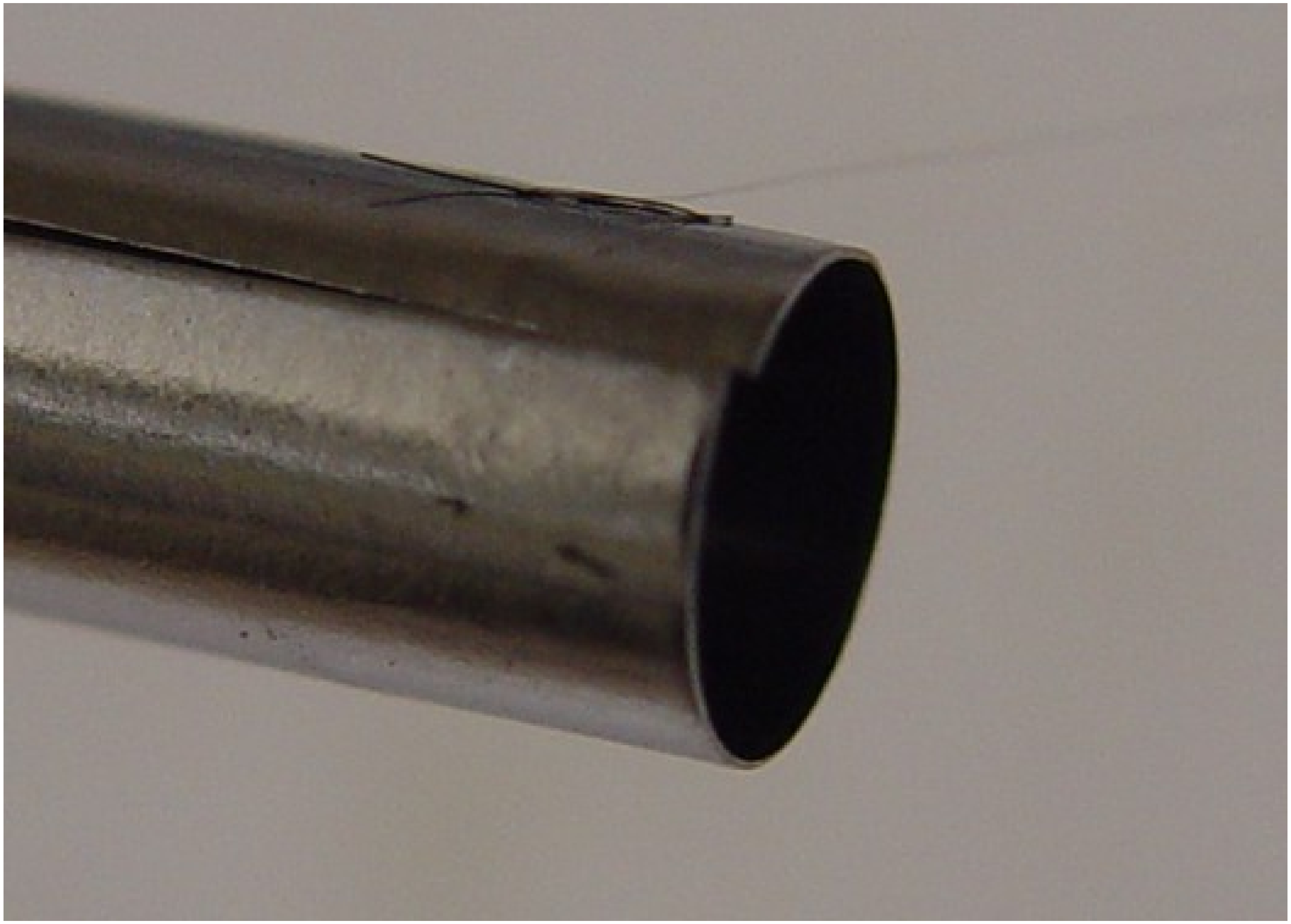


Figure 27 Cathode, burr and W-wire lead. The photo shows a 50 µm diameter W wire (burr) spot welded to the rolled Fe-foil cathode parallel to the seam. Also evident is the 13 µm diameter W-wire lead to be connected to the high voltage electrode; described later in the text.

2.7 Electrical Connectors

The GPC is a hermetically sealed envelope that operates at nominally -900 V to the cathode (Fe-foil liner) with the anode connected to ground. By filling the GPC with 100 kPa of a mixture of sample and P10 gas, it can detect a single beta emitted through its interaction with a neutral Ar molecule; the process known as the *Townsend Avalanche* (discharge) effect. To establish high-voltage input to the cathode and detector response output from the anode, the cathode and anode are connected to an electrical circuit by-way-of two gold (Au) coated connectors. Each connector is soldered to 0.26 mm diameter Cu wire using zero alpha solder to minimize radioactive contamination within the vicinity of the detector (Figure 28). The Cu wire from each electrode is then welded to the anode wire and the cathode (wire) lead using Ga.

1. Cut the rear (open end) section of the connector off with small wire cutters; file any burrs; connector should now be of uniform diameter across entire length
2. Inspect hole to make sure 0.26 mm diameter Cu wire will go in all the way
3. In a wooden block, drill a hole about 3 mm deep and diameter just over sized of the connector diameter
4. With a metal file, shave off some alpha-free solder from the ingot
5. Place connector in hole bored in the wood block and fill connector with solder particles
6. Wet the Cu wire end with solder
7. While heating them both with a solder gun, insert the Cu wire at the end of the connector; the Cu wire will slide into the connector; test by pulling on the wire while holding the connector
8. Using a very small file, file down any excess solder that may have leaked from the connector seam
9. Shape wire in ‘S’-shape and cut to length to fit 1 mm short of W-quartz seal (Figure 28 b). The distance between the end of the Cu wire and the entrance to the connector as measured along a straight line is about 10 mm. The distance from that imaginary line to the apex of the bend is about 5 mm. Distances will vary considering the length of the quartz tube beyond the hermetic seal. It is best to lay the counter in the cradle and check the electrode for fit before it is Ga welded in place.

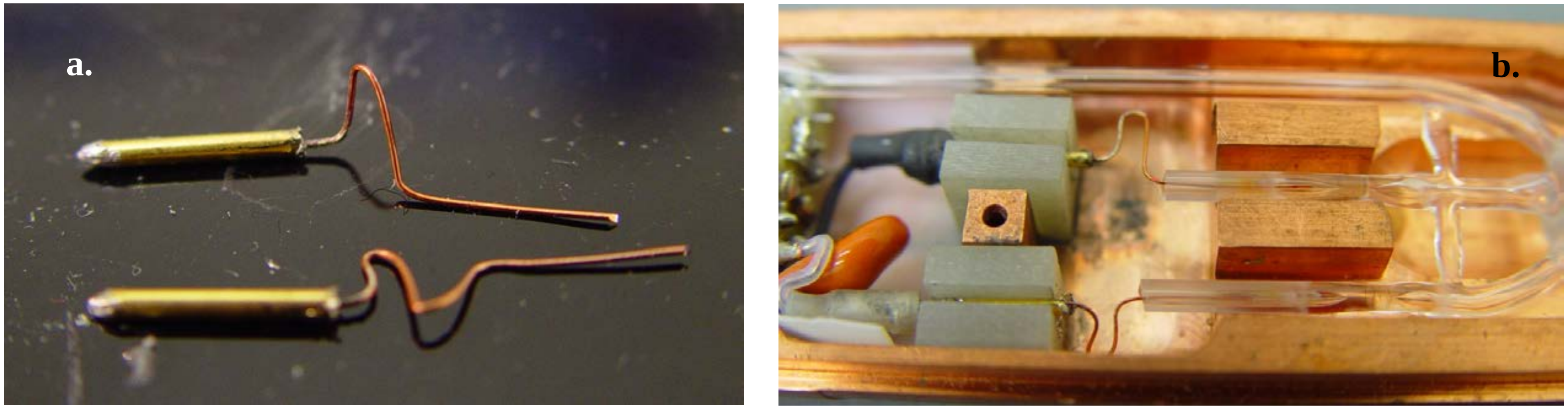


Figure 28 Electrodes sized and fit to a GPC and cradle. a. Gold coated connectors with Cu leads are used to connect high voltage supply to the cathode and GPC anode voltage to the preamp. These electrodes are shaped from 30-gauge wire and soldered to Au coated electronic connectors using zero-alpha solder. Before soldering, connector's (crimp) ends are clipped off with small wire cutters and filed to remove any sharp points. Connectors are filled with coarse particles (100 um) of zero-alpha solder and soldered to 0.26 mm diameter Cu wire. **b.** Once the GPC has been assembled (Fig. 44, p. 74) each electrode is then fit to a tube end before (Ga) welding to their corresponding W-wire leads. Their S-type shape provides additional length to align the connector to the insulated hold downs. The length of the Cu wire from the rear of the connector on is about 10 mm.

III. COUNTER ASSEMBLY

3.1 Insert Cathode into Body

On a clean workbench, the cathode is positioned within the GPC envelope by tying a 13 µm diameter W-wire lead to the cathode burr, threading the wire through the body and extension tube, and inserting the cathode. First, secure the wire to the cathode. This is best accomplished by mounting the spool of wire on a rod horizontal from a ring stand. Slide the cathode over a glass tube with an outer diameter slightly less than the cathode diameter. Extend the cathode beyond the glass tube and clamp the glass tube to an alligator clip on a jeweler's stand. Align the cathode horizontally and pointing directly to the bottom of the spool separated by about 10 cm (Figure 29). Rotate the cathode so the seam and burr are positioned on top. With tweezers, pull enough wire out to extend about 5 cm beyond the burr. Tie the wire to the burr as if you are securing a boat line to a cleat (cleat hitch knot for burr with two spot welds; or, a square knot for a burr with one spot weld). With scissors, clip the lead off as close as possible to the burr.

The next step is to thread the lead wire and cathode into the GPC body and tube extension. To do this, spool out another 10 cm of W wire from the spool and cut the wire. From a spool of 50 µm diameter W wire, cut about 10 cm. Thread this tether wire through the tube extension and the GPC body (Figure 30). The end of this wire is then glued to the cathode W lead using a cyanoacrylate adhesive. (A better alternative is to lap weld the two ends together.) Allow the glue to dry and outgas for at least one hour before proceeding to the next step. Pull the lead through the body and tube while maintaining tension and moving the cathode towards the body (Figure 31). Once the cathode is completely within the body, take a glass rod and push the cathode while maintaining tension on the lead wire until it bottoms out at the front plug.

1. Slide the cathode back over the tube used for fitting
2. Mount this tube to the jewelers stand via an alligator clamp about 10 cm above bench top; axially-align cathode with the W wire spool (Figure 29)
3. Roll out about 40 cm of 12-µm diameter W wire and tie end several times to burr on cathode; between ties, wrap wire around burr once; tying is accomplished by using two

pointed SS tweezers, one with a curved tip (I find the easiest knot is a sailors knot used to make fast to a cleat.)

4. Cut W wire on spool end; place two needles over the wire lying on the bench at about 1/3rd distance apart to prevent the wire from looping or kinking
5. Roll out about 40 cm of 50 μm diameter W wire and cut
6. Thread this wire through the HV quartz tube, plug channel and GPC body; it should be long enough to extend out both ends with more out the HV tube; this is to minimize solvent vapor reaching the GPC from the cyanoacrylate adhesive
7. With the cyanoacrylate adhesive, glue the end of W wire coming out the HV tube with the cathode lead; give at least one hour for the glue to set and out gas – the longer the better
8. Mount the GPC on a ring stand in horizontal position to facilitate pulling the HV lead through the GPC with the tethered 50 μm W wire; care must be taken to maintain enough tension on the wire assembly to avoid looping or kinking of wire
9. Once the cathode reaches the GPC body, begin to slide the cathode off the tube (jig) and into the GPC while maintaining tension as the cathode is slipped in mm-by-mm; wire tension can be better examined by wetting the GPC body slightly with water; also, make sure the burr is aligned with the HV channel in plug 1
10. Once the entire cathode is within the GPC, one can use a polished quartz rod to push the cathode to the end of the GPC body all along maintaining tension on the wire
11. Cut the W wire lead to expose about 4 cm of wire beyond the quartz tube
12. Check for electrical continuity between the cathode and lead; make sure cathode is pushed against the plug

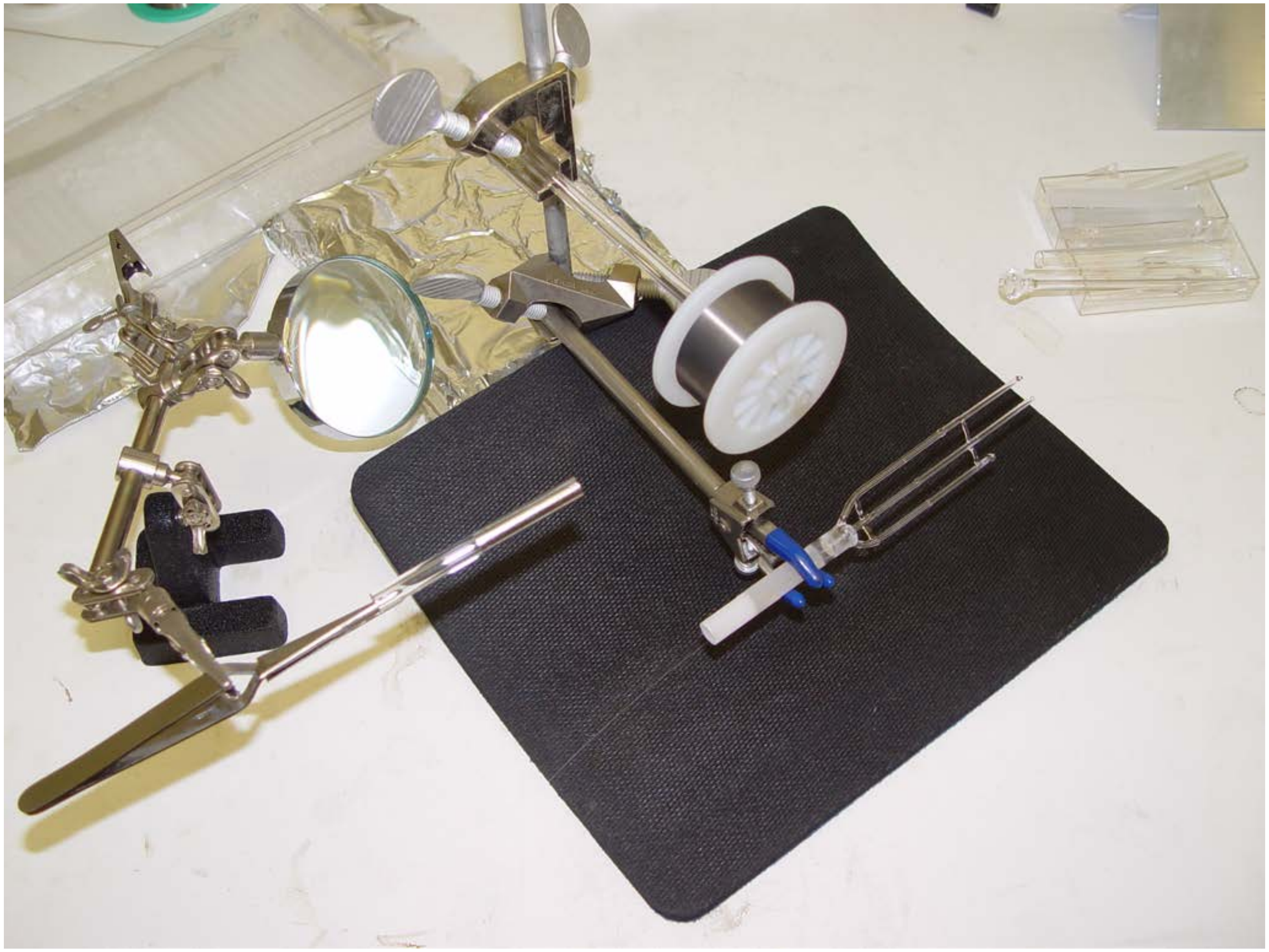


Figure 29 Setup to insert the W-wire lead and cathode into a GPC. The photo shows the setup for tying a 13 µm diameter W wire to the burr on the cathode, pulling the cathode W-wire lead through the body and HV tube with a tether wire (50 µm diameter W), and inserting the cathode into the body of the GPC. The procedure is carried out in a clean air bench without air flow. Note, in the upper right corner is a plastic box of tubes and rods used to roll the cathode to specifications.

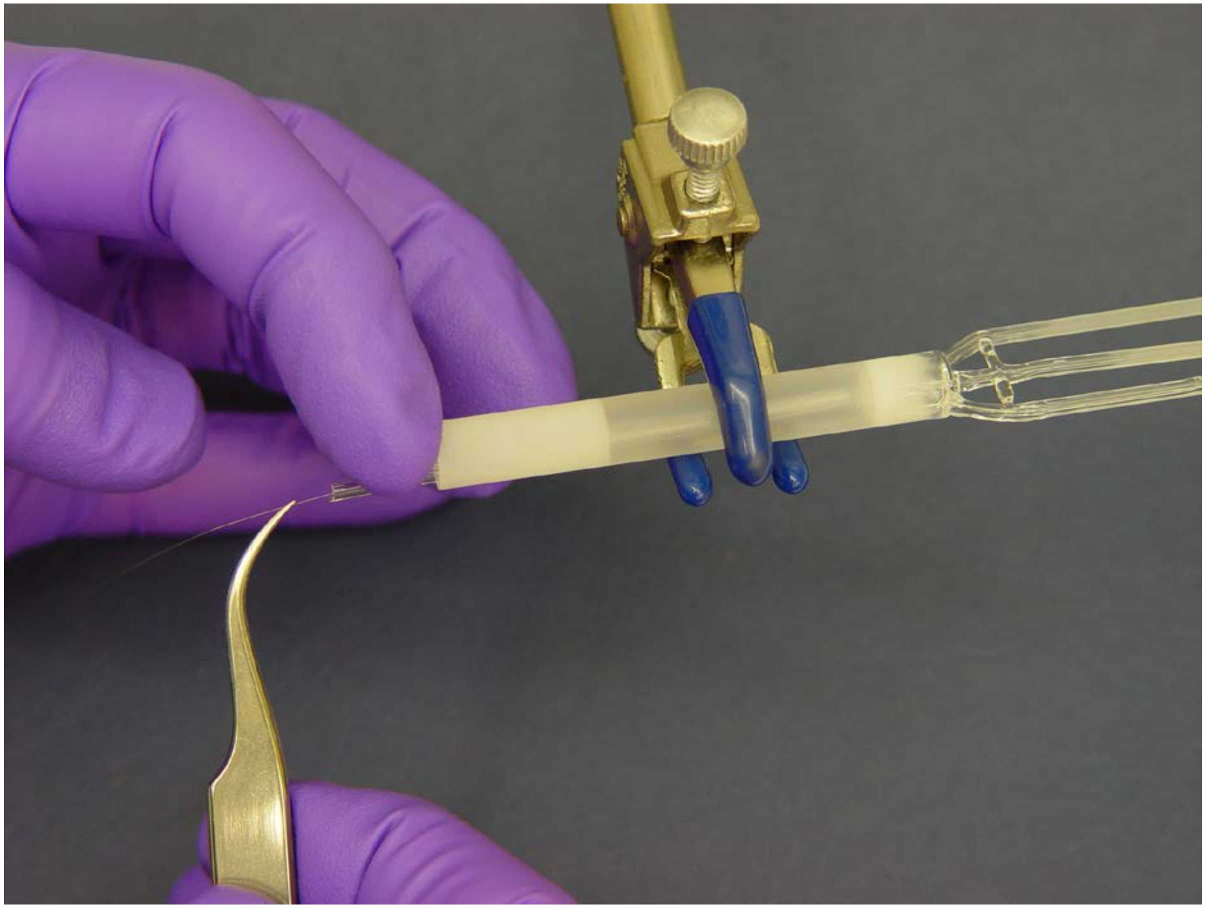


Figure 30 Threading a tether through a GPC body. A 50 µm W-wire tether (25 cm long) is threaded through either the HV tube (right bottom) or the open body (left). Regardless of its path, the tether wire may require a slight bend at the end to get by the transition from the plug to the tube (or vice versa). From the body side, you will need to cover the tether with a small diameter feeder tube that is slightly longer than the GPC body. Insert the feeder tube to within about 2 mm of the front plug HV channel. Push the tether into the tube and guide it through the plug channel and out. The feeder tube will keep the wire stiff while pushing it through the transition as shown in the photo.

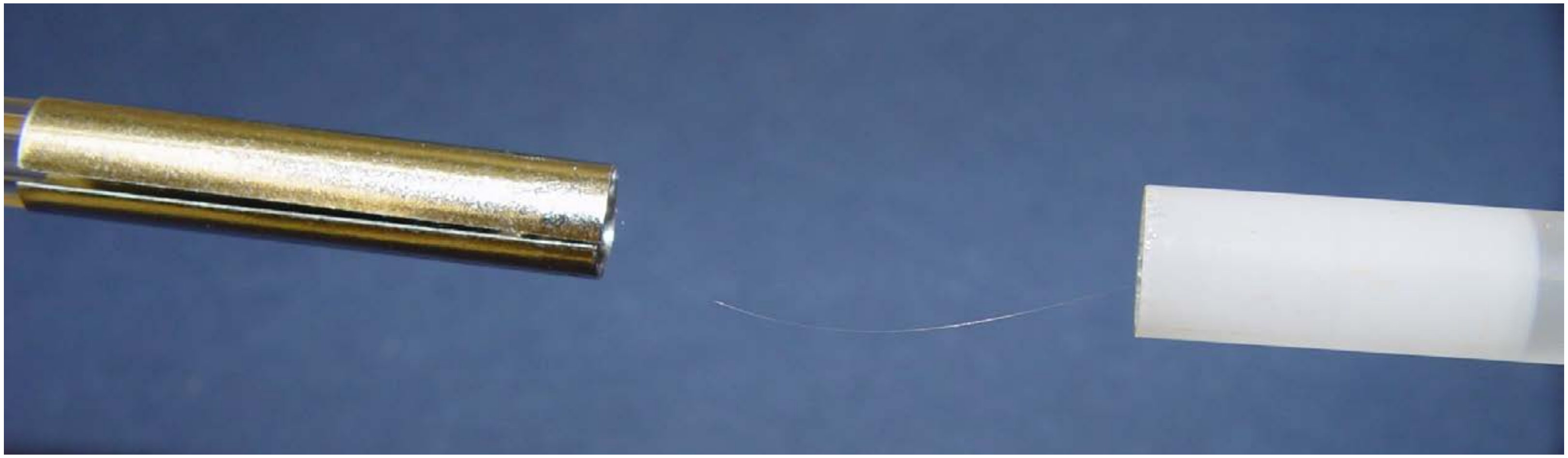


Figure 31 Guiding the cathode into the GPC body. First, align the cathode with the wire spool (see Fig. 29). With the 13 µm diameter W wire lead tied to the burr (Fig. 27) and the other end glued to the tether (Fig. 30), the cathode is slowly moved closer to the body while the tether is pulled to sustain slight tension on the lead (above). The goal is to maintain the wire taut enough so the lead will not kink or end up within the active volume once the cathode is in place. Once the cathode is within the counter body, use a polished glass rod to continue pushing the cathode in while maintaining alignment of the cathode burr and the HV channel. Make sure you sustain slight tension on the wire during this procedure.

3.2 Quartz Hermetic Seal on HV Lead

A hermetic seal of quartz around a tungsten wire can only be made by first removing the oxide coating on the wire in an inert atmosphere and then heating the quartz hot enough to collapse the tubing around the wire. On the glassblowing bench, purge the entire body with forming gas (5% N_2 and 95% H_2; composition varies with manufacturer) for about 2 h (Figure 32). With a H_2/O_2 flame, heat the quartz tube in the area where the seal is to be made just hot enough to glow the W wire but not enough to collapse the quartz. This removes the tungsten oxide from the wire and leaves a bright metallic sheen. Now, direct the flame at the tube with an intensity to collapse the quartz around the W wire. The seal should be at a distance away from the body that meets specifications when fit within the copper cradle as shown later (Figure 44). Clip the wire about 2 cm from the end of the quartz tube. Check the electrical continuity between the cathode and the wire lead.

The integrity of the quartz-to-W seal is tested at vacuum using a helium (He) leak detector. The detector is connected to the tube end (3.2 mm OD) with an o-ring fitting (Figure 16). While spraying the inside of the GPC with a gentle jet flow of He (barely perceived by the sensation on your tongue); any sign of He detected would identify a leak.

1. Crack open the very end of all three tubes
2. Mount counter in vertical position with window (open) end up using a ring stand
3. Crop HV lead at the end of the tube using surgical scissors; a slight motion upwards is used to avoid applying tension to the wire which may lead to breakage
4. Push any residual exposed wire just into the tube
5. Connect forming gas line from its regulator to the counter body using surgical tubing on window end
6. Connect surgical tubing to the HV, anode and gas inlet tubes and interconnect as seen in Figures 32-33
7. Purge counter volume with forming gas for at least two hours
8. With H_2/O_2 flame, first momentarily heat the quartz tube at the sealing point with just enough intensity that the wire glows red but the quartz does not begin to soften; this will remove the oxide layer of the W wire
9. Stop flow of forming gas
10. Without pause, heat the tubing at the same point with greater intensity around the entire circumference of the tube to soften the quartz and force it to collapse around the W wire

11. Check electrical continuity between the cathode and the W lead; with magnifying glass, check for any oxide residue deposit on the inside wall of the quartz tube; observe whether or not the W wire is continuous
12. Store GPC body assembly in inert gas until further assembly
13. High vacuum leak check seal by attaching counter HV tube to vacuum manifold using a 3.2 mm diameter o-ring fitting; be sure not to grab W wire when sliding the fitting on – it will break if you do
14. Evacuate against seal and check for leakage by isolating volume in the presence of a pressure sensor, e.g., capacitance manometer, pirani or ion gauge
15. A He leak detector is a more sensitive means of identifying any leaking
16. Store assembly in a dry box purged with Ar until ready for stringing anode wire

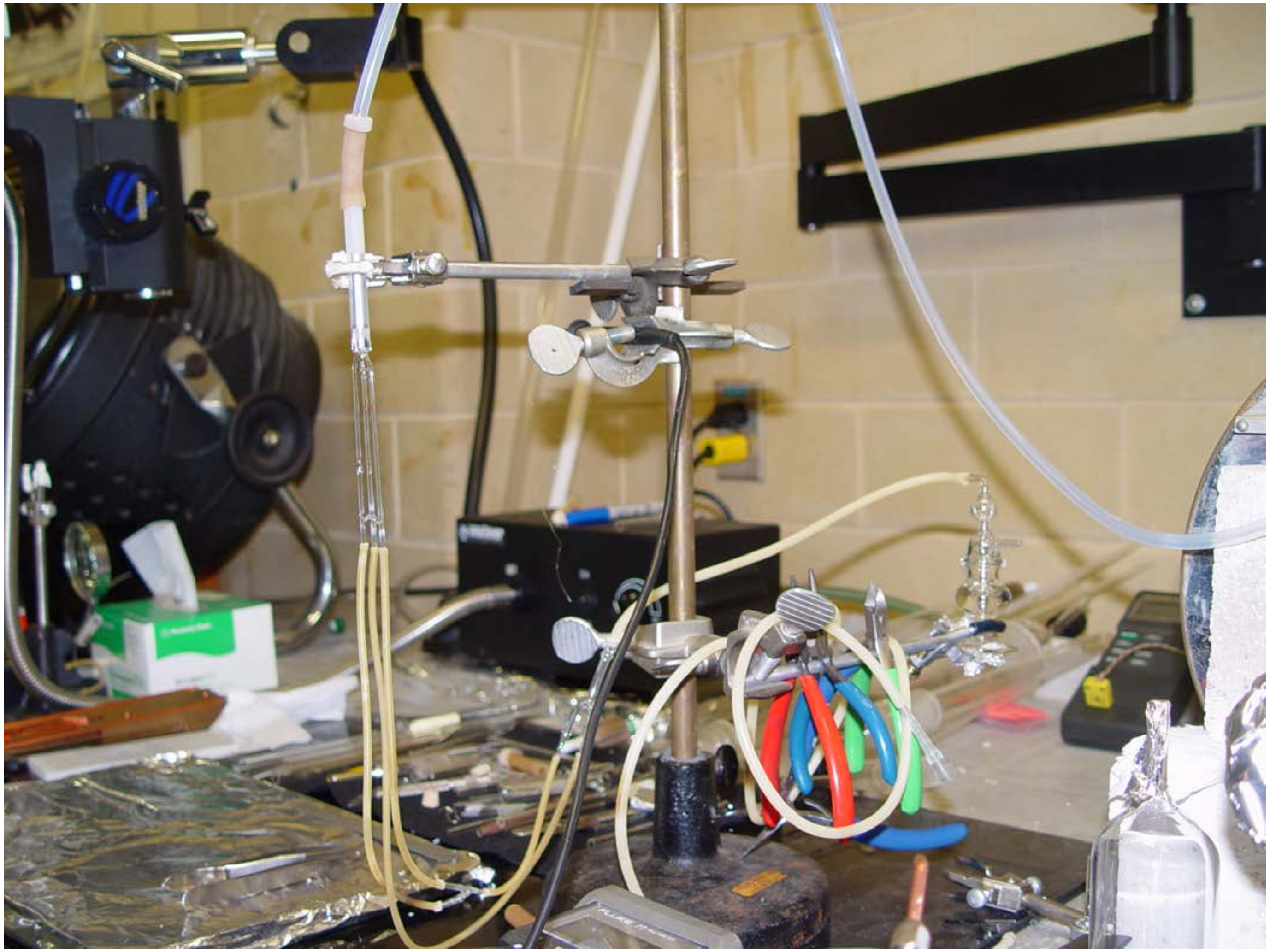


Figure 32 Setup for purging a GPC with forming gas. The photo shows the purge setup for flushing the counter with forming gas before collapsing the quartz around the tungsten lead. Flow of purge gas is through the rear (top) of the GPC, the three tubes and a bubbler of water to prevent back diffusion of air. Be sure that the forming gas line is leak free.

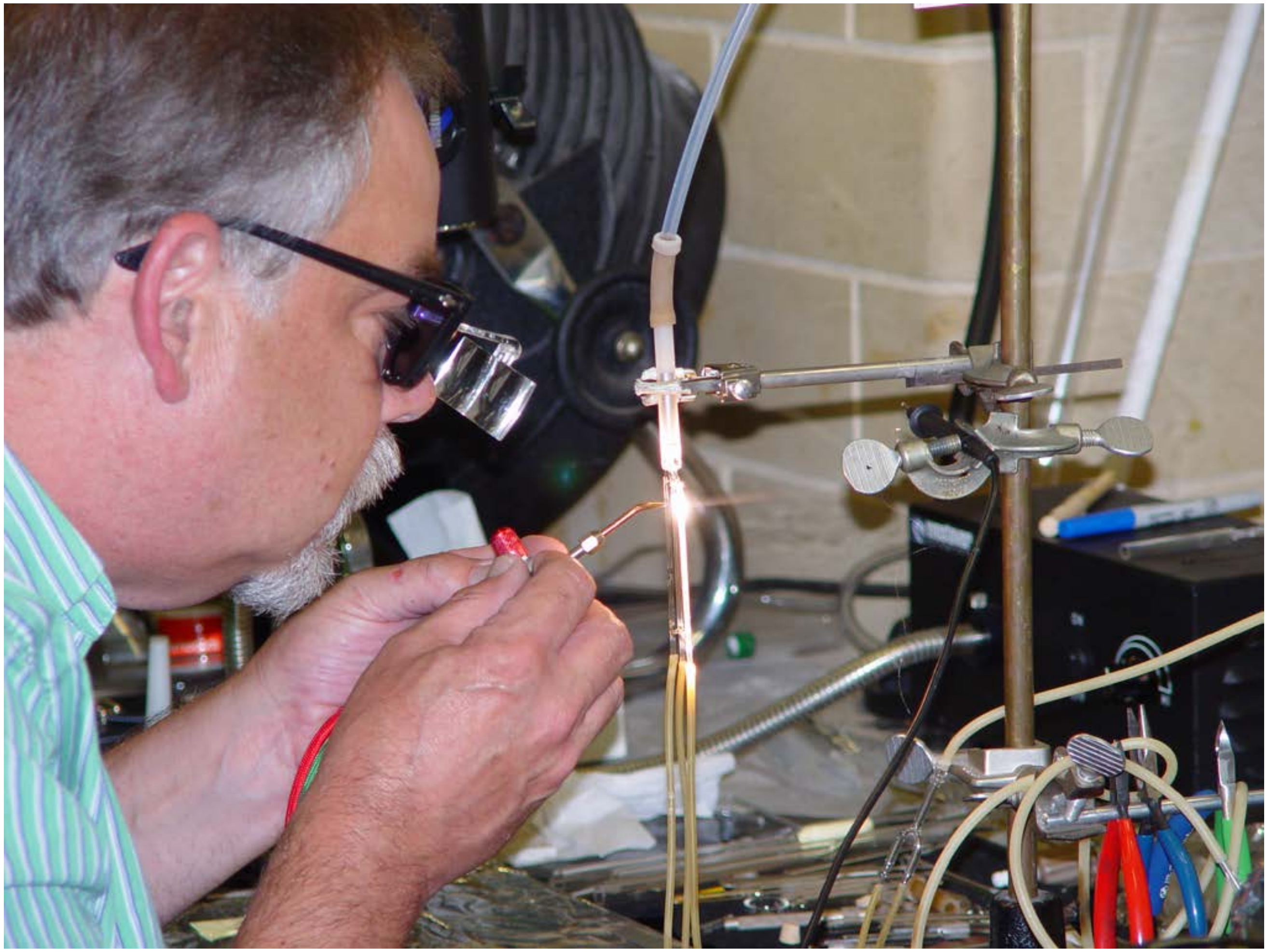


Figure 33 Sealing the quartz capillary tube around the HV lead. Before the seal is made, the W wire is heated sufficiently to remove the oxide coating on the wire yet not enough to collapse the quartz around the wire. Once a metallic sheen appears on the wire, the H_2/O_2 flame is intensified to draw the quartz against the W wire thus providing a hermetic seal. After the seal, electrical continuity is tested between the cathode and the lead wire exiting the quartz seal. There is no need to pull tension on the W wire while making the seal.

3.3 Ni/Cr Spring

The GPC body is now returned to the clean bench for addition of the anode wire. First, a spring is formed from a 4 cm length of Ni/Cr wire by creasing it over an anvil at an angle of about 30 ° (Figure 34). The spring angle is then tested by seating it within the Vee grove within the rear plug. To tie the W-wire to the spring, the spring is held in place at one end in a pencil vice which is mounted to a jeweler vice (Figures 34-35). A 13 µm diameter W wire is then tied to the spring using a square knot. Once secure, the Ni/Cr wire is cut to size so that each end is about 3 mm from its apex.

1. Cut (8 to 10) cm of Ni/Cr wire
2. Clean Ni/Cr wire with a methanol soaked wipe
3. Sandwich Ni/Cr wire between Al anvil and mating angled Al piece to bend wire at same V-angle as in the rear plug
4. Mount one end of Ni/Cr wire in a pencil (micro-) vise. Mount the vise to the jewelers vise using an alligator clamp and position in line with spool of W wire such that unspooled wire will be in line with right-angle Ni/Cr (Figure 35)
5. Pull about 40 cm of 13 µm diameter W wire from the spool and tie it at the apex of Ni/Cr spring. Note that the spool of W wire is secured to a rod mounted horizontally on a ring stand about 10 cm above the bench just as it was when attaching W wire to the cathode burr.
6. Tie a square knot, while making sure there is some tension on the wire. This can be done fairly easily using hook-style tweezers. Do not kink the W wire coming away from the spring – this section of wire will define the anode in the active volume region. If this happens, start over again.
7. With some slack between the spring and spool, cut the Ni/Cr spring to length on the unsupported side. Use a sort of a surgical style scissor.
8. Cut the other end of the spring to length. Take care not to let W wire coil, twist, kink or bend

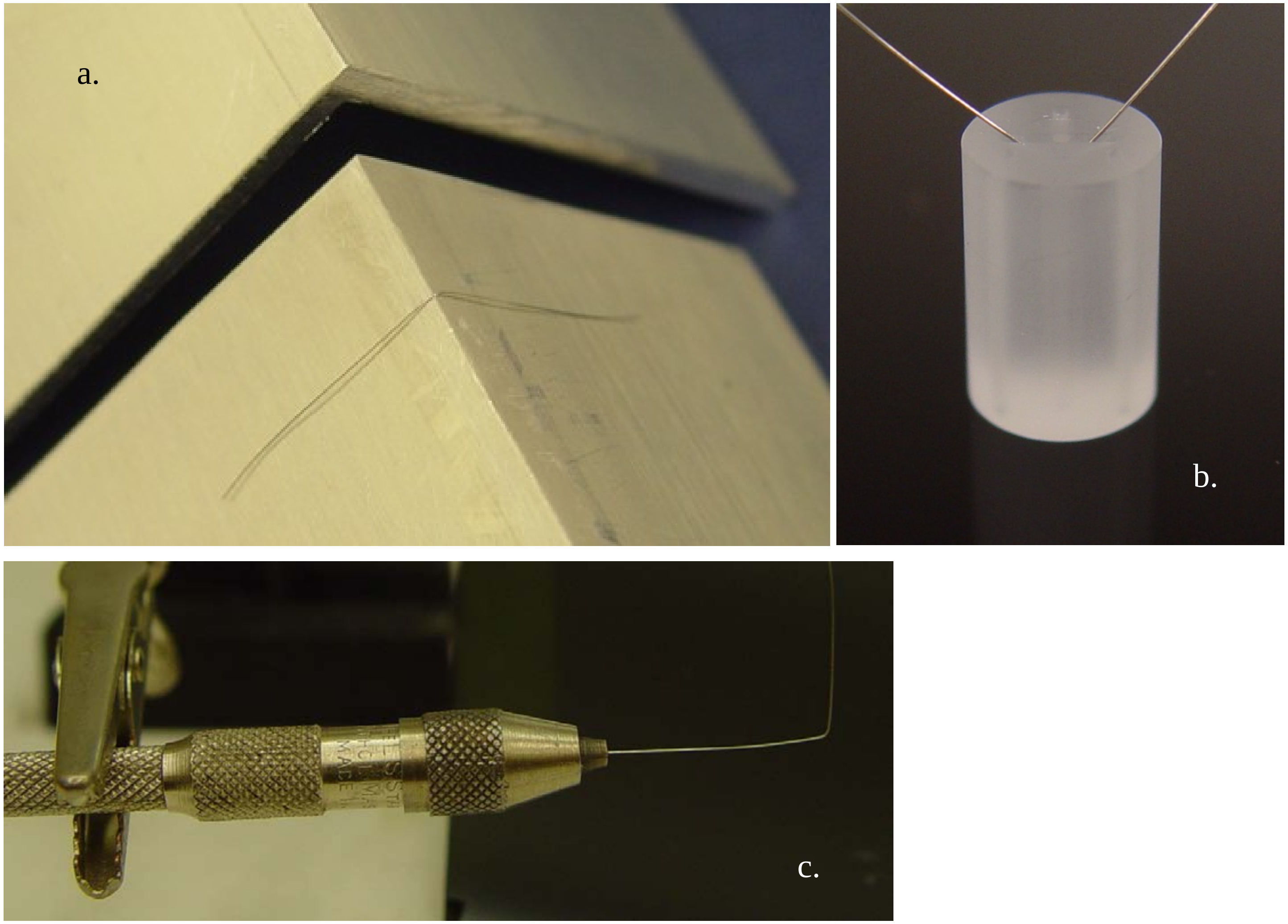


Figure 34 Shaping and sizing an anode spring. a. A piece of NiCr wire is bent at a 30° angle by pressing over an anvil (top left). **b.** The spring is checked against the V-shaped grove in the rear quartz plug (top right). Note that the spring has not yet been cut to fit within the plug. **c.** The spring is then anchored to a pencil vice, clamped to a jewelers stand and positioned horizontally in line with the W-wire spool (bottom; similar to configuration in Fig. 29).

3.4 Integrating the Spring, Rear Plug and Anode Wire

The procedure to thread the anode wire is similar to that used to thread the cathode lead. A piece of 50 µm diameter W wire – used as a tether – is threaded through the center tube. The anode lead is threaded through plug 2 being careful not to allow the wire to coil or kink. Glue (with cyanoacrylate adhesive) the anode wire end to the end of the tether. Allow at least an hour for the glue to set and outgas. Pull the tether and anode wire through the counter gently so as to avoid catching the transition on the cathode or pulling so fast as to compromise the attached wires. Once the wire transition has cleared the end of the center tube, pull directly on the 13 µm diameter W wire while nudging the plug/spring/wire assembly through the end and maintaining slight tension on the wire (Figure 36). Without tension on the wire, it may coil inside the counter active (cathode) volume. If this occurs, remove the cathode and wire and start over. (Again, a cleaner alternative to the adhesive is to lap weld the two wires together. Any residue of the adhesive left within the counter will add to the background and affect the counter operating characteristics.)

1. Cut the W wire at the spool about 40 cm from the spring
2. String a 40 cm long piece of 50 µm diameter W tether wire through anode leg
3. Glue the 13 µm diameter W anode wire to the tether wire as was done with the HV lead; allow at least one hour to dry and outgas
4. Slowly pull wire, plug and spring through counter taking extreme care to keep wire tight; wire can loop up and get wedged between plug and body if not kept with tension
5. Cut anode wire off 5 cm from end of leg
6. Glue a 50 µm diameter W wire 3 cm long to the anode wire; this lead is to support a standard weight to give tension to the anode wire when making the hermetic seal

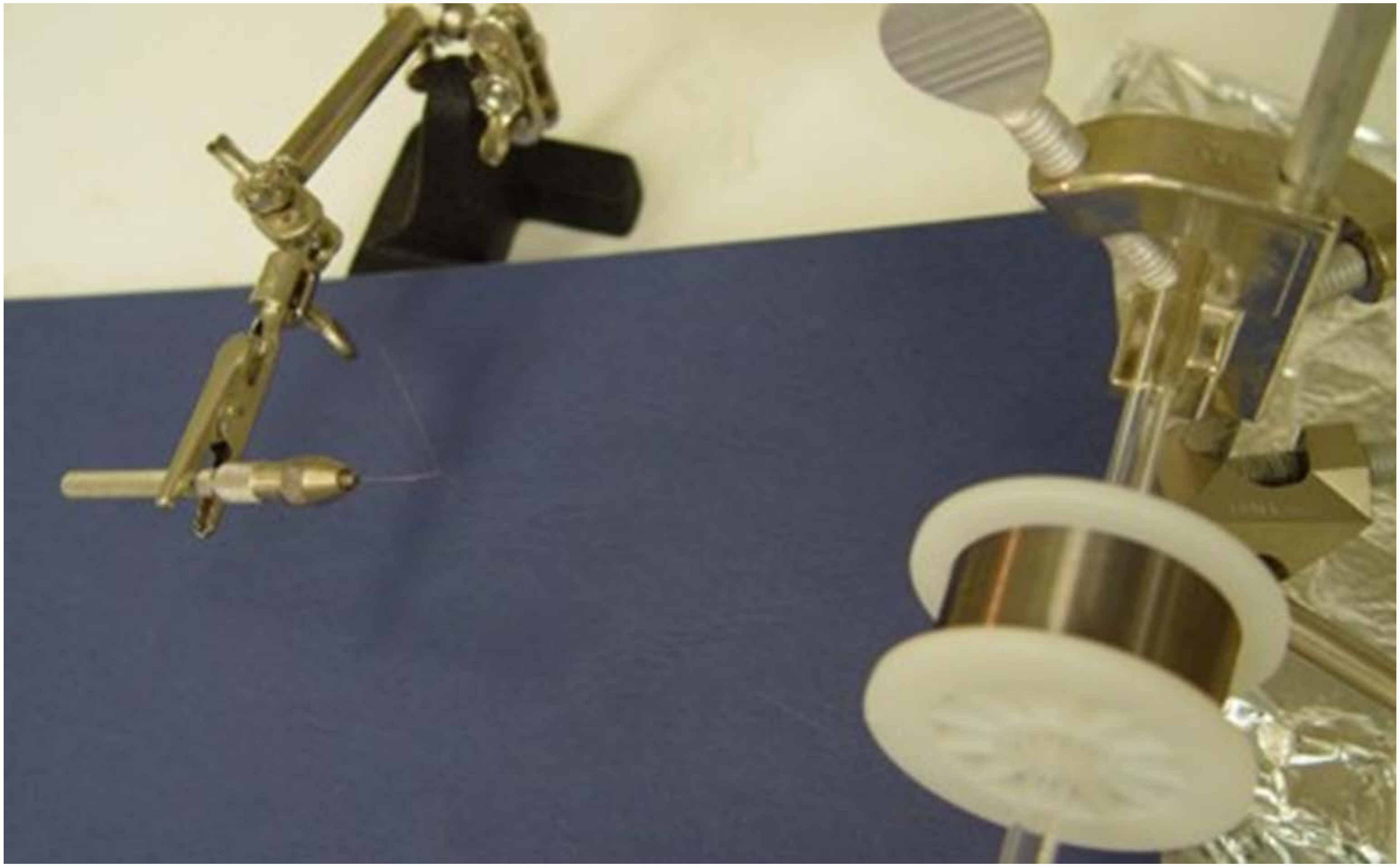


Figure 35 Tying a 13 µm diameter W wire to a spring. Unwind 13 µm diameter W wire from the spool using tweezers. While the spring is anchored by the pencil vice (left), tie the W wire around the spring at its apex using a square knot. Cut each end of the spring about 4 mm from the apex. Thread the W wire through the side of the rear plug with the V-grove and pull through. Make sure the V-spring does not extend beyond the plug (see Fig. 34b). However, the optimum length of the spring would end flush with the plug surface.

Figure 36 Securing the anode wire within the GPC body. Thread a 50 µm diameter W tether wire (25 cm long) through the center tube and out the body using the same technique to insert the cathode lead. The tether wire is now glued to the 13 µm diameter W anode wire. Allow at least an hour for the glue to set and outgas. Very slowly begin to pull the anode wire through the counter while moving the plug and spring toward the body with some tension on the anode. Inset at lower left shows the plug after it has been inserted into the body. Continue to push on the plug and maintain tension on the W wire.

3.5 Quartz Hermetic Seal on Anode Wire

The assembly is then moved to the glass-blowers bench and purged with forming gas much like the procedure for the cathode (Figure 37). An anchor mass of about 0.1 g is glued to the end of the anode wire and covered by a bell-jar style tube. After about 2 h of purging, the anode wire is heated enough to remove any surface oxidation and then the quartz tube is collapsed against the W anode to produce a hermetic seal (Figure 38). Figures 39 and 40 are examples of ‘good’ and ‘bad’ seals depending on the presence of oxygen. Electrical continuity is checked between the spring and the anode lead. The anode wire is then clipped at the end of the tube.

The integrity of the quartz-to-W seal is tested at vacuum using a He leak detector as described in Section 3.2.

1. Mount GPC in vertical position on ring stand with rear facing up
2. Epoxy standard mass to end of anode wire with counter mounted in vertical position
3. Place bell-jar style tube around suspended anode wire to minimize air diffusion into the counter
4. Connect surgical tubing from forming gas to counter body
5. Tee surgical tubing lines from the forming gas line to the gas inlet leg with a pinch off and to a bubbler
6. Purge counter with forming gas for at least two hours
7. Flame the tube at the seal point just enough for the W wire to glow red but not enough to soften the quartz
8. Interrupt gas flow and immediately seal quartz tubing to W anode wire. It is important to make sure the seal is at the specified distance from the GPC body.
9. Check electrical continuity between Ni/Cr spring and end of W wire anode

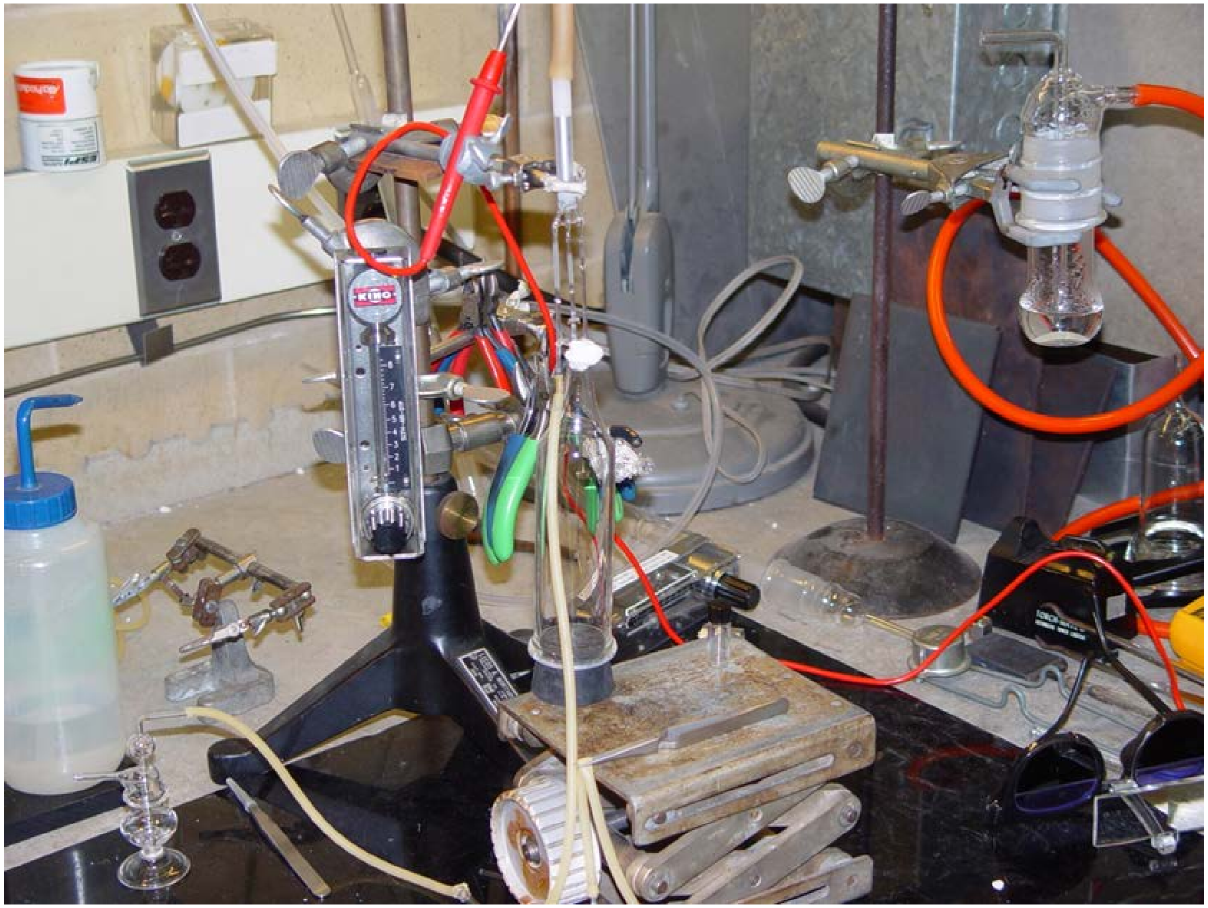


Figure 37 Setup for anode wire seal. The GPC is mounted vertically on a ring stand with a surgical tube connected to the rear. Using a cyanoacrylate adhesive, a 0.11 g mass is glued to the tungsten anode wire and suspended within a glass bell jar. To remove air, forming gas is flushed through the body, anode tube and gas inlet tube for 2 h. Be sure that the forming gas line is leak free. During this flush, the bell jar and bubbler prevent air from back diffusing into the system. Just before the seal is made, a short time of intense heating (glowing of wire without moving the quartz) in the region of the seal removes the oxide coating and minimizes potential fracturing of the quartz.

Figure 38 Quartz to W (anode) wire seal. The procedure for collapsing the quartz tubing around the W anode wire is identical to the procedure for sealing the cathode wire in place. After the seal, electrical continuity is tested between the Ni/Cr wire spring – positioned in the rear plug – and the anode wire exiting the quartz seal.

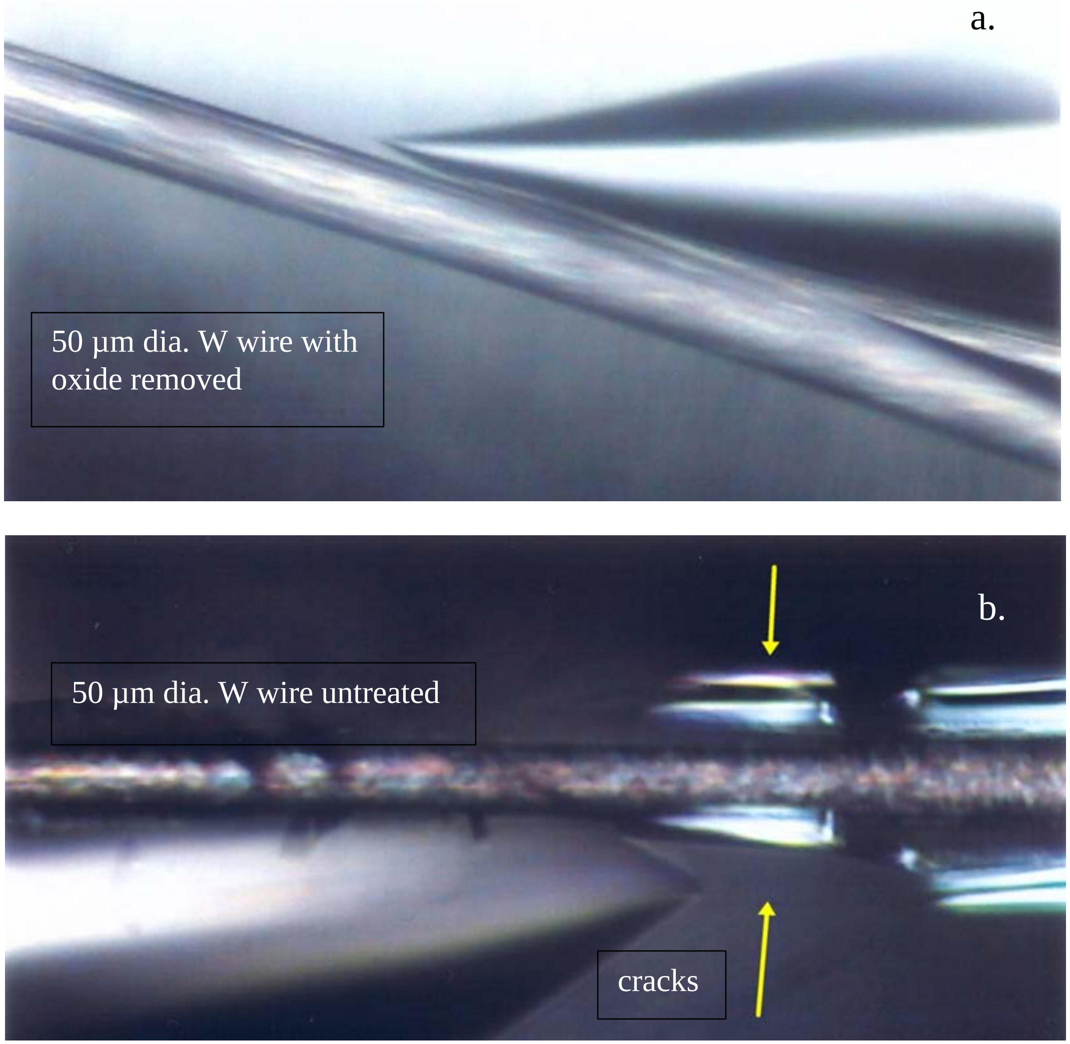


Figure 39 Notes on collapsing quartz tubing around W wire. a. a good quartz-to-W seal is made by thoroughly flushing the tube with forming gas, heating the tube just enough to brighten the W to remove the oxide layer, and then apply more intense heat to collapse the quartz around the W wire. **b.** a bad quartz-to-W seal with cracks present (bottom, arrows). This seal is likely to leak.

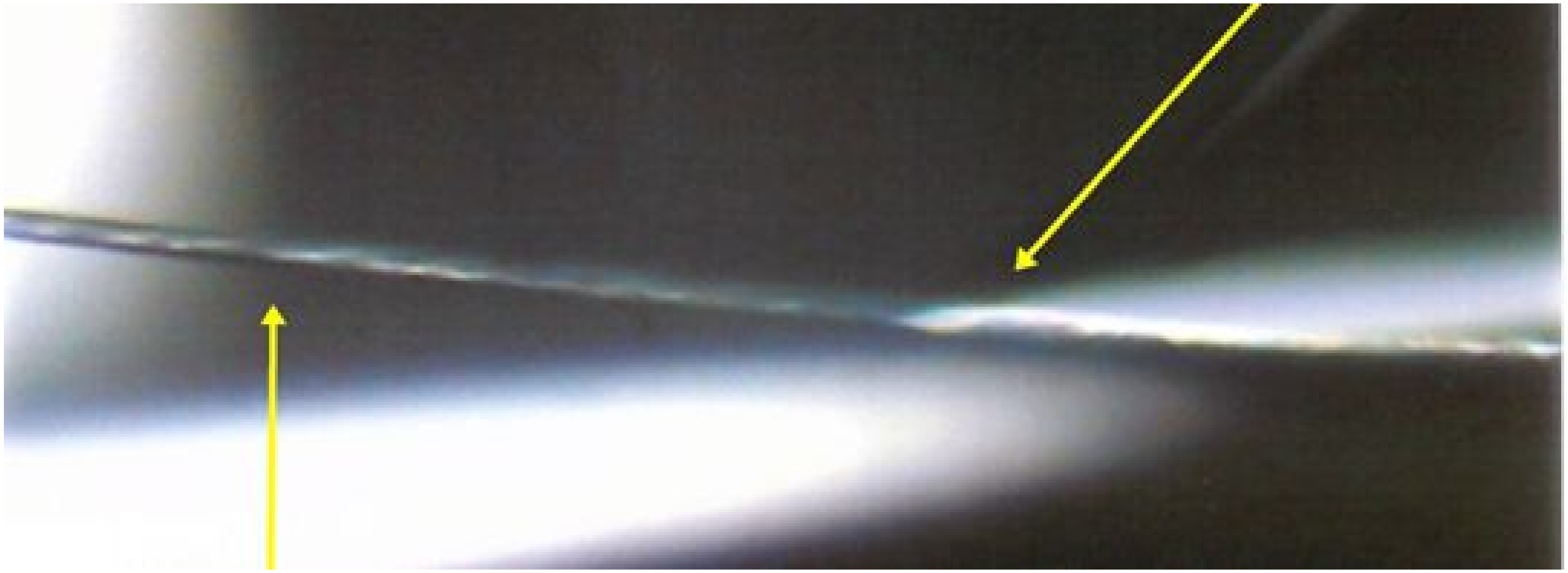


Figure 40 Tungsten anode and cathode lead seal. Photo shows quartz collapsed around a 13 µm diameter W wire that appears bright; enters quartz to the right of the right arrow. Arrow at the left identifies the same wire in the vacuum side of the GPC envelope. Immediately after creating hermetic seals, electrical continuity is checked between the HV lead and the cathode and between the spring and the anode lead.

3.6 Seal Window to Body

Figure 41 shows the glassblower making the final seal of the rear window to the counter body. Prior to sealing, the window had been checked to be sure that its edge is flush with the end of the counter body (Fig. 6). The body is again purged with forming gas for a minimum of 0.5 h primarily to keep from oxidizing the cathode (Section 3.5). Holding the window above the rear of the counter body, allow the window to fall under the force of gravity. Once settled, make sure that the window is in contact with the rear plug by applying pressure to the window. Also, the edge of the window/slide should be flush with the edge of the counter body. The glassblower then makes the final seal using a micro-flame of H_2/O_2.

1. Re-establish purge of GPC from gas inlet using forming gas for 0.5 h
2. Stop flow by removing the surgical tubing and drop the window down the rear of the body; allow it to free fall under pneumatic state; apply force to the window to assure that it has bottomed out on to the rear plug
3. Flame seal window to body
4. Immediately place counter in vacuum chamber on vacuum manifold to cryopump
5. Use an RGA to measure the initial out gassing; then RGA after one day of evacuating; continue to evacuate and RGA for at least a week or until the moisture is removed or reaches steady state

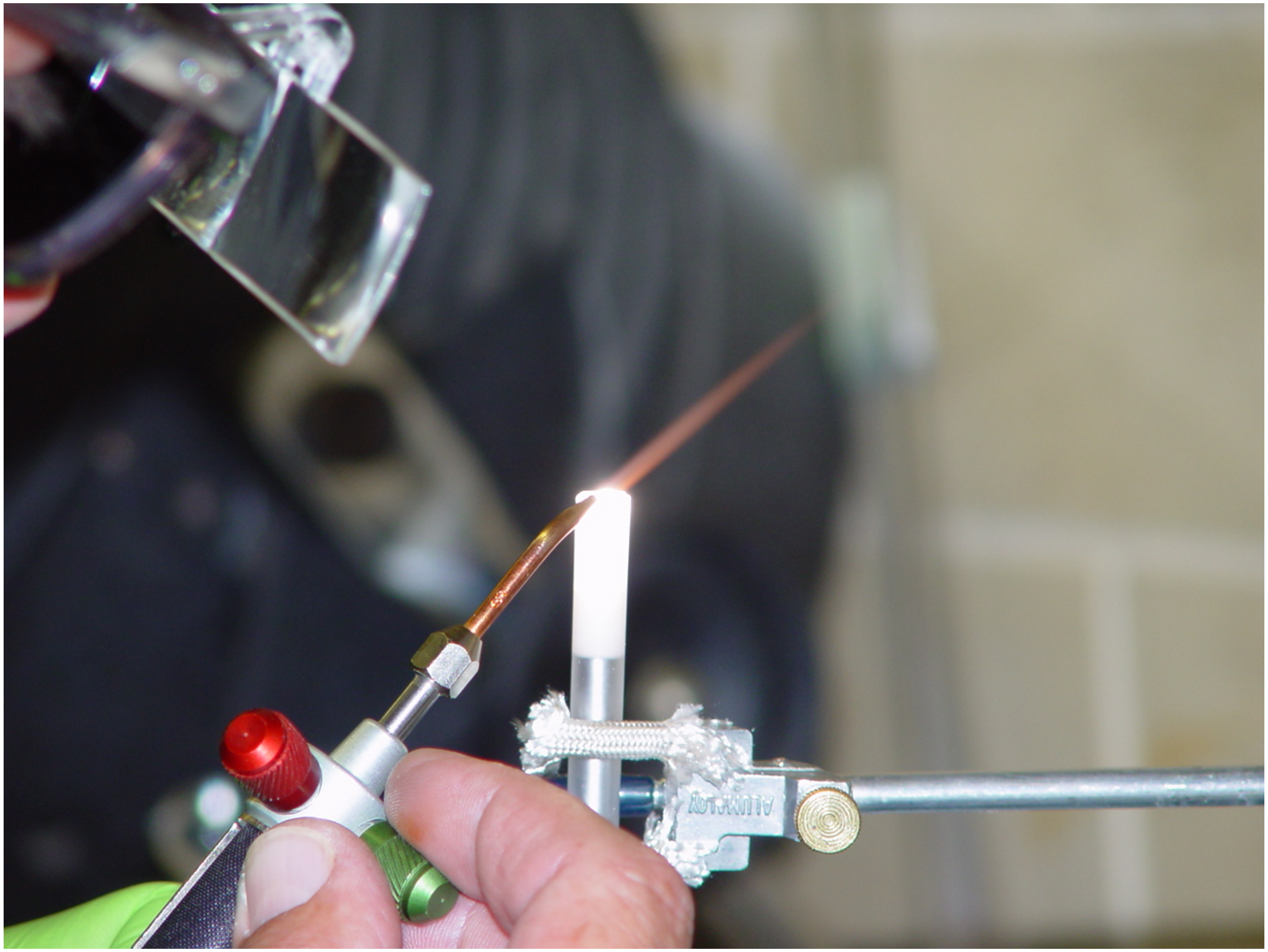


Figure 41 Sealing the rear window to the GPC. Here, both the cathode and anode have been positioned and their leads encased in the quartz. The photo shows Mr. Anderson making the final seal between the window and the GPC body using a micro-flame of H_2/O_2.

3.7 Seal Stopcock to GPC

Refer to Figures 42 and 43 for the setup to seal the stopcock to the GPC. (Note that these Figures illustrate the sealing of the stopcock onto a used counter.) Once the stopcock is sealed to the counter, check that it fits within the cradle. Figure 44 shows a GPC as it is positioned within the cradle with the six critical tolerance points highlighted. Finally, place the GPC in a vacuum chamber to remove moisture. When repairing a counter, drill a hole into the epoxy that encapsulates each of the Ga-filled electrodes before evacuating on. This will allow for the Ga to expand when it melts.

1. Check the clearance of the GPC and stopcock within the cradle; gas inlet, anode and cathode tubes should be in alignment requiring no modification (Figure 44)
2. Mount GPC body on one side of lathe and the stopcock connector on the other side of lathe; make sure the connector side of stopcock is axially aligned with the axis of the GPC (Figure 42); this should have been established using a jig (Figure 52) before the stopcock was leak tested; if repairing an old counter, see step 6 below
3. Seal components with a natural gas flame to minimize moisture then reinforce the seal with a H_2/O_2 flame.
4. Fit counter to cradle to check tolerances (Figure 44)
5. Immediately place counter in a chamber (Figure 45) and evacuate until no water is detected by the RGA (Figure 46). It will likely take days to remove the moisture. (If there are visible signs of rust on the Fe cathode surface, only after screening might one consider treating the cathode in an atmosphere of H_2 at elevated temperature, nominally at 350 °C.)
6. Figure 42 actually shows a stopcock being sealed to a used GPC with electrodes; this indicates a repair to the broken inlet tube only. Considering this circumstance, the following steps must be taken: thoroughly degrease stopcock with 1,2-dichloroethane and clean by acid soak with alternate rinsing using quartz distilled water (Section 2.4); times can be abbreviated. Immediately following the flame sealing, grease the stopcock with Apiezon N (Section 4.1) and attach it directly to a He cryopump to remove moisture. The connection to the vacuum manifold must transition via a metal bellows tube and clamp system to minimize any vibrations to the counter. Vibrations experienced with this kind of pump have known to compromise the anode (center) wire (Figure 47d)

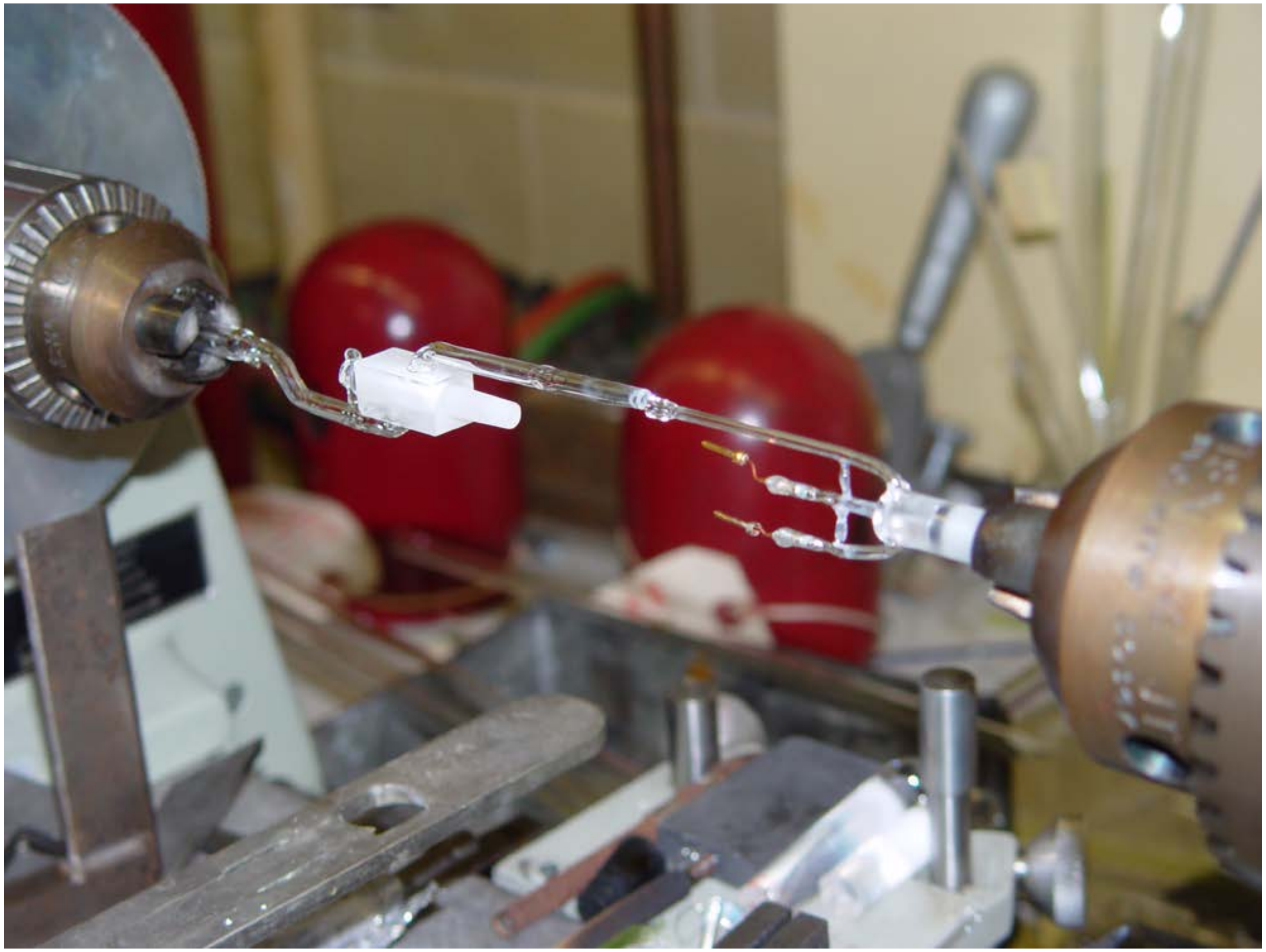


Figure 42 Sealing a stopcock to a GPC - step I. The counter and stopcock are mounted in a glassblowers lathe. Their tubes for the gas inlet should be in alignment with each other. The stopcock is configured earlier using a jig that aligns the barrel with the connector (left). The position of the gas inlet tube on the counter was aligned earlier to center the GPC with respect to the stopcock plug and connector. Note, electrodes are not present at this point for a new counter. This photo was of a working counter where the stopcock broke off and needed replacing.

Figure 43 Sealing a stopcock to a GPC - step II. The counter and stopcock are fused using a H_2/O_2 flame. Electrodes or W-wire leads are shielded from the flame with Al-foil-covered high temperature ceramic fiber (left of flame). The counter tube is preheated slightly before fusing the tubes in an attempt to drive most of the moisture left to the stopcock side. The purpose is to minimize the amount of moisture that will migrate to the virtual leaks within the GPC.

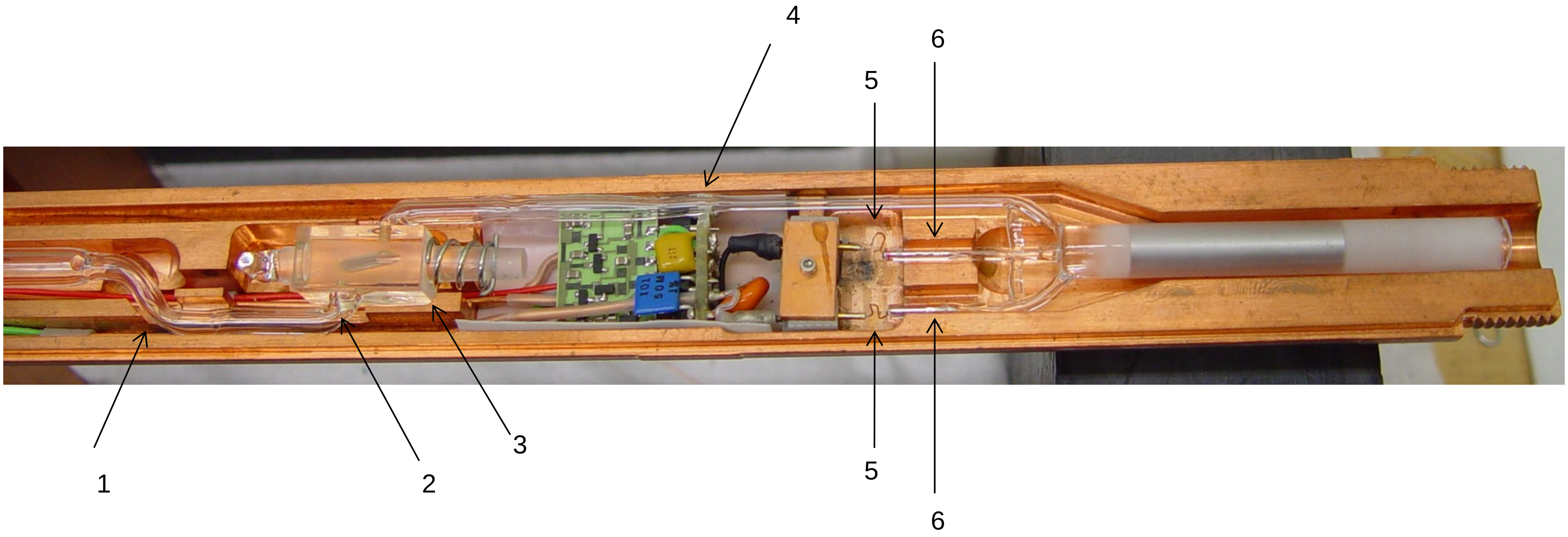


Figure 44 Final fit of the GPC to a cradle. The points identified are critical to the GPC fit within the cradle: **1** and **2** represent clearances for the gas inlet on the atmospheric side; **3** is the barrel edge clearance; **4** is the circuit board clearance; and, **5** is the length of the cathode and anode tubes. Note also, 5 points to the Cu leads that are "S-shaped" to provide additional wire if connector is broken off; and, **6** is the point within the tubes to their ends that is filled with Ga to weld the W wires to the Cu leads (**5**)

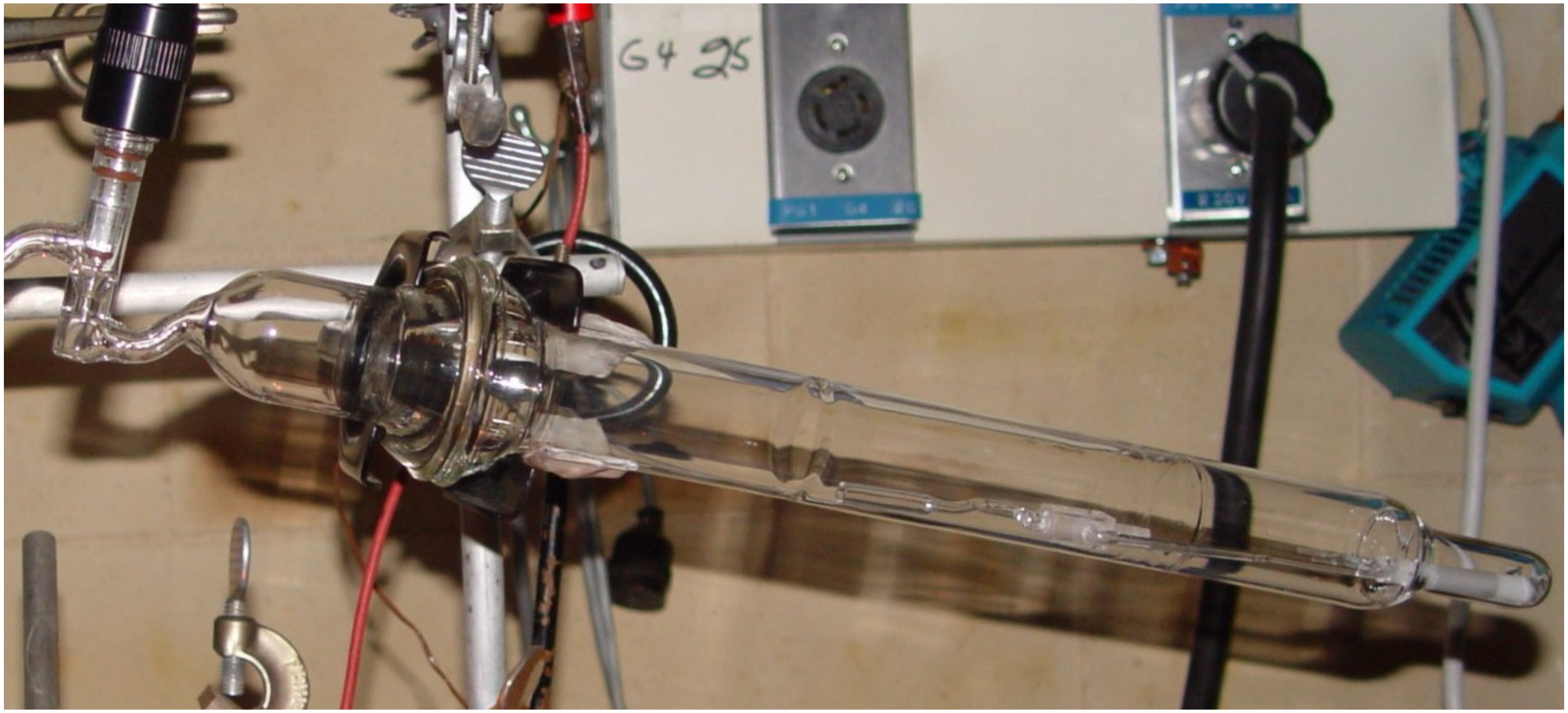


Figure 45 Removing volatile contaminants from a GPC. Before the anode and cathode electrodes are Ga-welded to their corresponding W-wire leads and before applying vacuum grease to the stopcock, the GPC is placed in a chamber fit with a large o-ring connector and stopcock. The chamber is then attached to a cryo-pumping system (Figure 46) to remove moisture and any other potential contaminates that will desorb at room temperature from within the counter envelope. Close tolerances between the plugs and body make for virtual leaks; thereby, requiring up to a week or more to clean up before the counter is placed into operation. Make sure that there are no vibrations from the pumping system that can be translated to the counter. We have direct evidence that pump vibrations can compromise the anode wire.

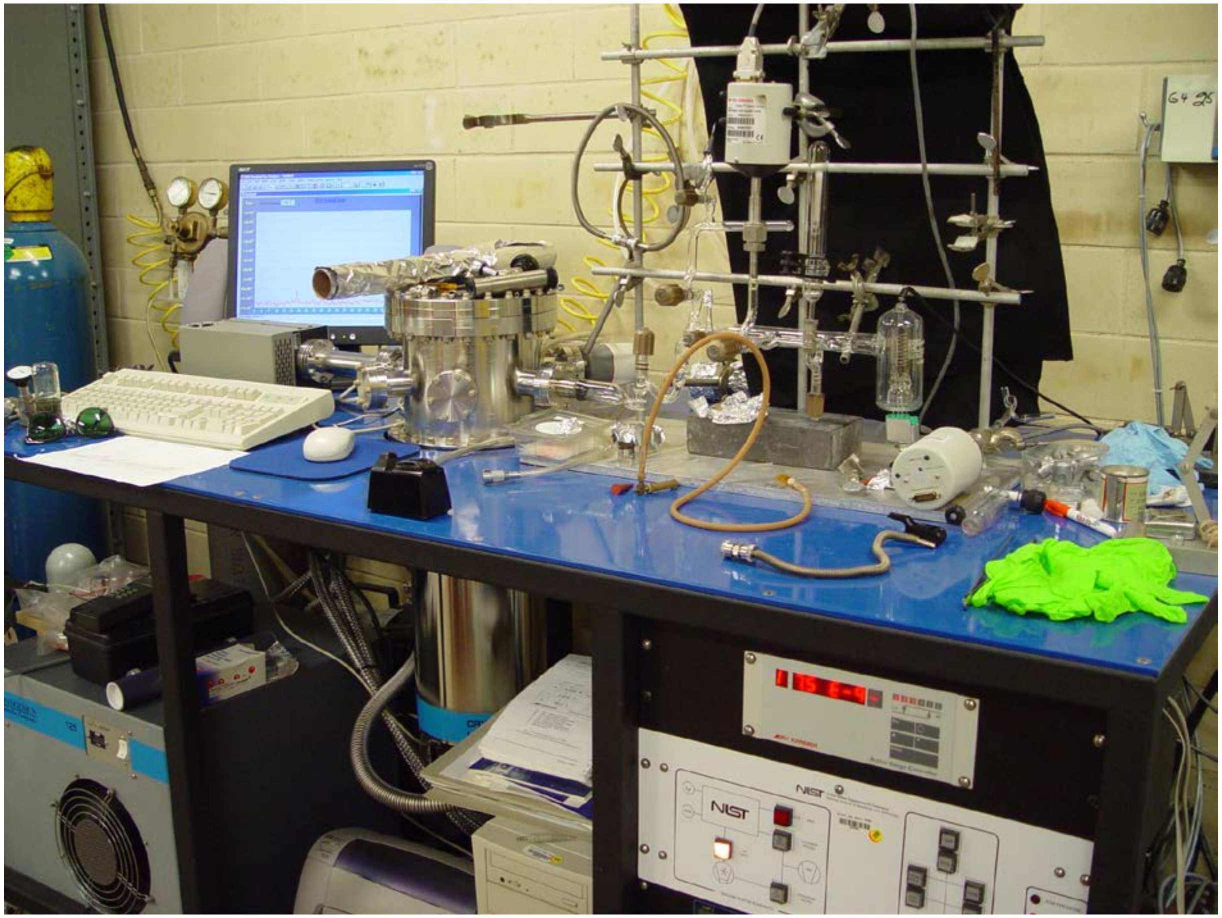


Figure 46 Cryopumping system for GPC cleanup. A He cryopump system is used to evacuate in and around a GPC to remove primarily moisture that is introduced from the H_2/O_2 flame when making seals and attaching the stopcock. The photo above is of the NIST He cryopump, squirrel-cage pump, residual gas analyzer, pirani gauge and capacitance manometer.

3.8 Renovating a GPC Body

For the most part, counters can be rebuilt unless the body has been compromised (Figure 47 a). The counter is dismantled by grinding the rear window off and removing the rear plug and cathode (Figure 48). The electrode seals are cracked off and replaced with new tubes. Prior to reassembly, the envelope is thoroughly rinsed with QDW and baked overnight at 170 °C. To be sure that the internal surfaces are clean, the assembly is heated with a H_2/O_2 flame to burn off any residue/particles that may remain within the counter volume.

If only the capillary between the counter body and stopcock is broke, one must clean the stopcock before sealing. Also, since the electrodes are in place one must pump directly on the inside of the counter and not the whole counter as described above. Moisture must be removed immediately to minimize rust formation on the cathode.

1. With a diamond blade, cut the window seal at a low and controlled rate
2. Apply slight air pressure through gas inlet to minimize the capillary action of water/grit to otherwise migrate up the counter body
3. Remove window
4. Tap out rear plug while pressurizing body with N_2 from gas inlet
5. Check if anode wire remains in contact with Ni/Cr spring and fused quartz at the seal by positioning GPC in the vertical
6. If plug remains suspended, check electrical continuity between the spring and the anode electrode; do whatever is necessary to determine if anode wire had been compromised
7. Crack anode seal; Is the anode wire still intact?
8. Remove rear plug; note observations regarding the state of the anode wire
9. Check electrical continuity between HV electrode and cathode
10. Crack HV seal; Is the W wire lead to the cathode connected?
11. Remove cathode carefully with gloves; try not to compromise the cathode
12. Inspect all pieces before initiating a complete rebuild
13. Replace the three tubes on counter body to specification as outlined above for a new fabrication; include the bridge connections for support
14. Rinse thoroughly counter body, rear plug and window with QDW; CAUTION – only if counter showed signs of or has evidence of grease migration would it be necessary to treat counter body with solvent and acid cleaning
15. Dry in oven at 170 °C overnight
16. Treat in pure oxygen at 700 °C for 1 h
17. Place in vacuum chamber and cryo-pump on body for (1 to 2) days
18. Store counter parts in N_2 dry box until assembly
19. Repeat above steps 3.1 through 3.7

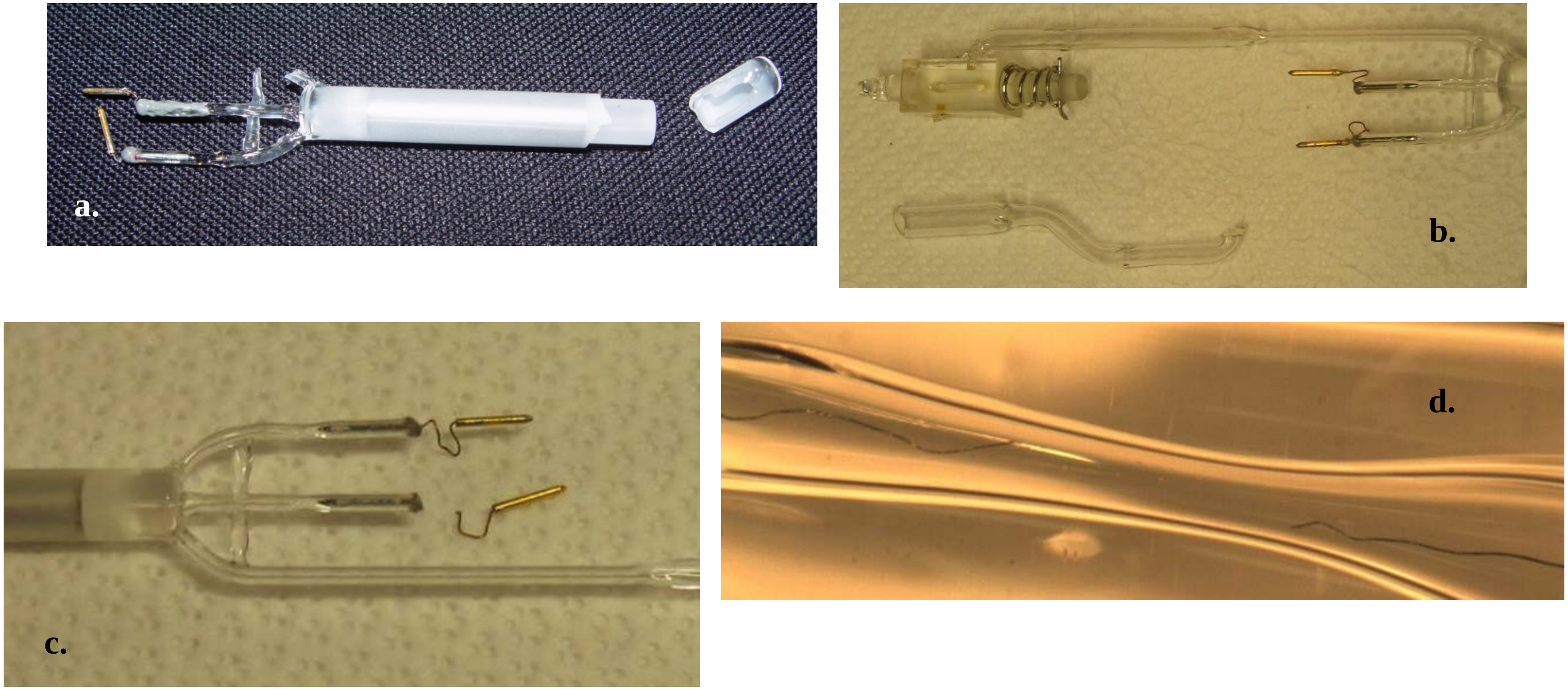


Figure 47 Examples of broken GPCs. **a.** catastrophic breakage from an overpressure on the vacuum line; cannot be repaired but the plugs can be salvaged. **b.** inlet tube to the stopcock was broken by applying too much torque to the stopcock plug when turning without sufficient support of the barrel – difficult to reseal tube to the barrel without distorting the taper. **c.** an electrode connection is broken most likely due to metal fatigue – relatively easy to fix by boring a hole through the epoxy and Ga then securing a new connector wire in the Ga using conductive silver epoxy. **d.** an anode wire is broken from extensive vibrations as the GPC was mounted via an o-ring fitting on a glass vacuum manifold connected to a He-cryopump system.

Figure 48 Disassembling a GPC. Counters can be rebuilt as long as the body has not been compromised. **a.** GPCs are disassembled at the window seal by securing the body in an anvil using tar or beeswax. **b.** Position the seal parallel to a diamond blade, and advance the assembly with a gear mechanism to remove quartz at a slow rate. The automatic feed minimizes the possibility of fracturing or breaking the body.

IV. FINAL PREPARATIONS

4.1 Grease Stopcock

1. With gloves on, directly dab grease onto plug from tube being careful not to come in contact with the metal tube
2. Place dabs axially defining a line for each quadrant
3. Insert plug into the barrel with the bore through the plug aligned with the corresponding bores on the barrel
4. Rotate plug through 360 ° once
5. From then on, use the same quadrant to open and close stopcock
6. If plug gets difficult to rotate after use, backfill GPC with argon to one atmosphere, remove plug and re-grease plug; Note: even a small amount of tension forced on the plug-barrel from the spring will force the grease to migrate away from the plug-barrel contact surface; the optimum material (grit) for lapping the plug and barrel is 9.5 µm diameter silicon-carbide particles; when surfaces are matched, a vacuum of < 0.1 Pa can be sustained for at least two weeks; also, this roughness is enough to sustain the grease depending on the usage
7. Spring tension is best described – short of a way to measure it – by compressing the spring just to the point when no tension is present; then, stretch the spring just to the point that the spring requires compression to insert the anchor. Make sure that when rotating the plug, the tension does not change.

4.2 Leak Test GPC Envelope

1. Attach GPC to cryopump/RGA system for analysis of H_2O, N_2, O_2 and Ar; also, measure any out gassing with a capacitance manometer, ion, pirani or ionization gauge
2. Evacuate overnight using cryopump; RGA analysis of any out gassing
3. Evacuate GPC and record the vacuum pressure
4. Close stopcock and remove counter from vacuum manifold
5. Store counter at least over night
6. Attach counter to vacuum manifold and pump out dead volume
7. Measure the ultimate vacuum pressure (background)
8. Using the smallest volume on the manifold to expand into, open counter to volume
9. Record the pressure and account for volume differences to estimate the amount of leakage (or out gassing) into the counter

4.3 Gallium-Weld Electrodes

The final step in the assembly of a GPC is to use Ga to weld each electrode/connector to its corresponding W-wire lead. This step is especially important since a flawed approach can cause a break in the W lead at the hermetic seal thus making it difficult if not impossible to achieve an acceptable connectivity to the anode or cathode. A mistake at this point likely means starting the process all over.

1. Using a light microscope, anchor the GPC horizontally across the field of view with the focus centered at the anode tube seal; use a small ring stand and three prong clamp to secure the GPC body (Figure 49)
2. With a heat gun, heat to about 40 °C the general area of the GPC tube section and a glass ladle filled with Ga metal; the ladle is mounted to the ring stand and appropriately positioned for heating. Do not direct heat towards the greased stopcock. (<u>Note: some information on the toxicity of Ga is given below*. One should review an MSDS for Ga before proceeding. Always wear gloves if handling the solid material</u>.)
3. With a heat gun, slightly warm the pipette tip to above 40 °C; yet keep below the softening point of the epoxy that seals the capillary fused silica to the pipette tip; the heat gun is positioned about 50 cm away lying on its side and propped up using a wooden block to give it the same height as the GPC on the microscope stage. Do not continuously heat the GPC; only heat to the extent needed to maintain Ga in liquid state; fresh Ga is preferred
4. From the ladle, pipette about 150 µL of Ga using a positive displacement pipette with a modified tip (see below)
5. While observing the anode tube through the microscope, gently insert the fused silica tip of the pipette into the anode tube just short of touching the W-quartz seal
6. Slowly dispense fresh Ga into the tube while gradually moving the tip outward from the tube; you should see Ga migrate as a growing blob moving towards the open end; the secret to a successful fill is to maintain an air gap between the fused silica and the ID of the tube; closing this void near the opening leads to loss of Ga as quickly as you dispense it
7. Fill tube with Ga just short of tubing end (Figure 50)

* Ga metal causes severe skin burns and eye damage. Wear protective gloves, clothing and eye and face protection. If in eyes, rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. Immediately call a POISON CENTER or doctor/ physician. Ga may be harmful if inhaled. The metal is extremely destructive to the tissue of the mucous membranes and upper respiratory tract. It may be harmful if absorbed through the skin and can cause skin burns. It can cause eye burns and may be harmful if swallowed.

8. Wet the Cu wire lead with Ga beforehand; insert the lead into tube very slowly while observing through the microscope; once again, try and maintain a void space between the Cu wire and the tube ID; if Ga is being displaced while inserting the Cu wire, carefully move Cu wire so as to obtain a void; always observe the W wire while attempting these manipulations, otherwise, you run the risk of breaking the wire. Take your time. This is the final step which can easily compromise the GPC (Figure 50).
9. Repeat this procedure for the HV lead
10. Use very gradual vapor cooling with a wipe soaked with methanol to solidify the Ga. Do not make direct contact with the tube during cooling. A rapid quench of the Ga can lead to a fractured tube due to expansion
11. Fit counter to cradle and make fine adjustments to leads to fit cradle
12. Check electrical continuity across the electrical leads
13. Check continuity on each 13 μm W wire with its corresponding Cu wire lead
14. Do not (conductive) epoxy tube ends until the counter passes the operational testing (see below)

<u>Modifying a Gilson Microman positive displacement pipette tip for Ga welding</u>:

15. Cut a 10 mm length of fused silica tubing (0.15 mm ID, 0.22 mm OD); this size tubing will provide the means to fill the electrode tubes with Ga so the Cu wire leads can be welded to the anode and HV W wires
16. Mix up a small amount of Devon FasMetal 10 HVAC 19770 epoxy in 1:1 ratio; enough to modify several pipette tips; a blob the size of a pea is sufficient
17. Withdraw plunger of tip about 2 mm and insert fused silica tubing
18. Smear epoxy around the fused silica at the pipette tip to provide an air-tight seal
19. Allow the epoxy to set overnight

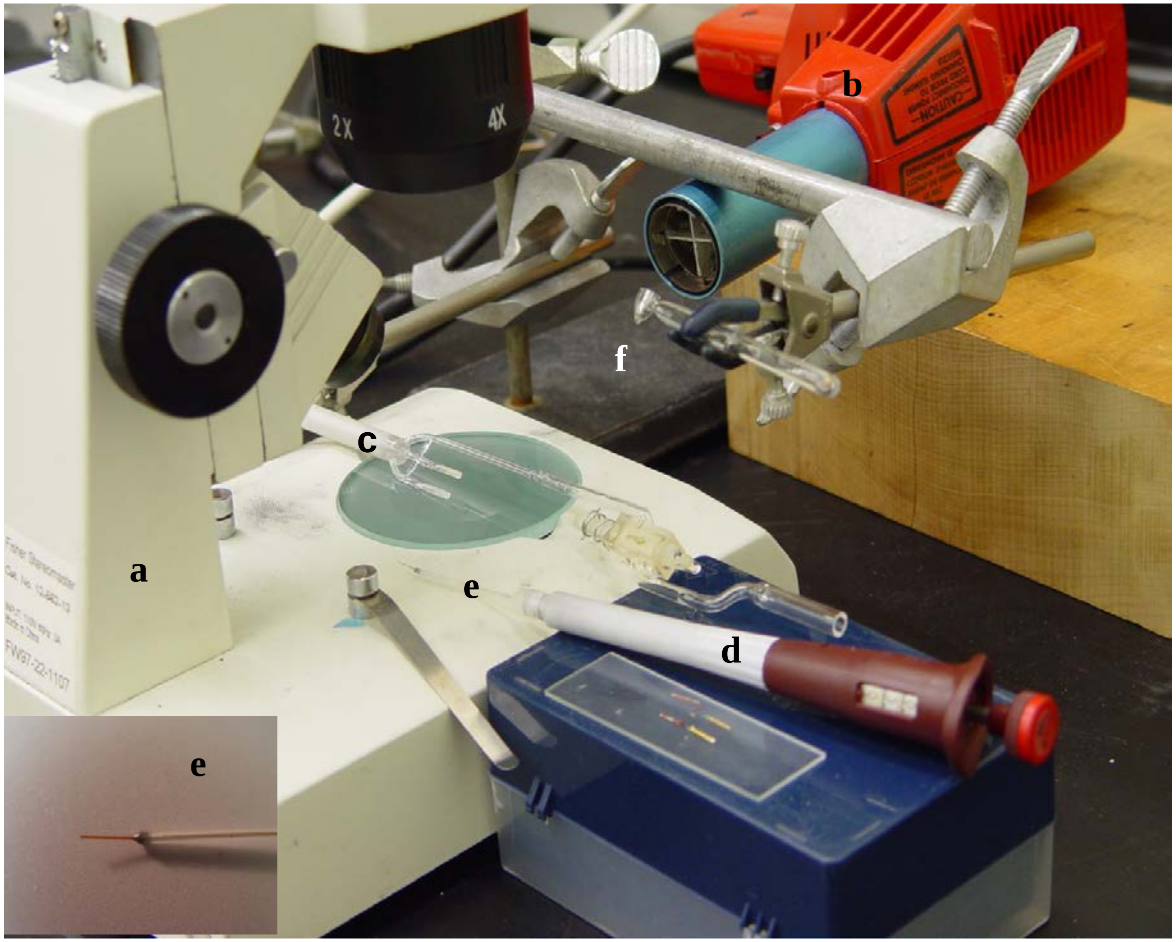


Figure 49 Tools and setup for Ga welding. Electrodes are Ga welded to their corresponding W-wire leads while observing the leads through a microscope. This is required since Ga can cool at room temperature solidifying the wire to the pipette breaking the wire when retracting the pipette. The necessary tools are identified as: **a.** microscope, **b.** heat gun, **c.** GPC, **d.** pipette, **e.** modified pipette tip, and **f.** ladle with Ga metal. The positive displacement pipette tip (**e**) is modified by epoxy seal of fused silica capillary tube (0.15 mm ID and 0.22 mm OD) for dispensing the Ga within the tube well.

Figure 50 Gallium welded electrical connectors. **a.** Photo shows the quartz to W-wire seals made of the anode wire (center tube) and the cathode-lead wire (right tube). Each tube end is filled with Ga using a pipette (Figure 49). **b.** Copper leads should be wetted with Ga before inserting in the tube well filled with Ga. **c.** Copper leads are then slowly pushed into each well. If a cradle is available, the counter can be fit to it to test if the leads are of suitable shape and length.

Miscellaneous Figures:

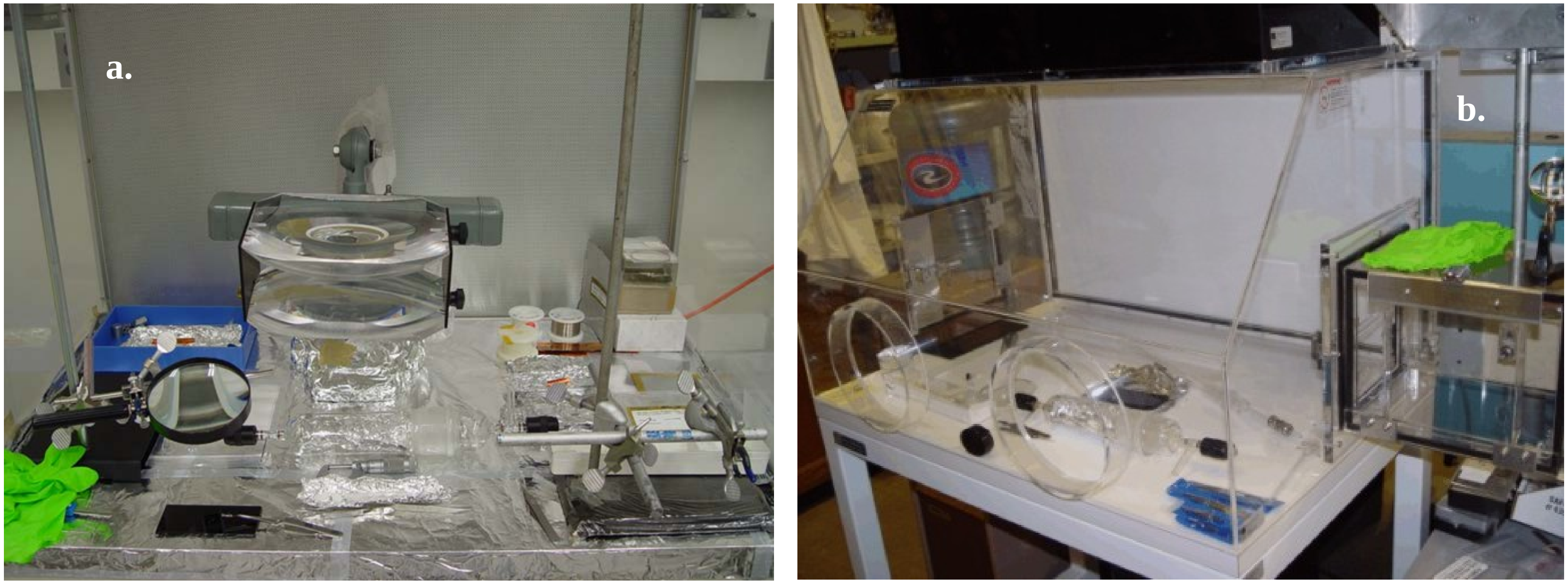


Figure 51 Clean bench for fabricating GPCs. a. A clean air bench is used in the laboratory to: store raw materials, roll the cathode, and outfit the GPC envelope with the anode and cathode (Figures 29 and 36). Some of the tools and equipment used in the assembly are shown, e.g. magnifying lenses, tweezers, ring stands, *etc*. **b.** A clean air bench is also available in the glass blowing shop to store GPC sub-assemblies, clean parts, annealed cathodes, and clean stopcocks. The side pass-through box is used to store partially complete or completed counters and cathodes in an argon atmosphere. A hand-carry size dry box similar to the pass-through box is used to transport counter assemblies in an argon atmosphere to and from the glass shop. When fitting the counter with a cathode and anode, air flow is shut down to minimize the effects of static charge.

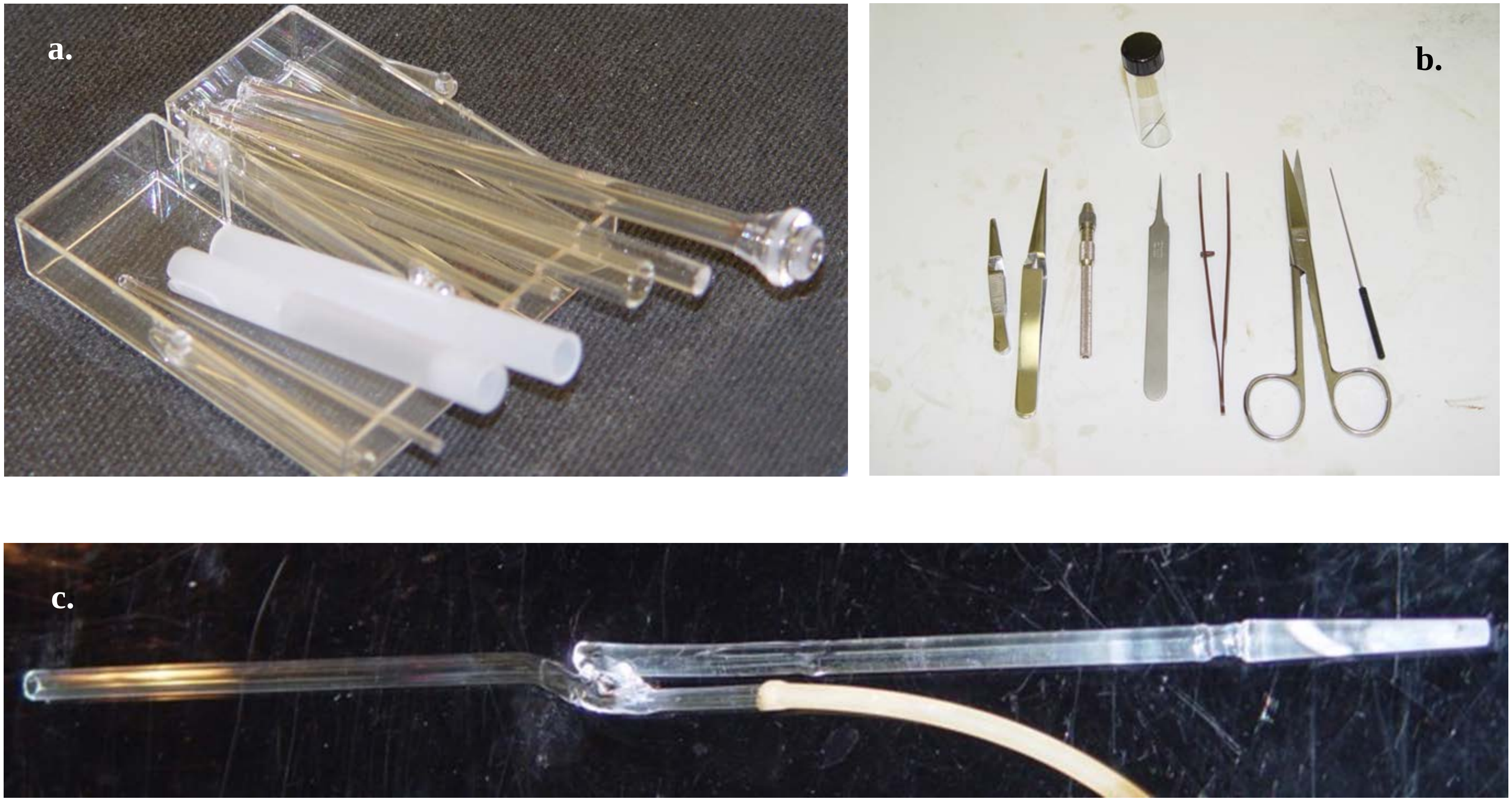


Figure 52 Jigs and tools. a. At the top left is shown counter bodies used to check the cathode dimensions, glass rods to roll Fe foil into cylinders for cathodes, glass tubes to support cathode while inserting into counter body, and tubing to guide tether wire through the high voltage capillary. **b.** Assorted tools are shown: *e.g.,* clamp tweezers, pencil vice, normal tweezers, scissors, needle-point probe and drill (in vial). **c.** The bottom photo is a jig used to align the stopcock barrel (right side tapper) to the center point of the GPC (left side to be chucked in lathe).

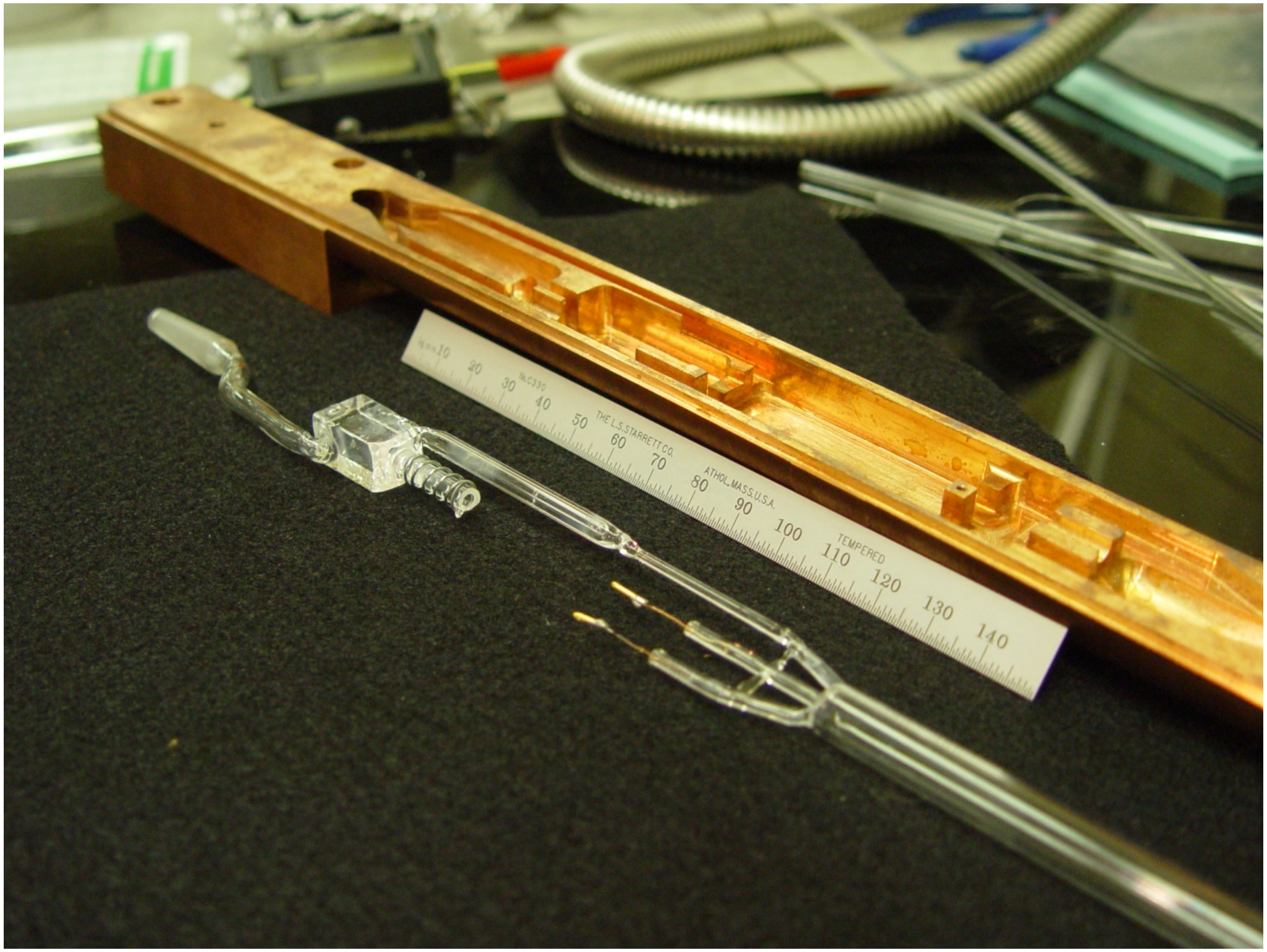


Figure 53 GPC mock up and Cu cradle. A mockup of a GPC is shown alongside a Cu cradle used to check the tolerances and fit of the GPC at each stage of the assembly process.

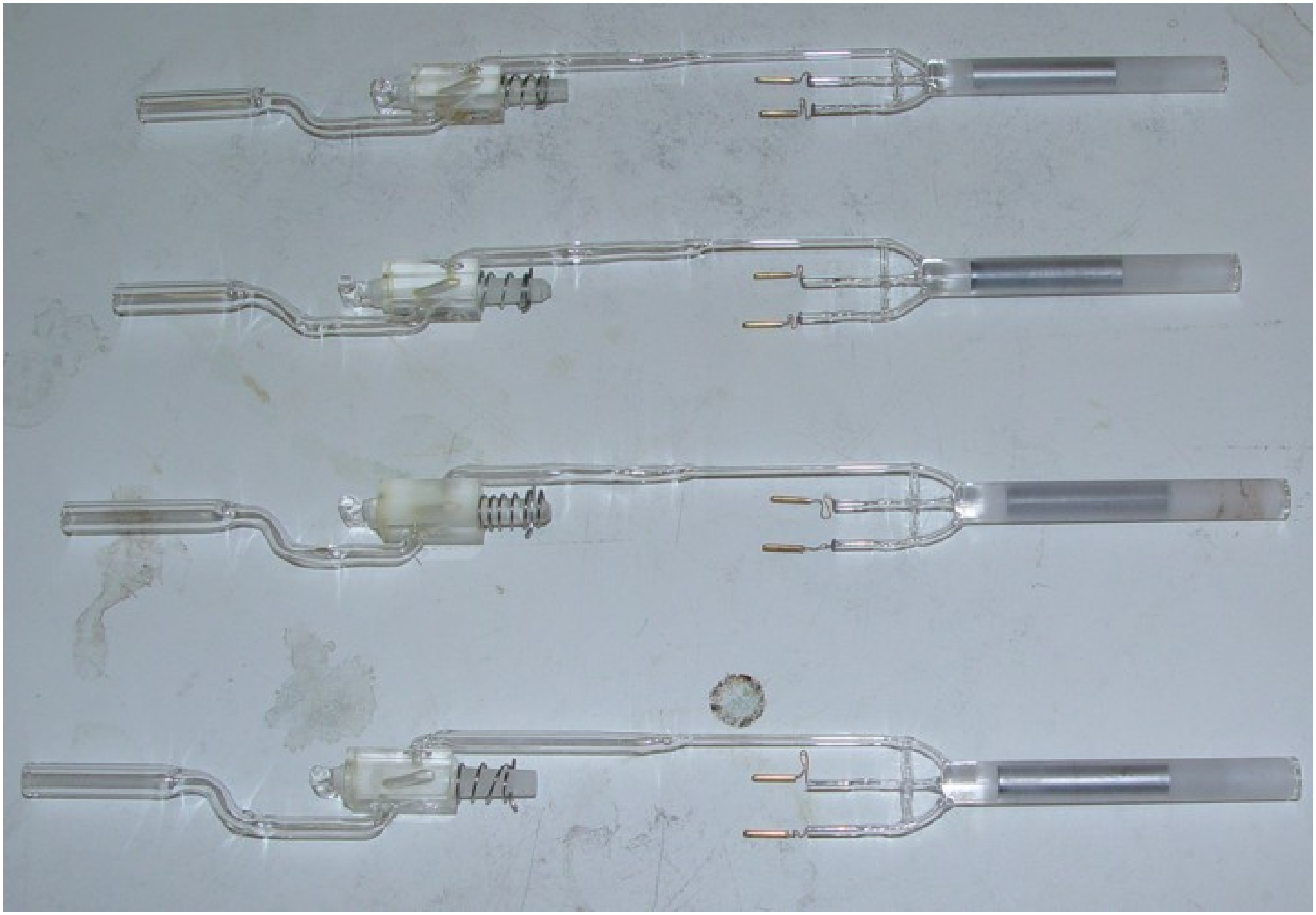


Figure 54 Replicate GPCs. Photo shows four GPCs that were completed within a few months of each other: GPC 19, 18, 15 and 20 (top to bottom). Their dimensions are closely replicated to meet the tolerances established by the cradle geometry.

V. OPERATIONAL TESTS AND CALIBRATION

The NIST *llc* Facility – established over four decades ago – provides the means to certify GPC operational characteristics under conditions that yield background count rates that are within the performance acceptance criteria. After extensive evacuation of the counter envelope to remove moisture, each assembled GPC is filled with P10 counting gas to a pressure of 100 kPa and tested for its operational characteristics and calibrated for energy. The following Figures provide details of the equipment and software capabilities of the NIST *llc* system as last updated in 2005. GPC background rates reported by Klouda and Filliben (2012) are unique to the NIST counting system configuration and calibration; though, one would expect to obtain similar GPC operating characteristics with comparably designed systems. For those unfamiliar with the counting process, two references that describe beta counting and that are likely to be useful to the reader are Knoll (2000) and Watt and Ramsden (1964). Other references that specifically describe the capabilities of the NIST *llc* system are Currie et al. (1983; 1998), Band et al. (2008), Klouda et al. (2008) and Klouda and Filliben (2013).

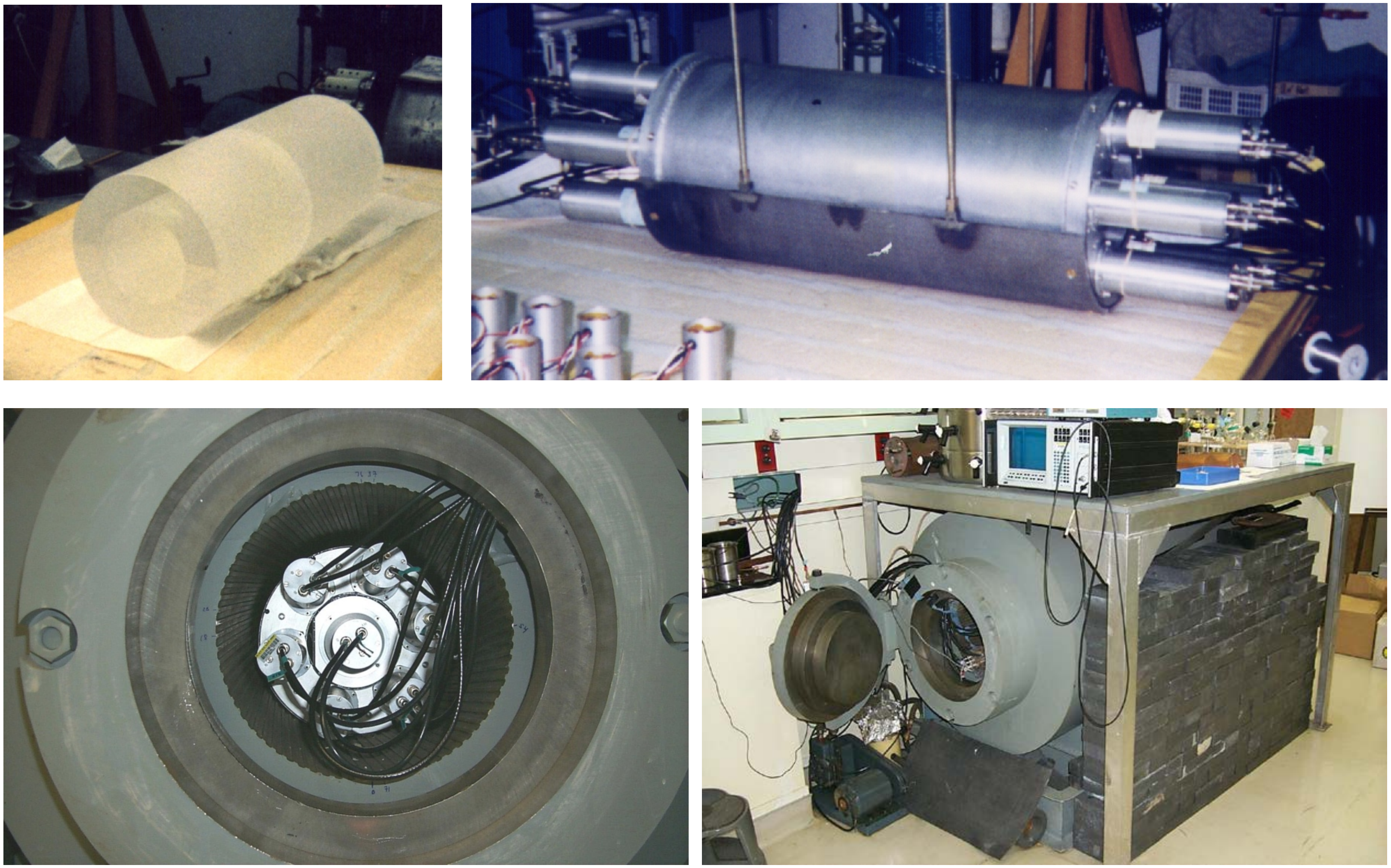


Figure 55 NIST *llc* system: active and passive shielding. NaI(Tl) coincidence (guard) detector specifications are comparable to HPGe γ-ray detection systems for anti-Compton suppression for high sensitivity (Heusser G, 1991). Passive shielding is a World War I gun barrel.

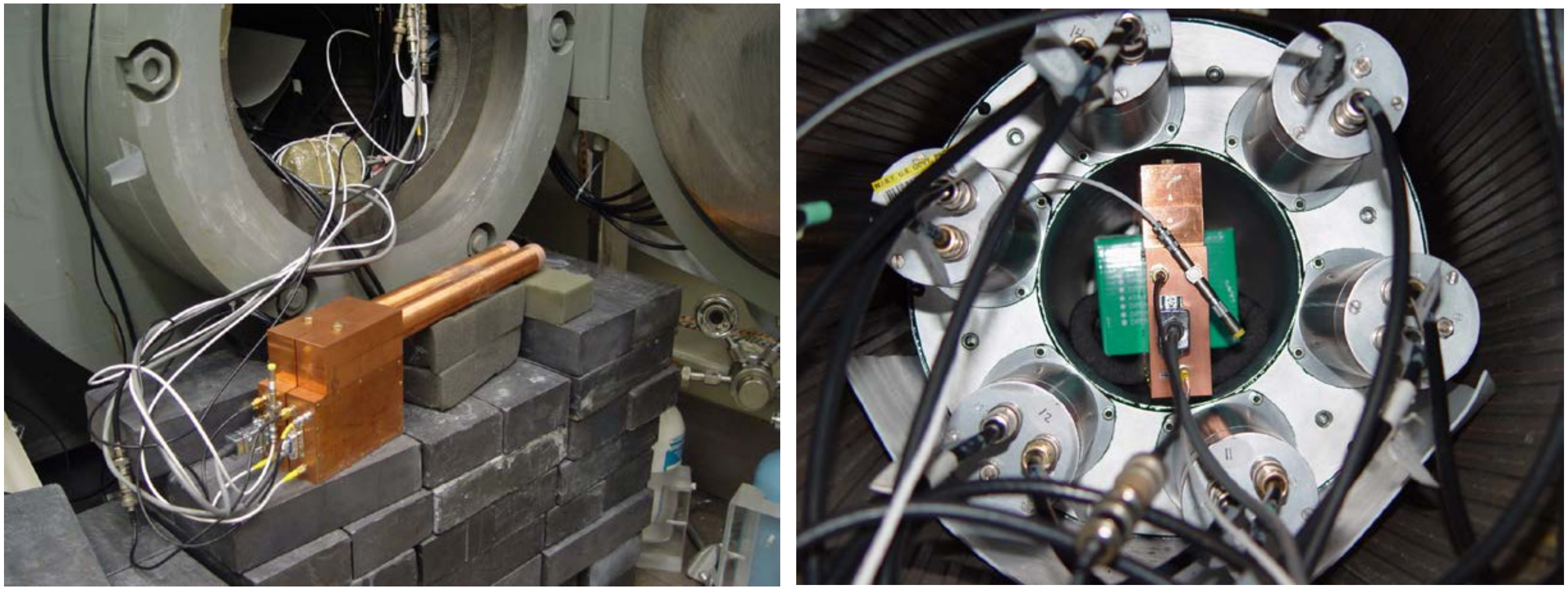


Figure 56 GPCs within Cu cradles and the NaI(Tl) coincidence guard ring. Two GPCs and their associated electronic circuits are housed in two Cu cradles (left) and placed within the NaI(Tl) coincidence guard ring (right). NaI(Tl) was manufactured with low background HP materials by Bicron.

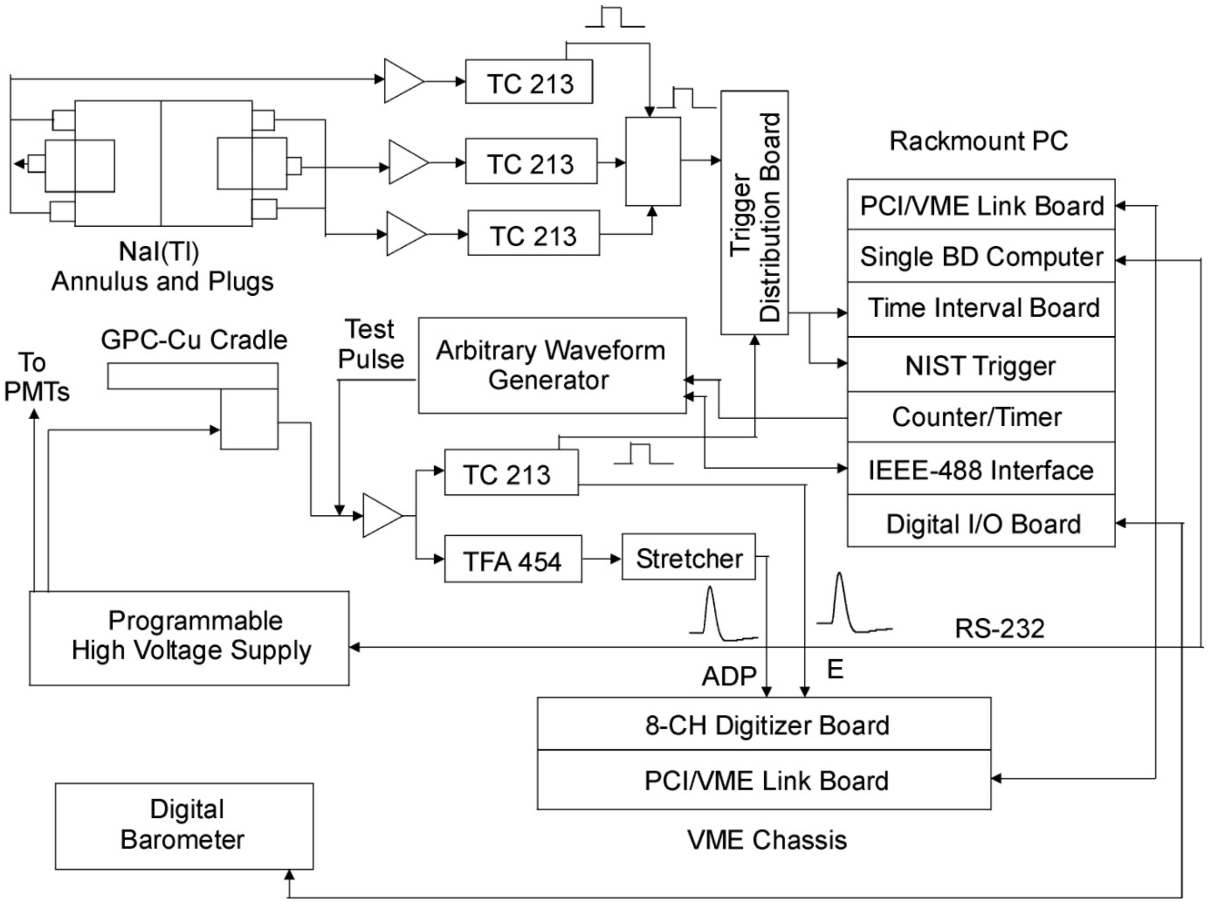
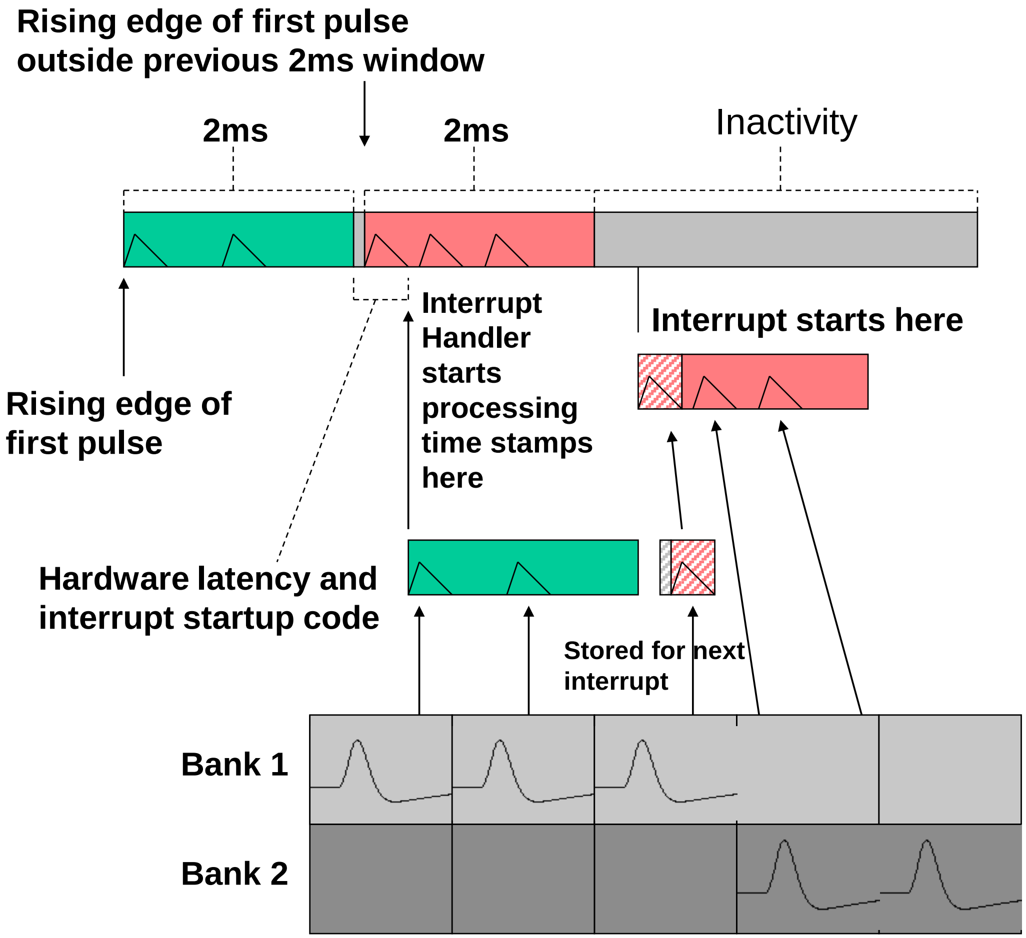


Figure 57 NIST *llc* system block diagram. A flow diagram is shown of the NIST low-level counting system hardware and electronics for pulse acquisition and analysis. For simplicity, the GPC and Cu cradle are displayed outside of the NaI(Tl) annulus.



Signal triggers 2 ms even window

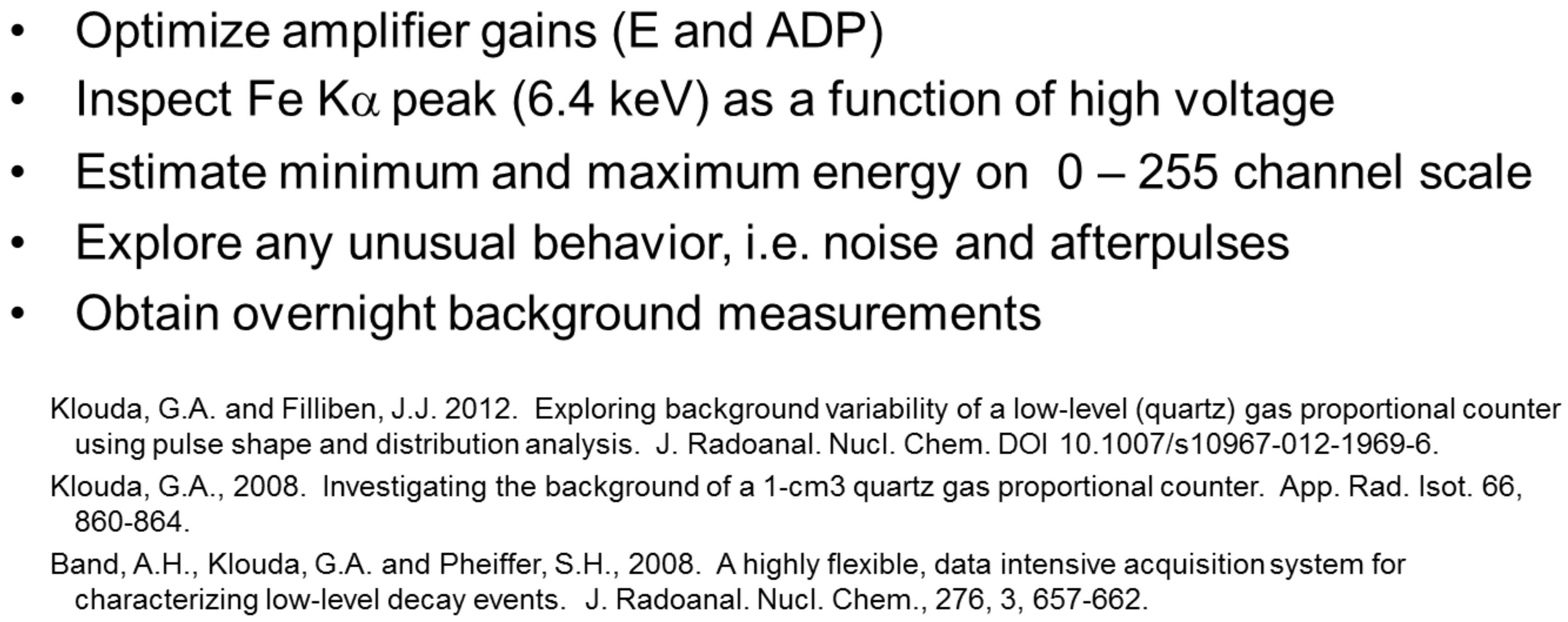
Subsequent activity within 2 ms is included in the current event window

At end of 2 ms-window, interrupt handler begins; simultaneously, a waveform for a subsequent 2 ms window can be digitized

Handler waits until digitization has stopped (status register bit test) before swapping memory banks

Handler stores extra time tags in special buffer, which is appended to the beginning of the data in the next interrupt.

Figure 58 Pulse processing algorithm. Digitizing pulses by buffering two deep.



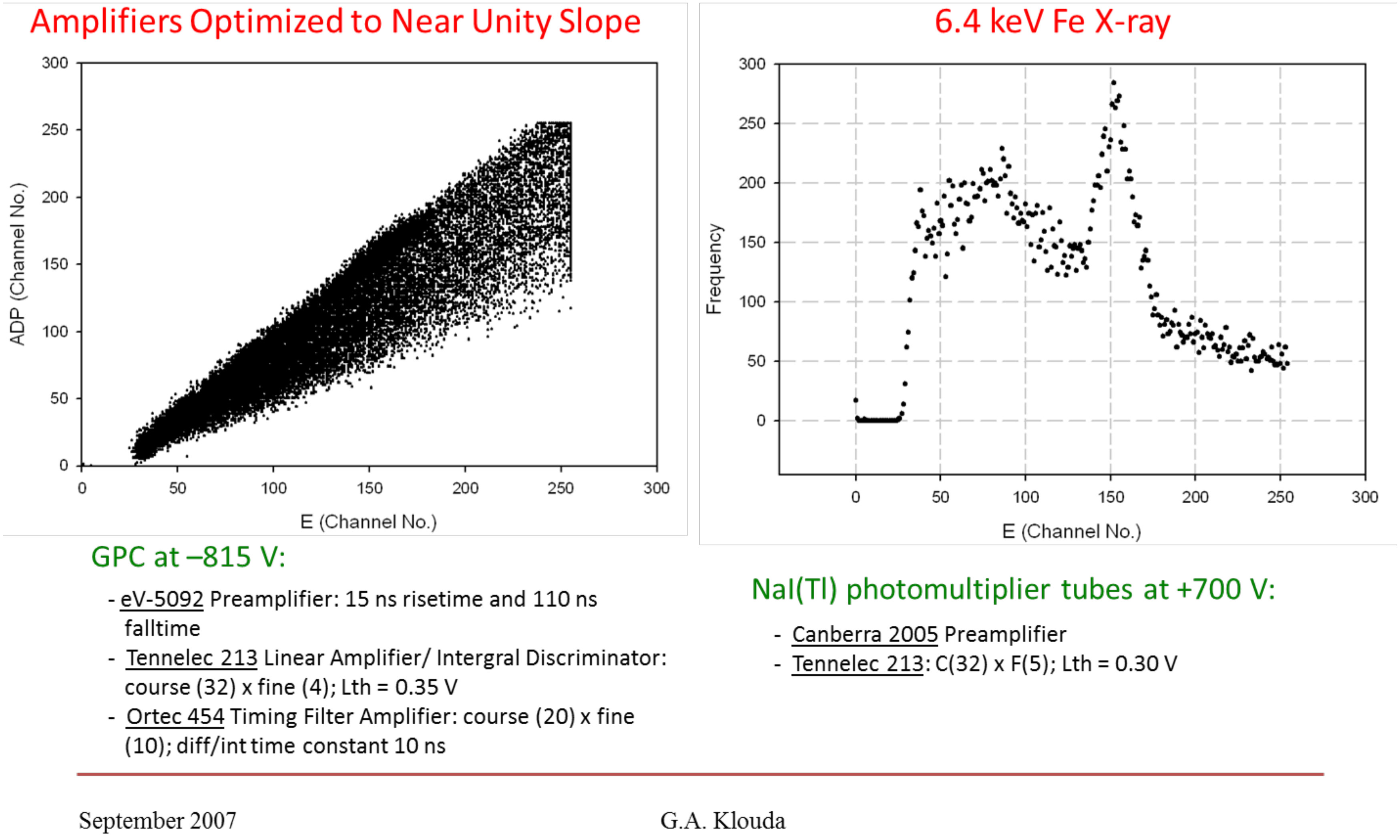
- Optimize amplifier gains (E and ADP)
- Inspect Fe Kα peak (6.4 keV) as a function of high voltage
- Estimate minimum and maximum energy on 0 – 255 channel scale
- Explore any unusual behavior, i.e. noise and afterpulses
- Obtain overnight background measurements

Klouda, G.A. and Filliben, J.J. 2012. Exploring background variability of a low-level (quartz) gas proportional counter using pulse shape and distribution analysis. J. Radoanal. Nucl. Chem. DOI 10.1007/s10967-012-1969-6.

Klouda, G.A., 2008. Investigating the background of a 1-cm3 quartz gas proportional counter. App. Rad. Isot. 66, 860-864.

Band, A.H., Klouda, G.A. and Pheiffer, S.H., 2008. A highly flexible, data intensive acquisition system for characterizing low-level decay events. J. Radoanal. Nucl. Chem., 276, 3, 657-662.

Figure 59 Protocol (outline) for screening GPCs. Energy calibration and background measurements protocol used to certify that each GPC operates as expected and yields a background count rate that is within the acceptance criteria (Klouda and Filliben, 2012).



Amplifiers Optimized to Near Unity Slope

6.4 keV Fe X-ray

GPC at –815 V:

- eV-5092 Preamplifier: 15 ns risetime and 110 ns falltime
- Tennelec 213 Linear Amplifier/ Intergral Discriminator: course (32) x fine (4); Lth = 0.35 V
- Ortec 454 Timing Filter Amplifier: course (20) x fine (10); diff/int time constant 10 ns

NaI(Tl) photomultiplier tubes at +700 V:

- Canberra 2005 Preamplifier
- Tennelec 213: C(32) x F(5); Lth = 0.30 V

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Figure 60 GPC energy calibration. For a GPC filled to 100 kPa with P10, the energy is calibrated by exposing the counter to an external ^{241}Am source. The left plot is a 2-dimensional representation of events described in inverse rise time (ADP) and energy (E), in arbitrary units of channel number. The right plot is the same count data displayed in terms of the frequency of occurrence as a function of E channel number (≈ 22 eV/Ch).

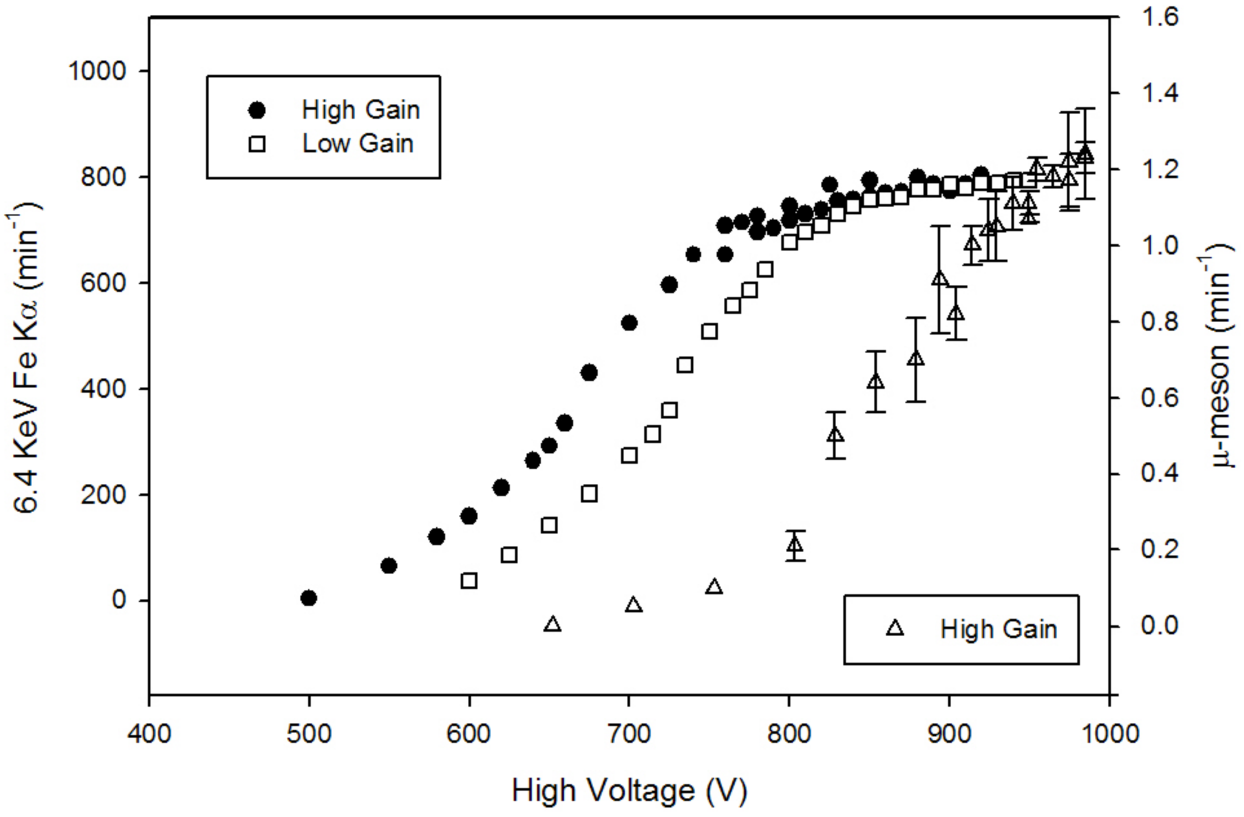
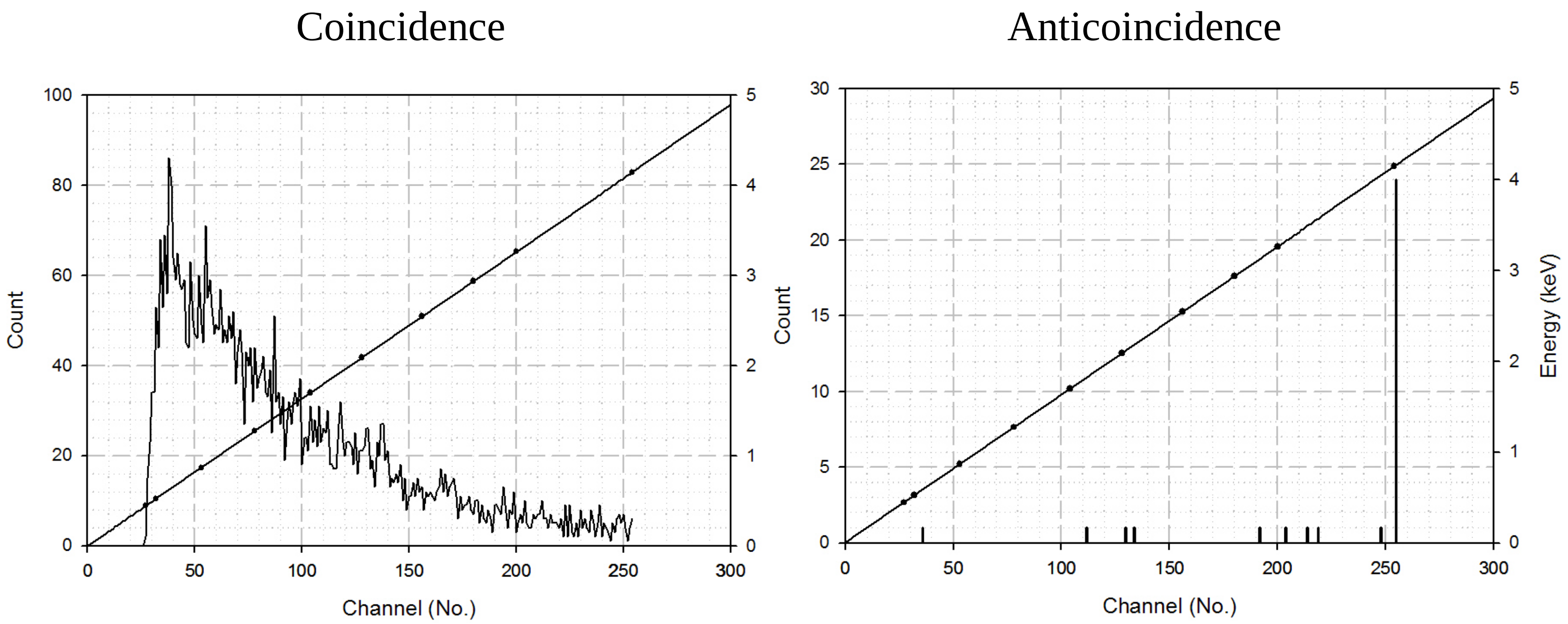


Figure 61 GPC plateaus. Plateaus of a GPC where pulses were generated by the interaction of ^{241}Am 59 keV gamma ray with the Fe foil cathode that produces Fe Kα x-rays (circle and square symbols; left ordinate) and cosmic ray μ-mesons (triangle; right ordinate) as a function of high voltage. Uncertainties are based on counting statistics and are only reported if significantly greater than their symbol size.



$\text{AC Rate} = (8.79 \pm 1.53)\ \text{d}^{-1}\ (\Delta t = 3.75\ \text{d})$

$\text{Coin Rate} = (1.07 \pm 0.01)\ \text{min}^{-1}$

Median μ-meson Channel = (98.32 ± 1.20) Ch.

Figure 62 Energy spectra. Background measurement of GPC59 at high gain (Channel number 25-256; 0.4 keV to 4.1 keV): μ-meson coincidence (Coin) counts as a function of channel number (left) and anticoincidence (AC) counts as a function of channel number (right). Energy (right hand axes on both plots) is obtained from the linear curves for a given channel number.

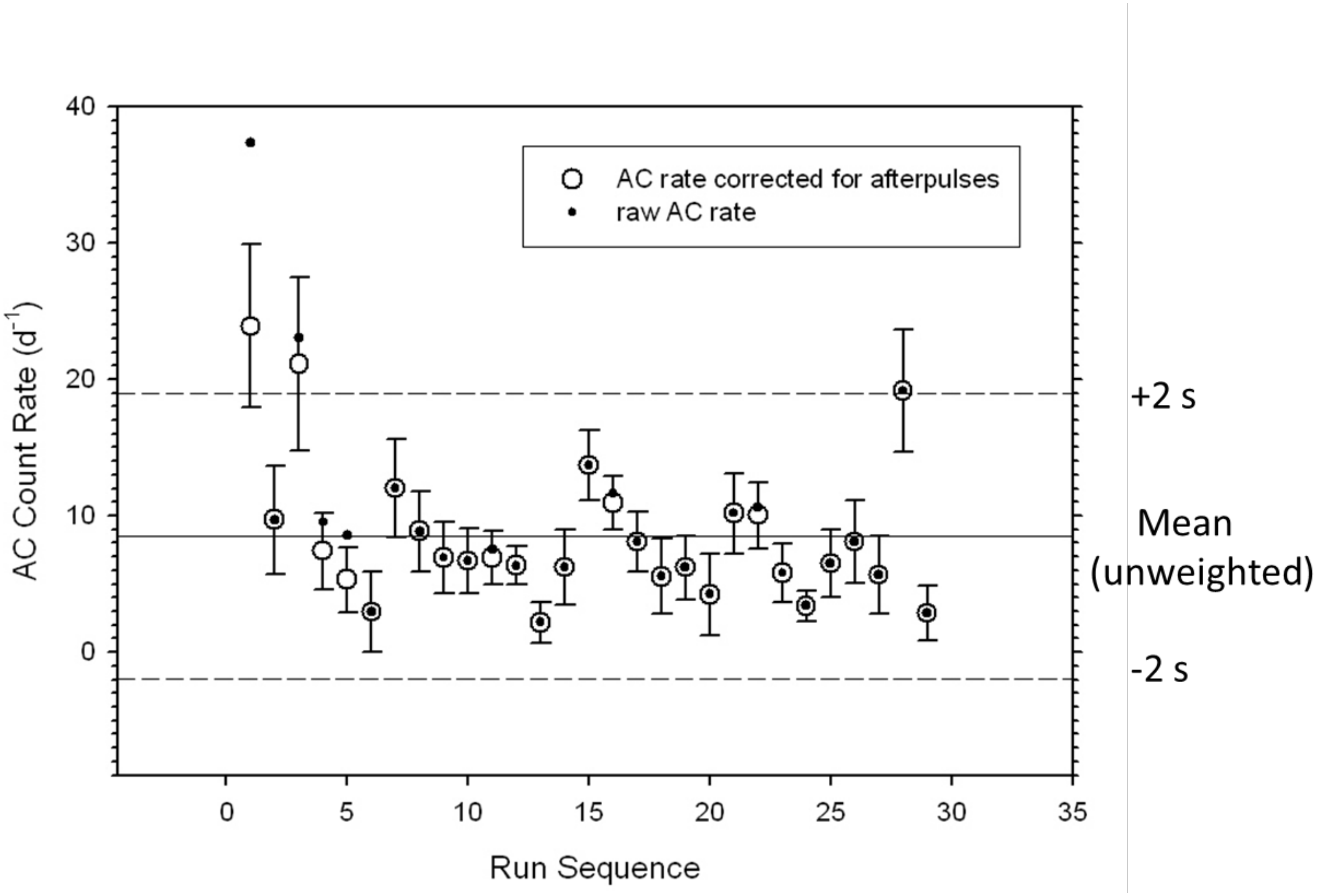
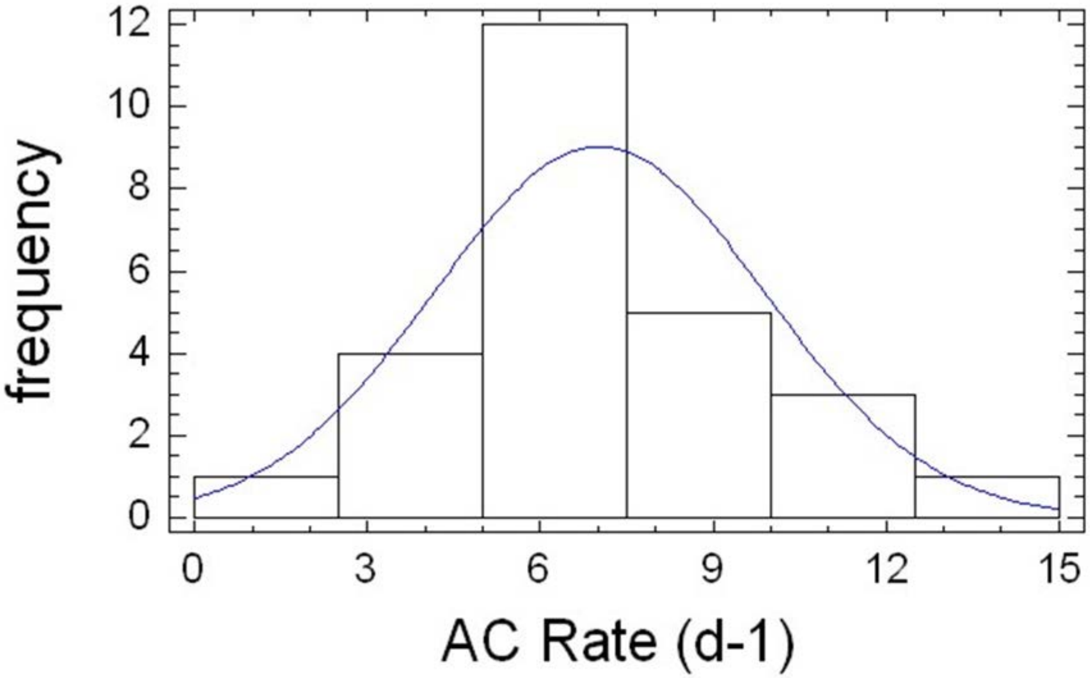


Figure 63 GPC52 anticoincidence (AC) background rate. Background measurements displayed in a time-ordered sequence of the same gas filling for at least 900 minutes each; total AC rate and corrected for after pulses indicate initial conditioning with higher background compared to lower and more stable count rate following a few days of operation. Unweighted mean of all data is 8.5 d^{-1} with a standard deviation of the day of 5.3 d^{-1}. There are three potential outliers observed beyond ± 2s.



- Grubbs’ test for outliers identified runs 1, 3, 28 as outliers
- Assuming normality, remaining observations fit a normal distribution at 5% α
- Unweighted mean and standard uncertainty is [7.02 ± 0.56 (s/√n)] d^{-1}, outliers omitted
- Weighted mean and standard uncertainty is [6.18 ± 0.44 ($1/\sigma^2$, 1σ-Poisson)] d^{-1}
- With outliers, weighted mean and standard uncertainty is [6.46 ± 0.43] d^{-1}

Figure 64 Frequency distribution of AC background. Data from Figure 63.

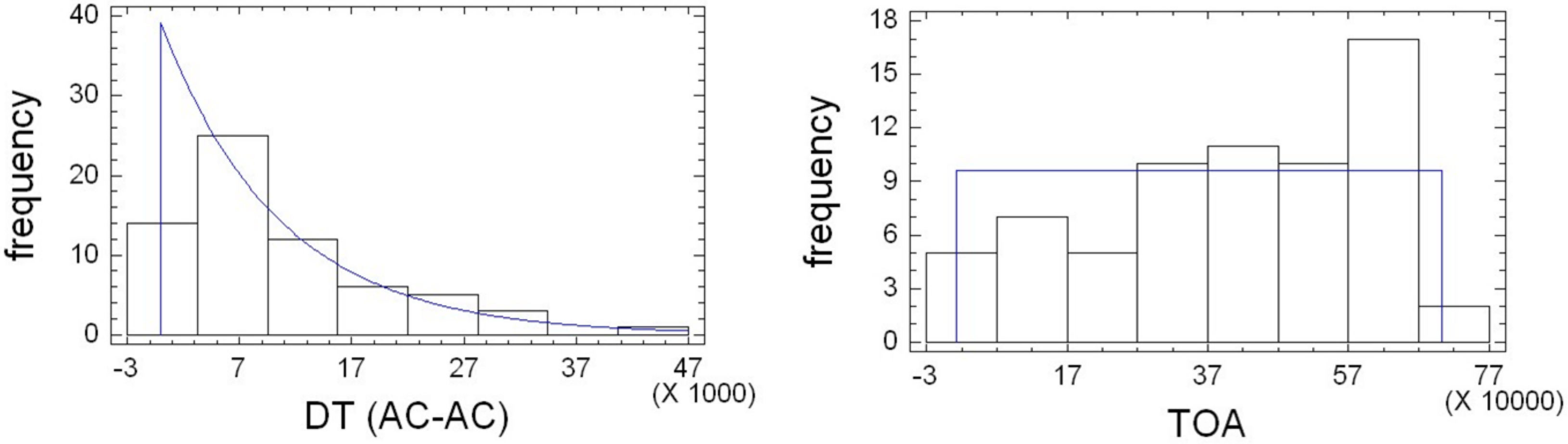


Figure 65 AC inter-arrival time (DT) and time-of-arrival (TOA). Distributions of background counts are shown from GPC51 for an 8.2 day period. The AC rate was (8.19 ± 1.00) d^{-1} and the COIN rate was (0.984 ± 0.009) min^{-1}. No after pulses were observed. Inter-arrival times (DT) are exponentially distributed at 90 % confidence with a mean of 10,517.9 s (8.2 d^{-1}). The time-of-arrival distribution failed tests for uniformity at 90 % confidence.

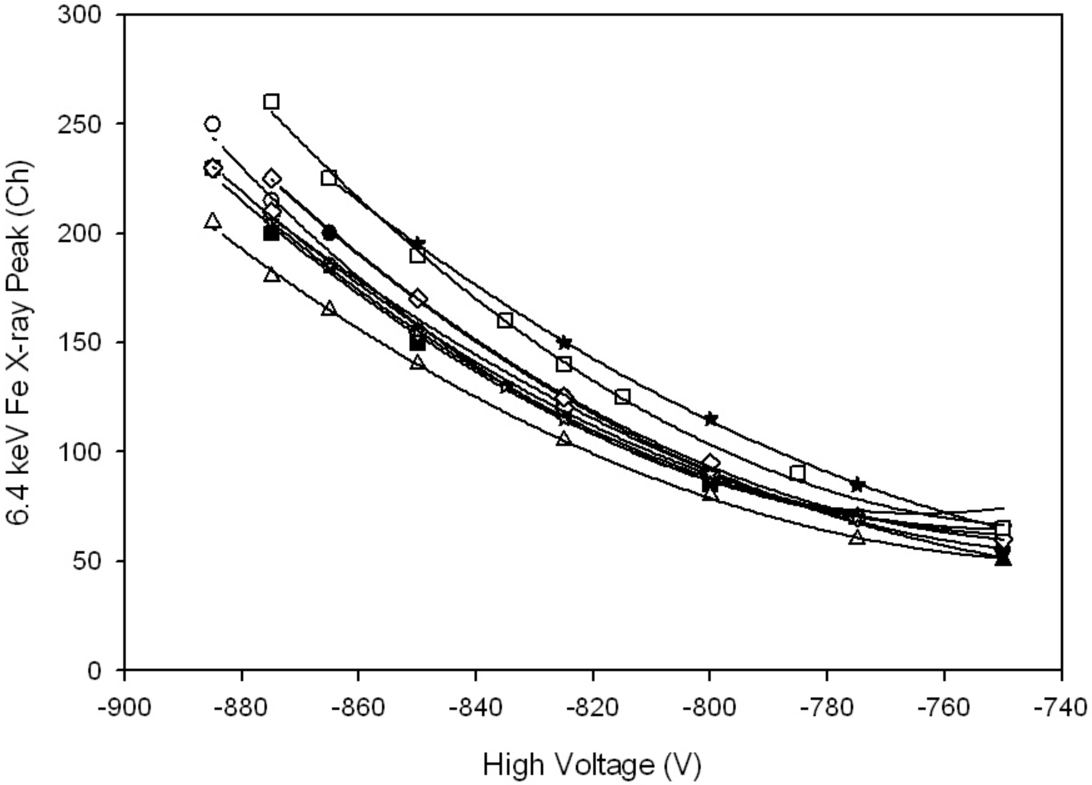


Figure 66 ^{241}Am calibrations (FY 2009). Each counter (symbol) was calibrated using a ^{241}Am source generating the 6.4 keV Fe x-ray peak while operating at voltages ranging from -750 V to -890 V. The low-energy threshold for these curves is about 0.5 keV (Ch. 25). Apart from three curves – triangle, open square and star – calibrations are similar.

VI. ACCEPTABLE PERFORMANCE

Acceptance criteria of future freshly assembled GPCs were obtained from background count rates of seventeen counters each operated up until a steady state rate is reached. The mean of the population was 14.0 counts per day (day^{-1}) with 95 % confidence limits of 11.2 day^{-1} to 16.8 day^{-1}. The tolerance limits of 95 % confidence and 95 % coverage are 2.5 day^{-1} and 47.8^{-1}. The results and statistical distribution are presented in Figures 67 and 68 (Klouda and Filliben, 2012).

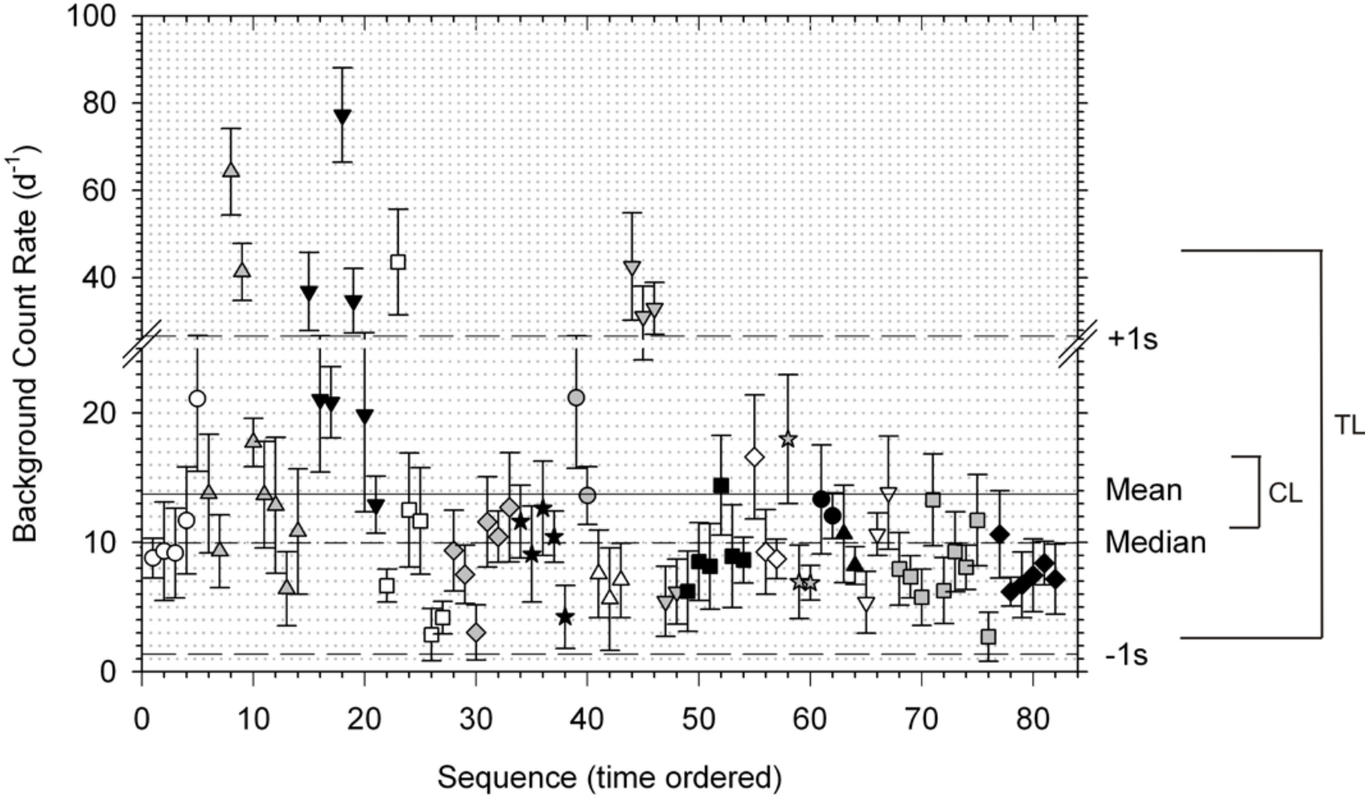


Figure 67 AC background count rates of 17 GPCs. Time ordered sequence plot of AC background count rate of either newly- or re- constructed GPCs (noted by a change in symbol) and their 1σ-Poisson uncertainty bar. Lines identify the mean (solid), median (dash-dot) and standard deviation of the data (± 1s, dashes). The inside bracket represents 95 % confidence limit (CL) and, the outside bracket represents 95 % confidence and 95 % coverage of the population tolerance limit (TL).

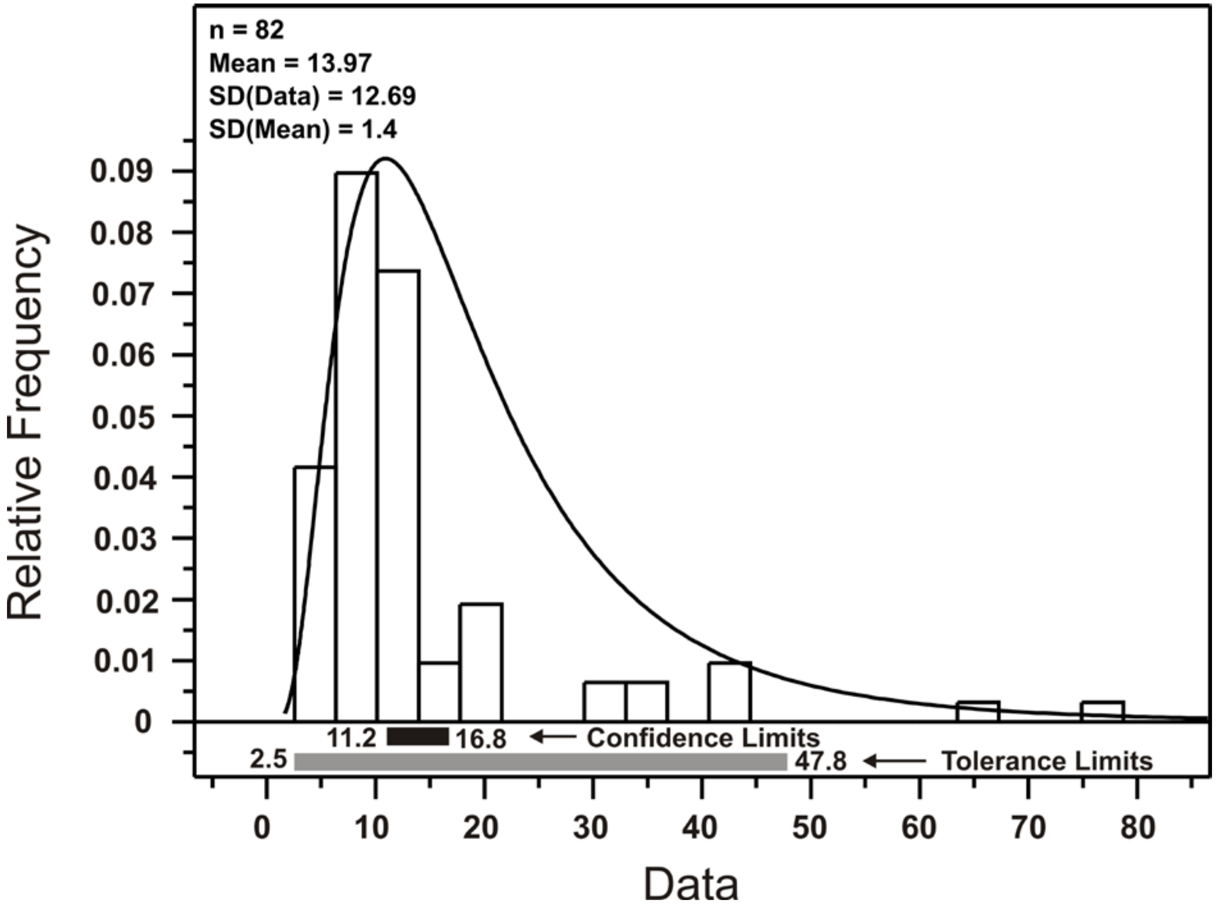


Figure 68 Statistical distribution and limits of AC background. Raw data histogram (*n* = 82 from Fig. 67), mean, standard deviation of the data and standard deviation of the mean in counts per day (upper left corner); overlay of transformed normal curve back to original space. Raw background count rate of 95 % confidence limit (*black rectangle*) and 95 % coverage of the population tolerance limit (*gray rectangle*) are indicated on the *abscissa*.

VII. SUMMARY SPECIFICATIONS

1. GPC is of similar design to the HD-2(Fe) type GPC reported by Wink et al. (1993).
2. Design drawings and specifications include tolerances that allow for the GPC to fit within a specified housing (cradle) that contains circuitry required to operate the GPC.
3. All materials used to produce GPCs are screened for their radio-purity by high-purity germanium counting (Keillor et al, 2009).
4. The GPC envelope is made of quartz (synthetically fused, #1 grade, Suprasil) rod that represents the highest purity available. Since quartz #1 is only available in solid rod, the counter components are precision ground from this material. The envelope consists of:
    - a cylindrical body
    - a front (solid) plug with one channel (groove) for the high voltage lead, one groove for the gas inlet, and a center bore hole for the anode wire
    - a rear (solid) plug with three bore holes for evacuating and a center bore/wedge to accommodate a spring to anchor the anode wire
    - and, a rear window (slide)
5. Prior to GPC assembly, all quartz components are cleaned using: (1) a detergent, (2) a series of acid treatments, and (3) quartz-distilled-water rinses between steps. The final cleaning is a high temperature (700 °C) oxidation.
6. A technical glassblower seals quartz Suprasil 300 tubing to the front plug to provide passages for the high voltage lead, the anode lead, and the connection between the GPC envelope and the stopcock.
7. The GPC cathode is made of HP Fe foil, cut to specifications, rolled, annealed, and re-rolled to a concentric cylinder and brightened. Prior to the final rolling, a W burr is spot welded to the cathode to provide a mechanical contact with it and the high voltage lead.
8. Insertion of the cathode (Fe foil) and the anode (W wire) into the GPC body is carried out in a clean bench area that provides air filtered to remove > 0.3 μm diameter particles.
9. A quartz (#1 grade) stopcock is sealed to the counter; configured to fit within the Cu cradle housing.

10. All quartz seals are made using a H_2/O_2 flame to produce a hermetic (GPC) envelope that sustains a vacuum at < 0.1 Pa.
11. Moisture is removed from the completed GPC by placing the counter in a vacuum chamber and evacuating the chamber using a high vacuum He cryopumping system.
12. Gas impurities within the GPC are determined using a residual gas analyzer to detect any trace gases remaining in the GPC envelope down to < 0.1 Pa.
13. A vacuum leak test is performed on the GPC to certify the integrity of the hermetic seals.
14. The GPC is capable of containing a gas sample at a pressure of 100 kPa for several weeks without leakage.
15. For a GPC filled with 100 kPa of P10 counting gas (90% Ar, 10% CH_4), the operating voltage in the proportional region is (- 930 ± 10) volts which translates to a lower energy threshold of 500 eV with saturation at 5.0 keV considering the NIST (*llc*) counting system; all events > 5.0 keV are counted in a saturated channel. System energy is calibrated using an external radioactive source of 10 μC of ^{241}Am.
16. The GPC has a background count rate of no greater than approximately 0.01 anticoincidence counts per minute under the above stated conditions using the NIST *llc* Facility.

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IX. APPENDIX

1.1 Reagents

- HP forming gas
- H_2 gas
- O_2 gas
- Ar gas
- He gas
- Quartz distilled water
- HP concentrated HNO_3, HCl, and HF (Seastar Chemicals Inc.) all purchased and stored in PTFE (virgin) Teflon bottles
- Trichloroethylene
- Ethanol
- Methanol

1.2 Lab Ware

- Gilson Microman 250 Positive-Displacement Pipette; CP10 tips
- Teflon bottles for acids (min 250 mL)
- Teflon bottles for distilled water (1 L)
- Teflon beakers (200 mL)
- Glass rod and tubing; specified size to roll cathode to ID of body
- Fused silica capillary (0.22 mm ID, 0.32 mm OD)
- Watlow tube furnaces
- Variac
- Thermocouples
- Ring stands, clamps
- Scissor jacks
- High temperature ceramic fiber
- Al foil

1.3 Specialty Tools

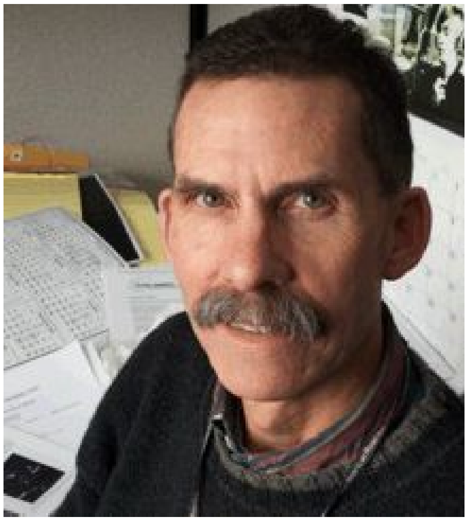
- Micro-welder (spot welder)
- Glass blower lathe
- Micro torch
- Ultra-high vacuum (UHV) manifold, He cryopump, calibrated volume

- Pirani or ion gauge
- Capacitance manometer (133 kPa)
- Residual gas analyzer (RGA) on ultra-high vacuum manifold
- Redressing tool for grinding stopcock plug to match barrel
- Stainless steel tweezers of various tips
- Teflon tweezers
- Soldering gun
- Heat gun
- Microscope
- Semi-analytical balance

X. AUTHOR BIOGRAPHY

Mr. Jeffrey R. Anderson. A glass fabricator for forty years, Jeff spent the first ten in private industry, and the subsequent thirty in highly specialized research fields within the federal government. Entities that have utilized Jeff's services include NIST, NASA, Air Force, ARL, NRL, NIH, DOE, DOD, in all cases, the glass instruments and components Jeff fabricates are inimitable in design and frequently made from exotic and challenging materials.

Mr. John (Jack) E. Fuller. Jack worked 12 years with a small scientific glass shop engaged primarily in fabricating prototype optical cells, laser tubes, and custom glassblowing. He started his career at NBS/NIST in June 1976 by providing many types of optical fabrication services, and using optical processes to work with a wide range of materials.



Mr. George A. Klouda. With formal training in chemistry and geological sciences, George started his career at NBS/NIST in 1978 and spent the next twenty seven years applying low-level radiocarbon decay and atom counting techniques to answer questions related to the transport of atmospheric carbonaceous gases and particles. He used carbon isotope measurements of select organic fractions or individual compounds to determine their fossil and biogenic source attributions. Since 2005, he has focused on leading the team in fabricating gas proportional counters and providing support in the area of laboratory quality management for the NIST Analytical Microscopy Group.