Pacific Northwest
NATIONAL LABORATORY

PNNL-37304

Quartz Ampoule Loading, Sealing, and Inspection

January 2025

Erin C Morrison
Charles Doll
Brian J Riley
Michael J Schweiger

U.S. DEPARTMENT OF ENERGY

Prepared for the U.S. Department of Energy
under Contract DE-AC05-76RL01830

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Richland, Washington 99354

Abstract

This procedure applies to the loading of materials into quartz ampoules and flame sealing these ampoules. Flame-sealed quartz ampoules are commonly used as a sealed containment for neutron irradiation. Due to the importance of maintaining containment for this application, this procedure involves a double encapsulation design wherein a sealed inner ampoule is in turn sealed within a larger diameter outer ampoule. General guidance for inspection of ampoules by destructive, non-destructive, qualitative (e.g., visual), and other methods is given. Materials to be placed inside the ampoules may consist of stable element materials or radioactive materials in either solid or liquid form. Liquids shall be dried prior to sealing the ampoule.

Safety and Hazard Mitigation

The user is responsible for complying with applicable industry and facility requirements and controls. General safety and hazards information is provided in this section to assist the user in identifying relevant requirements.

Sealing of a practice ampoule prior to sealing a batch of loaded ampoule(s) is recommended to establish quality of the seal before work. In addition, routine sealing of ampoules (several times per year) is recommended to maintain skills in sealing ampoules. Appropriate training is considered a hazard mitigation tactic due to the potential for hazardous conditions during sealing and in the event of handling an ampoule with a failed seal.

Laboratory safety rules shall be observed. Appropriate Personal Protective Equipment (PPE) shall be worn.

Hazards and their controls are listed to assure that proper safety measures are used to mitigate the hazards:

- Radiological – If the ampoule is to be prepared with radioactive materials, staff performing work described in Section 3.2 – Section 3.4 of this procedure must be appropriately trained to work with radioactive materials.
- Chemical - All work with chemicals will comply with the applicable safety documentation. Staff shall be familiar with applicable Safety Data Sheets (SDS's) and the hazards associated with working with hazardous materials or chemicals.
- Physical –
    - Potential Airborne Emissions – Quartz sealing activities must be conducted within a fume hood.
    - Work with open flame, hot surfaces – Staff performing work described in Section 3.4 must be trained to work with open flame and use appropriate PPE. Contact your Health and Safety Professional for PPE requirements.
    - Potential Eye Flash Burn – While fusing quartz, ultraviolet light is present. Contact your Health and Safety Professional for PPE requirements.
    - Sharp Surfaces – When preparing sealed fused quartz ampoules for the next steps (i.e., inner ampoule for outer ampoule, outer ampoule for shipping container) the process requires the quartz to be scribed and broken, which will expose the user to very sharp surfaces. To prevent cuts and/or scratches, hand protection must be worn during these steps to prevent injury and possible contamination.

Acronyms and Abbreviations

ID – Inner Diameter

OD – Outer Diameter

PNNL – Pacific Northwest National Laboratory

PPE – Personal Protective Equipment

PSI – Pounds Per Square Inch

PSIG – Pounds Per Square Inch Gauge

SDS – Safety Data Sheets

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1.0 Prerequisites

Fused quartz and fused silica sealing surfaces must be maintained in a clean condition free from debris and fingerprints otherwise the integrity of the seal may be compromised. Mitigation steps are identified within the procedure. These two materials (i.e., quartz and silica) should not be mixed interchangeably when sealing, e.g., a fused silica ampoule should not be welded to a fused quartz end cap, to avoid bubbles at the sealing surface.

Individuals shall complete training appropriate to their role in the procedure, e.g., handling radioactive materials, work with open flame, handling sharp objects.

Sealing of a practice ampoule prior to sealing a batch of loaded ampoule(s) is highly recommended to establish quality of the seal before work as well as refresh the operator with the procedure before working with the (valuable and potentially hazardous) sample.

2.0 Equipment and Materials/Reagents

2.1 Ampoule Loading

The list of parts and equipment needed for ampoule loading are summarized as follows:

- Quartz ampoules. The selection of appropriate ampoule size and materials is important to the success of the application.
  - Ampoules that will be irradiated will be fabricated from Suprasil® 300 (fused silica), GE-214 (fused quartz), or equivalent-grade fused silica or fused quartz tubing. Use of high-purity fused quartz or fused silica minimizes activation of impurities during irradiation.
  - Custom commercially prepared ampoules prepared from nominally 4-mm inner diameter (ID), 6-mm outer diameter (OD) fused quartz and 7-mm ID, 9-mm OD fused quartz have been tested. Diagrams of the ampoules commonly used at PNNL are provided in Figure C.1 and Figure C.2 (see Appendix C).
  - The endcap used to close off the open end should fit the interior of the ampoule with < 2-mm slack in any direction.
  - Other tube compositions and ampoule sizes may be approved following completion of appropriate testing to demonstrate the performance of the tube material under the intended use conditions. For example, use for irradiation would require testing leak rates of the sealed end. Use in an oven would require thermal testing.
- Magnifying glass or dissection microscope
  - Optional: additional equipment can be used to aid in identifying hairline fractures or imperfections during inspection
- Aluminum foil
- Hot plate for drying liquids.
- Transfer pipet to transfer liquids into the ampoule.
- Spaghetti pipet tips
- Beaker (50 mL)
- Analytical balance. This is a 4-digit, calibrated balance capable of measuring mass with an accuracy of four decimal places (e.g., 0.0001 grams).
- Funnel for loading solids into the ampoules.
- Quartz wool
- The PPE can vary depending on the application but requires eye protection and hand protection at a minimum. Contact your Health and Safety Professional for PPE requirements.Nitric acid (2%). Add 2 mL of concentrated Ultrex® or Optima® grade nitric acid to 98 mL of 18.2 MΩ-cm deionized water and mix well.

2.2 Ampoule Sealing

The list of parts required for ampoule sealing are summarized as follows, with example equipment shown in Figure 1:

- Pressure manifold
- Nitrogen gas—If desired for evaporating liquid or backfilling ampoule. The appropriate purity for the application may be selected by the cognizant scientist.
- Tygon tubing (gas transfer lines)
- Stiff tubing for vacuum line (may be any material that does not collapse under vacuum)

- Oxygen-propane torch – supplied with 15 pounds per square inch (PSI) oxygen and 5 PSI propane
    - Torch stand
    - Torch tip
    - Spark lighter
- Silicone o-ring (ph.parker.com)
- Oxygen supply bottle or house oxygen supply, with regulator
- Propane supply bottle or house supply, with regulator and flame arrestor
- Vacuum pump or building-supplied vacuum
- PPE—The PPE can vary depending on the application but requires eye protection and hand protection at a minimum. Contact your Health and Safety Professional for PPE requirements.
- Magnifying glass or dissection microscope
    - Optional: additional equipment can be used to aid in identifying hairline fractures or imperfections during inspection
- High-hardness (on Mohs scale) scoring tool for aiding in breaking off unwanted ends of quartz tubes. The most common options for this is a diamond or a tungsten carbide (WC) scoring tool.

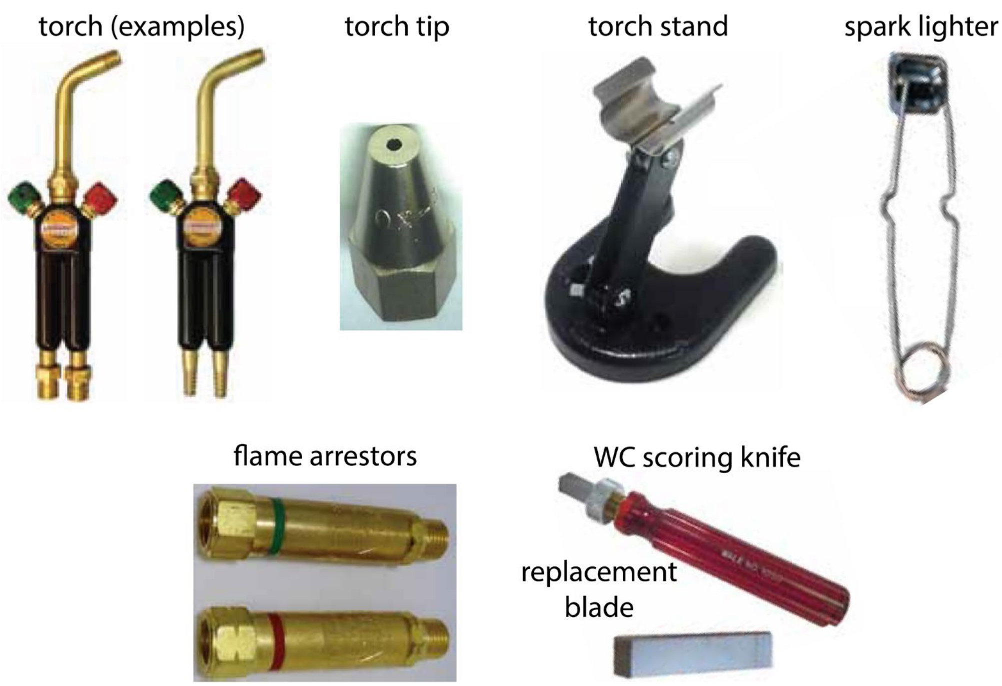

Figure 1. Pictures of equipment listed above including examples of oxy-propane torches, a torch tip, a torch stand, a spark lighter, flame arrestors, and a WC scoring knife.

3.0 Work Instructions

Work instructions are divided into sub-sections. Activities in each sub-section may require different expertise or training of the individual performing the action.

3.1 Preparation of Ampoules Prior to Sample Loading

1) Obtain quartz ampoules that are appropriate to the desired application (explanation in Section 2.0 EQUIPMENT and MATERIALS/REAGENTS above).

2) If the ampoules have not been previously cleaned, leach ampoules by submerging overnight in 2% nitric acid and rinsing with 18.2 MΩ-cm deionized water.

3) Air dry the ampoules on a clean towel. Lay a Kimwipe® over the top of drying ampoules to minimize exposure to dust or lay the ampoules out in a laminar flow hood if available.

4) Handle clean ampoules with Nitrile® gloves to keep surfaces clean. Note that placing the cleaned end caps inside plastic bags in the past has sometimes resulted in unwanted coatings on the caps so, if the tubes/caps are to be stored following the cleaning process, place the cap into the ampoule and then place the ampoule into a container. This will help keep the pristine surface of the cap free from contamination.

ATTENTION: QUARTZ AND FUSED SILICA SEALING SURFACES MUST BE MAINTAINED IN A CLEAN CONDITION FREE FROM DEBRIS AND FINGERPRINTS OTHERWISE THE INTEGRITY OF THE SEAL MAY BE COMPROMISED.

ATTENTION: STORE QUARTZ AND SILICA MATERIALS SEPARATELY IN LABELED CONTAINERS. THE TWO MATERIALS ARE VISUALLY IDENTICAL. ATTEMPTING TO SEAL THE TWO MATERIALS TOGETHER RESULTS IN BUBBLES AT THE SEALING SURFACE.

5) Staff shall inspect ampoules before loading to determine if there are any obvious imperfections or cracks, usually indicated by a whitish line. The body and factory sealed end of the ampoule should be completely clear and colorless before use. A magnifying glass may be used to facilitate this examination. Dispose of ampoules with defects.

3.2 Loading Ampoule with Liquid Sample

Liquid samples containing stable or radioactive materials may be loaded into an ampoule directly or dried onto a small piece of aluminum foil that is then inserted into the clean, dry ampoule. Processes are described below.

ATTENTION: GO TO SECTION 3.3 IF LOADING AMPOULE WITH A SOLID SAMPLE.

1) Tare an empty beaker or other appropriate container on a 4-digit analytical balance (which will be used to hold up the quartz ampoule) and weigh the foil or clean quartz ampoule in the container.

2) To avoid contaminating the ampoule-sealing region (above the dimple on the ampoule) with debris, use a long pipette tip or place a funnel extending below the sealing surface into the cavity of the quartz ampoule prior to loading material into ampoule.

3) Solution may be transferred directly to the ampoule or prepared as a dried deposit on a small piece of aluminum foil before insertion into an ampoule.

ATTENTION: MAKE SURE THAT ALL MOISTURE IS EVAPORATED. REMAINING MOISTURE INSIDE THE AMPOULE CAN VOLATIZE DURING SEALING AND RUPTURE THE AMPOULE.

a) To transfer solution directly to the ampoule: use a calibrated transfer pipette with a spaghetti pipette tip to transfer the liquid sample into the quartz ampoule, making sure that the bead of solution only touches the bottom of the ampoule, not the sides. Remove the funnel and re-weigh the ampoule to obtain the mass of solution delivered. Evaporate liquid in the ampoule by wrapping the quartz ampoule with aluminum foil and heating on a hot plate while a gentle stream of nitrogen is flowing into the quartz ampoule. The hot plate should be set to a medium-low heat (approx. 150-200°C). Alternatively, an oven may be used to evaporate the liquid. Rinse the inside of the quartz ampoule with small aliquots of 2% HNO_3 to wash any debris from the walls of the ampoule, then continue dry down. By either method, expect the dry down to take several hours.

   If the ampoule is to be moved or temporarily stored in place prior to sealing (Section 3.4), place a plug of quartz wool into the top of the quartz ampoule. Continue to Section 3.4.

ATTENTION: MAKE SURE THAT THE LOADED AMPOULE REMAINS UPRIGHT AT ALL TIMES TO AVOID POTENTIAL LOSS OF MATERIAL.

b) To prepare a deposit on aluminum foil: the operator may choose whether to aliquot liquid directly onto the foil or into a tared vial or similar container for weighing. Aliquoting into a vial for weighing may make it easier to obtain an accurate solution mass. If the liquid is dispensed into a vial, it will need to be quantitatively transferred to the foil prior to drying. After the transfer, the liquid on the foil is dried using a hot plate, heat lamp, or similar. Use moderate temperatures of ~100-150 °C to avoid spattering. Repeat drying steps may be required between each rinse of the vial, at the discretion of the cognizant scientist.

   When the deposit is dry and the foil has completely cooled, roll the foil into a cylinder to enable loading into the ampoule. If necessary, use a weigh paper funnel during loading to protect the sealing region of the ampoule from any debris that may be on the outside of the foil. Insert the foil into the bottom of the ampoule.

   If the ampoule is to be moved or temporarily stored in place prior to completion of Section 3.4, place a plug of quartz wool into the top of the quartz ampoule. Continue to Section 3.4.

ATTENTION: MAKE SURE THAT THE LOADED AMPOULE REMAINS UPRIGHT AT ALL TIMES TO AVOID POTENTIAL LOSS OF MATERIAL.

3.3 Loading Ampoule with Solid Material

ATTENTION: SKIP THIS SECTION IF PREPARING A LIQUID SAMPLE (SECTION 3.2).

1) Obtain clean quartz ampoules that are appropriate to the application (see Section 2.0 EQUIPMENT and MATERIALS/REAGENTS above for details).

2) Tare an empty beaker on a 4-place balance (which will be used to hold up the quartz ampoule) and weigh a clean quartz ampoule standing upright in the beaker.

3) Place a funnel of aluminum foil or weigh paper in the opening of the quartz ampoule prior to loading material into the ampoule. The funnel should extend below the sealing surfaces to avoid contaminating the sealing region with debris.

4) If using metal foils, scrape off loose oxide from foils using an appropriate tool (e.g., metal spatula, forceps). This is to remove loose oxide that may flake and contaminate the sealing region of the ampoule during loading (e.g., through static). Alternatively, oxide may be removed by briefly submerging the foils in nitric acid, followed by water, then acetone. After the acetone rinse, the foils should be allowed to dry completely before weighing.

5) Load scraped foils, rolled weigh paper, or similar material into the upright ampoule, being careful not to contaminate the sealing region with debris. After adding the sample, remove the funnel and re-weigh the quartz ampoule standing upright in the beaker.

6) If the ampoule is to be moved or temporarily stored in place prior to completion of Section 3.4, place a plug of quartz wool in the top of the quartz ampoule.

<u>ATTENTION</u>: MAKE SURE THAT THE LOADED AMPOULE REMAINS UPRIGHT AT ALL TIMES.

3.4 Evacuation and Sealing of Quartz Ampoule

See Appendix A for helpful tips and techniques for adjusting flame properties and fusing quartz. Appendix B contains figures depicting the manifold. Figure 2 below provides some examples of a correct seal, sealed ampoules with different sample types, and a failed seal made with powder in the sealing region.

1) Remove all flammable items from the immediate work area and locate other items to be used at an appropriate distance from the flame.

2) *(Recommended) Practice flame sealing and self-inspection of at least one empty quartz ampoule prior to sealing ampoules containing any loaded materials.*

3) Place the quartz endcap into the open end of the ampoule. The worker may insert a plug of pure quartz wool just inside the top of the ampoule to help stabilize the end cap and prevent it from sliding out and reduce the potential for loaded materials to be drawn into the vacuum system.

4) Attach the quartz ampoule to the manifold using the built-in o-ring embedded in a hand-tightened flange. Seal the ampoule to the vacuum apparatus by tightening the flange.

<u>NOTE</u>: If available, use a manifold with a sensitive evacuation valve to allow slow application of vacuum to avoid drawing debris into the sealing region or the vacuum pump.

5) Turn on the vacuum until you obtain a slight suction – the pressure on the gauge should read 100 – 200 Torr (absolute), or -11 to -13 pounds per square inch gauge (PSIG).

6) Close the vacuum valve before sealing the quartz ampoule. There will be enough vacuum in the tube to allow the heated ampoule to shrink and seal to the inner quartz end cap.

7) *(Optional) The ampoule may be backfilled with a low pressure of inert gas, e.g., helium, nitrogen. The maximum backfill pressure is 200 Torr absolute, or -11 PSIG. If backfilling, the ampoule is typically cycled 3-5 times between vacuum and inert gas to remove residual air. A pressure relief valve set to release at <2 PSIG is recommended in the sealing manifold design for safety.*

<u>CAUTION! WEAR A WELDER'S MASK FOR QUARTZ/SILICA FUSING STEPS!</u>

<u>CAUTION! HOT GLASS LOOKS LIKE COLD GLASS!</u>

8) Before starting work, check that an adequate supply of oxygen and propane gases are available and that the cylinder valves are open. With the torch valves in the off position, turn on the main oxygen and propane supply valves located in the hood and set the target pressure to 15 psi for the oxygen and 5 psi for the propane. Position the propane torch onto the torch stand. Adjust the gas flow to obtain a good flame and ensure the torch is working properly.

9) To seal the ampoule, heat the ampoule in the region containing the end cap, just above the constricted point of the ampoule, while rotating the torch to evenly heat the tube around the circumference. Continue until the ampoule and the end cap soften and completely fuse together. A visible line forms as the two surfaces fuse. The newly sealed end of the ampoule should remain in the flame for a few seconds to completely seal the opening of the ampoule and end cap. Use caution to avoid over-heating the quartz/silica, which will cause the ampoule to collapse.

10) Allow the ampoule to cool to room temperature before handling as it can burn the operator even when appearing to be cool to the touch – hot glass looks like cold glass. Disconnect the ampoule from the manifold by loosening the knurled nut. Visually inspect the o-ring for integrity and replace if there are any signs of degradation.

11) The ampoule sealer should visually inspect the ampoule being sealed paying attention to bubble formation, the thickness of the sealed ampoule end cap which should be equal to or greater than the thickness of the walls, and stress fractures. Additionally, make certain that there are no pinhole leaks, which are indicated by an indentation in the seal and a fine line through the glass traveling up from the ampoule chamber to above the top of the end cap. Further review of new seals in direct comparison with a verified sealed ampoule by an individual experienced with sealing quartz ampoules is recommended. At user discretion, the visual inspection of ampoules may be validated by first preparing several blank ampoules and testing them by destructive, qualitative (e.g., optical microscopy), or quantitative (e.g., helium leak testing, Section 4.0) leak testing methods.

    It is not recommended that cooled ampoules be re-heated in an attempt to re-work an inadequate seal. Should a seal be deemed inadequate, the ampoule should be broken open and the material transferred into a new, clean ampoule.

12) (Optional) Seal at least one empty quartz ampoule at completion of work. Blank ampoules prepared before and after sealing loaded ampoules may be visually evaluated by the sealer and/or an equivalently experienced peer to assess the consistency of the seal quality.

13) Turn off the propane and oxygen supplies.

14) Take individual pictures of the completed ampoules if desired. It is recommended to document the date of sealing, sample ID, sample mass, initials of the person performing the sealing, and any additional/necessary comments for each ampoule sealed (from the sealer and/or inspector).

15) Samples prepared for irradiation are often doubly encapsulated, e.g. a 6-mm OD ampoule inside of a 9-mm OD ampoule. If a doubly encapsulated sample is prepared, the inner ampoule must be scored to break off excess ampoule length and inspected prior to sealing the second ampoule.

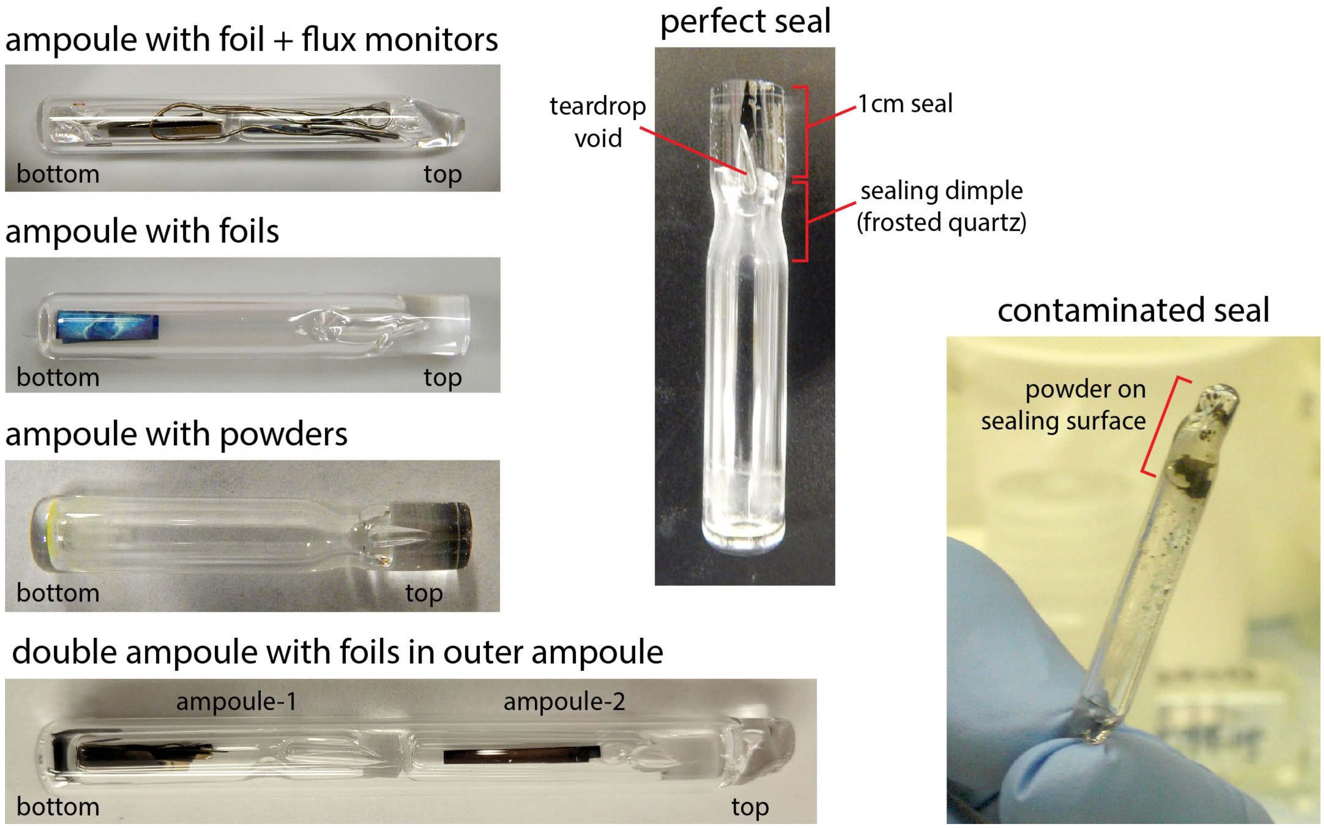

Figure 2. Pictures of different types of ampoules showing inner ampoules with powders, foils, as well as foils with flux monitors. A picture is included of a double inner-ampoule assembly inside a single outer ampoule. Additionally, a perfect seal is shown along with a bad seal where the sealing surface is contaminated with powder.

4.0 Leak Testing (optional)

At the discretion of the user, the blank ampoules prepared to assess consistency of seal quality may be subjected to testing by helium leak rate analysis, or other method determined by user (e.g., optical microscopy), before use. The blank ampoules may also be subjected to additional destructive or non-destructive analysis at user discretion or the discretion of the cognizant scientist.

At user or cognizant scientist discretion, loaded and sealed ampoules may undergo helium leak testing or other non-destructive evaluation as a mechanism to assure seal quality.

5.0 Storage

Once the ampoule is cooled, wipe the outside of the quartz ampoule with 2% nitric acid and rinse with deionized water to remove any residual salts that might impact the use of the ampoule.

The clean ampoule should be stored in a padded, rigid-sided vessel (e.g. a capped centrifuge tube with Kimwipes® inside). The vessel containing the ampoule must be labeled with the ampoule ID and contents, date, and initials of the individual who sealed the ampoule.

Appendix A – Tips and Techniques for Fusing Quartz

One of the issues we have been consistent about with preparation of the ampoule is to keep the glass surface clean. The manufactured surface of the quartz comes to us with a factory cleaned surface (the cleaning process by the manufacturer can be added here if needed). When we handle the ampoule, we use Nitrile or Latex gloves. As we load the ampoule with our powders, we use a long funnel that extends to the bottom of the ampoule so that the powders are dropped directly through the funnel to the bottom of the ampoule and do not contact the walls of the ampoule. The powders then do not interfere with the sealing of the ampoule.

Along these same lines, as we evacuate the air from the ampoule using our vacuum system, we are very careful to start the air evacuation process very slowly so that the powder is not agitated by the air being removed from the powder. At times, we have opened up the valve too quickly to remove the air through the vacuum pump and the sudden drop in pressure causes the powder to fly up with the evacuated air and contaminate the walls of the ampoule, including the area where the flame sealing occurs. We then have to clean the ampoule. We do not want our material to interact with the sealing area of the quartz. The mismatch of our material with the quartz may cause coefficient of thermal expansion issues which could lead to cracking or breaking of the ampoule sometimes during our testing process.

The ampoule needs to be stationed in a way that the flame is not interfering with the material in the ampoule or with the vacuum equipment. The powder is 4 to 5 cm below the sealing area so that it does not get hot enough to react while flame sealing the ampoule. Likewise, the framework of the vacuum system cannot be overheated by the flame. In our system, there is a high-temperature (silicone rubber) o-ring that can take ~200°C temperature. We do not want the heat of the flame to overheat this gasket so we have about 6 to 8 cm between the hot sealing area of the ampoule and the vacuum system as heat transfer through metal fittings can melt the o-ring.

It is important to have the experience to get the torch flame as hot as possible. This helps to heat the quartz quickly and seal the ampoule in a very short time. If the flame is “cool”, it takes a very long time to heat the quartz up and I am uncertain how well the quartz seals if it is at a high viscosity (moves slowly and doesn’t have the fluidity to bond well with another glass surface).

Practice with adjusting the oxygen and propane valves on the torch to obtain the hottest flame possible is necessary for sealing quartz. The optimal starting points on the compressed gas regulators are 15 PSI oxygen and 5 PSI propane. This type of adjustment and gaining experience with the hottest flame can be done with a piece of quartz tubing (0.25-inch OD) held in the hands. When the operator thinks they have the hottest flame, they can put the torch in a mechanical torch holder. With the quartz tube held in both hands, put it into the hottest part of the flame (near the tip of the white-hot flame) and within 5 to 6 seconds, the tube should be soft and molten. If it takes longer than that, the flame is not hot enough. Adjust the touch to a hotter flame. For quartz this can seem awkward since it seems like you are pushing so much gas through the torch that is sounds like a jet engine (not quite the same decibels but the effect is the same). The torch is noisy, the flame is super hot (white/blue in color – now it is really hot!) and it’s ready to seal quartz. You must have ultraviolet eye protection because the white light generated during sealing will burn the eyes. There is a noticeable difference between a hot flame, the normal flow of gas through the torch, and the super hot flame needed for quartz sealing; from the corner of the eye protection you are wearing the amount of white light increases dramatically (2 to 3 times as bright) when you adjust the flame to the super-hot flame! When you finally get the flame that hot, it will melt the quartz tube in 3 to 4 seconds; this is the temperature you are seeking for melting the ampoules.

It is recommended to put the flame on the ampoule at the tip of the flame cone (see Figures A.1 and A.2) by applying heat slowly to heat the quartz over 4 to 5 seconds rather than putting the white-hot flame directly onto the cold ampoule. Instances where the quartz has cracked have been observed when the flame is applied directly to the surface of the cold ampoule.

A.2

Figure A.1 A properly adjusted oxygen-propane flame as seen by the naked eye.

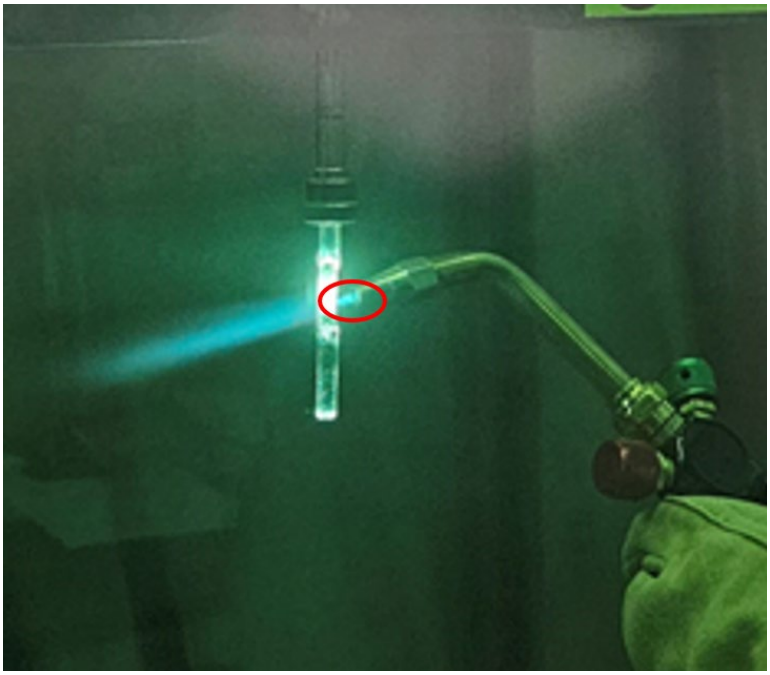

Figure A.2 Flame sealing a quartz ampoule with an oxygen-propane torch as seen through eye protective PPE. The red oval indicates the flame cone. The tip of the flame cone is in contact with the quartz, which becomes white hot and begins to flow and fuse.

With a white-hot flame applied to the ampoule, the glass softens within seconds and the torch can be moved quickly around the ampoule. As the glass softens it will collapse onto the inner glass tubing and a line appears where the two glasses form into a solid, singular, sealed glass piece.

The user should take extra care to make sure they're not overheating the vicinity including the fumehood sash (when heating the far side of the ampoule, the torch is aimed right at the user's face), the vacuum system, or other pieces of equipment sitting within the fumehood. While this is obvious without welding glasses on, when the welding glasses are on, it is difficult to see things that are not white hot due to the high tint factor associated with this level of eye protection. Smaller ampoules made from 4x6 mm tubing may not require the torch to be moved in a full circle around the ampoule.

Appendix B – Quartz manifold diagrams

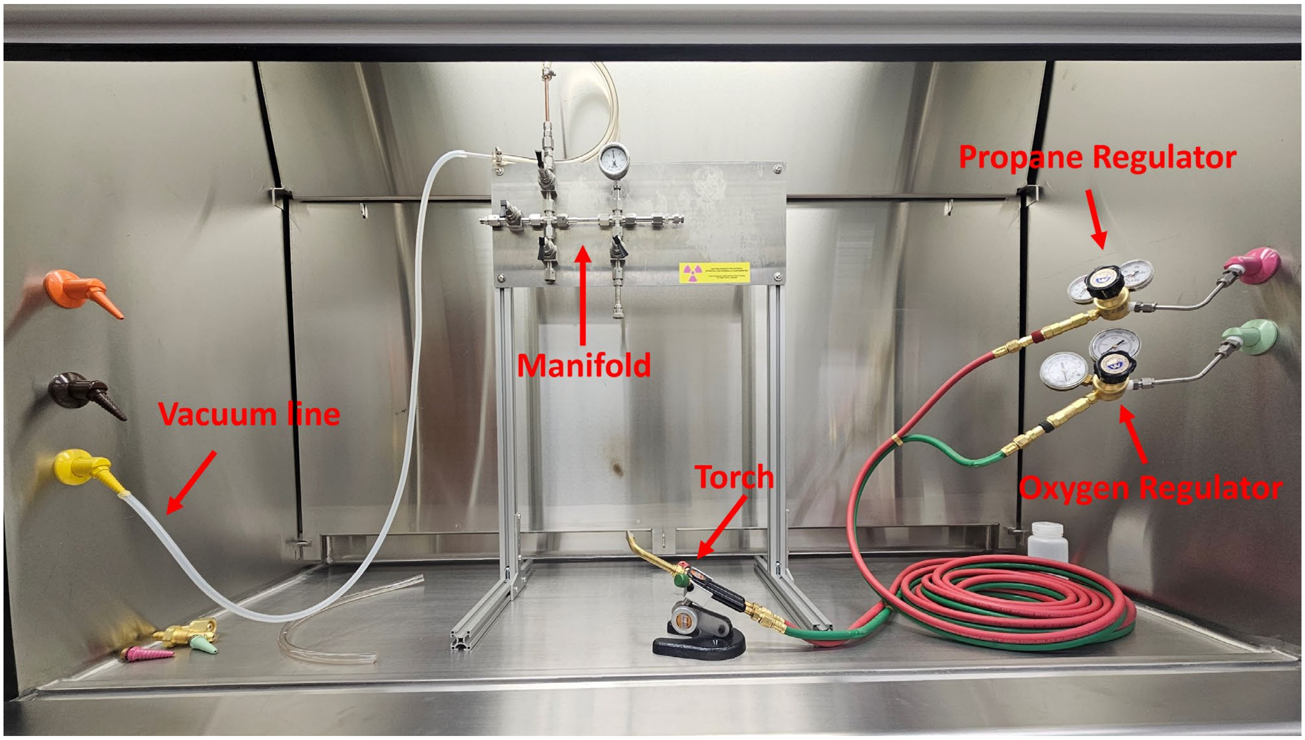

Figure B.1 Fume hood with torch and manifold for flame sealing quartz ampoules. Major system components are labeled.

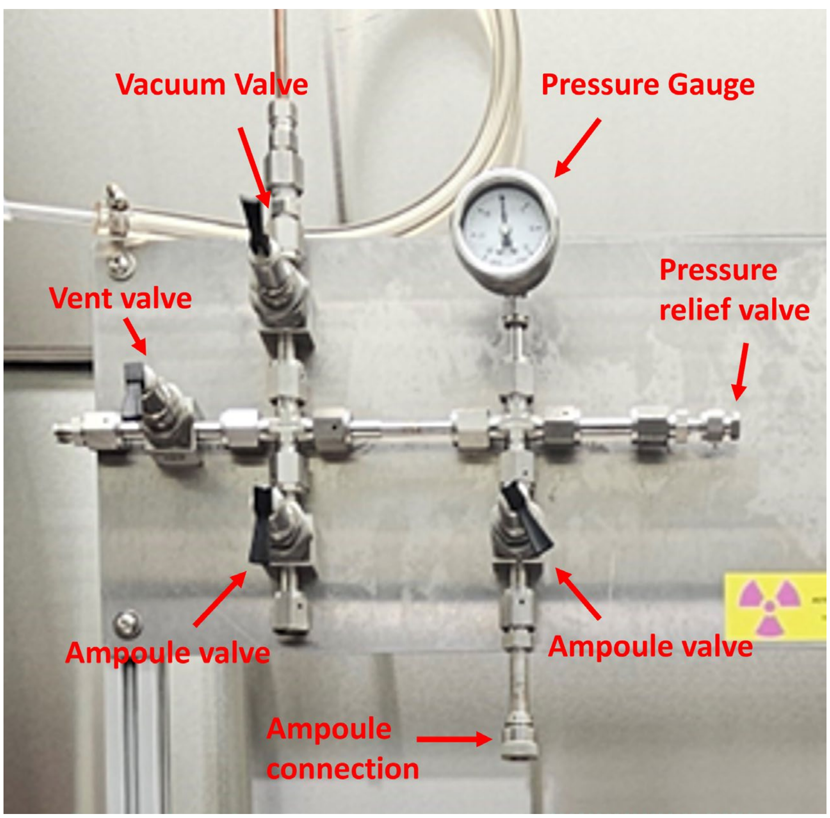

Figure B.2 Closeup view of quartz sealing manifold valves and gauges. Valves are open when the toggle is in the up position (see vacuum valve in photo). System is shown at atmosphere.

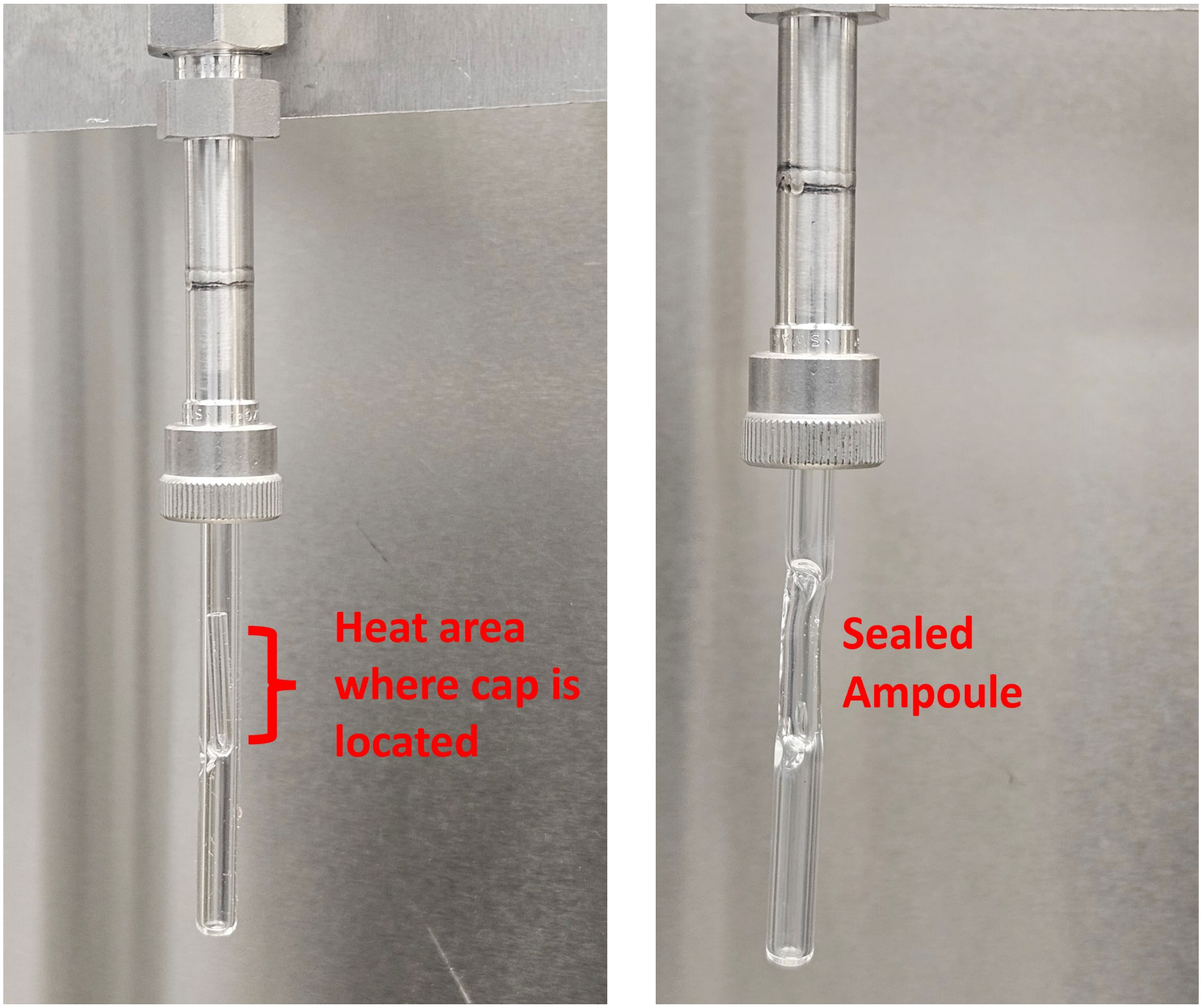

Figure B.3 Ampoules attached to the manifold. (left) Ampoule is unsealed. The endcap region should be heated to effect sealing. (right) A sealed ampoule attached to the manifold.

Appendix C – Ampoule dimensions

The dimensions of the inner and ampoules used at PNNL are shown in Figure C.1 and Figure C.2. The bottoms of the ampoules must be flat with thickness greater than or equal to the thickness of the side walls. Use of an end cap avoids the need to collapse the full diameter of the ampoule, which has the potential to thin the walls of the finished product. Sealing with an end cap, as in the design shown in Figure C.1 and Figure C.2, is faster than sealing without an end cap and should result in >99% sealing efficiency, i.e., sealed ampoules that are accepted for use.

Suprasil grade quartz ampoules are available from https://sandfire.com/quartz-ampoules, who Suprasil. Other companies which do not stock Suprasil require a bulk purchase of Suprasil to prepare ampoules. C-Quartz (Ferndale, WA, 360-393-1254) and other fused quartz distributors can fabricate GE-214 tubing to the specifications given below.

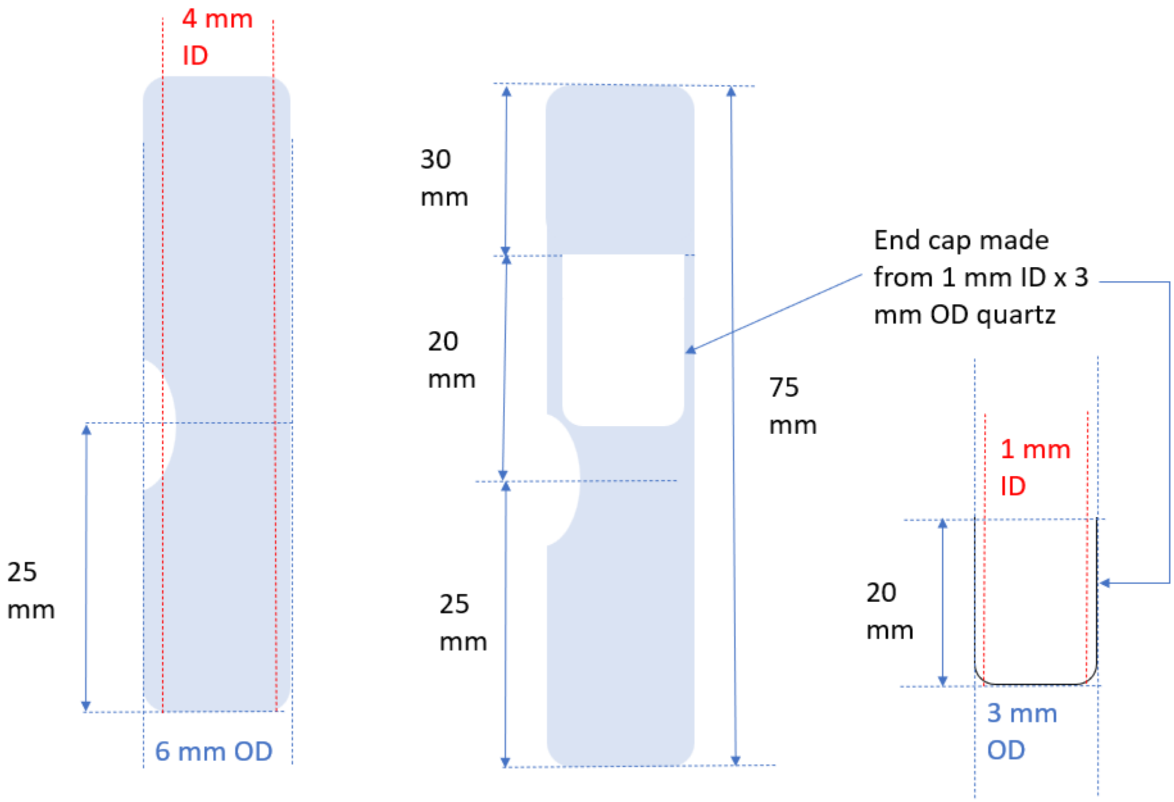

Figure C.1 Dimensions of inner ampoule (left) and ampoule with end cap (middle). The dimple 25 mm from the bottom end of the ampoule provides a resting place for the end cap so that the sample is not directly exposed to the heat of sealing and can be recovered after use of the ampoule. The end cap dimensions are shown far right.

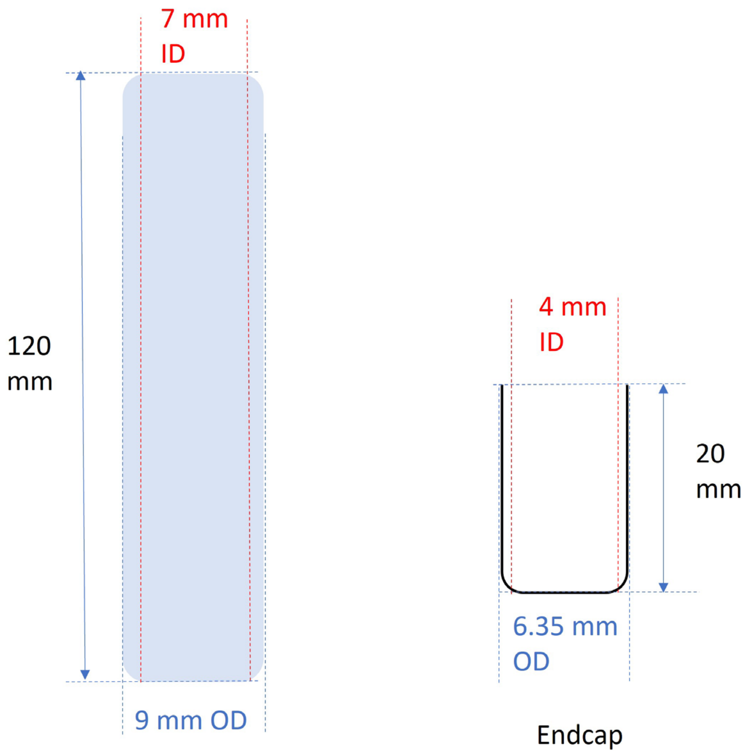

Figure C.2 Dimensions of outer ampoule (left) and endcap (right). The endcap for the outer ampoule rests atop the sealed inner ampoule so the outer ampoule does not require a dimple.

Pacific Northwest National Laboratory

902 Battelle Boulevard
P.O. Box 999
Richland, WA 99354

1-888-375-PNNL (7665)

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