#1 CONTACT INFORMATION:

<table>
<thead>
<tr>
<th>Procedure Title</th>
<th>Sealing Quartz Tubes: Hydrogen Torch Operation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Procedure Author</td>
<td>James O’Sullivan</td>
</tr>
<tr>
<td>Date of Creation/Revision</td>
<td>9/15/15</td>
</tr>
<tr>
<td>Name of Responsible Person</td>
<td>Dr. Chong Zheng</td>
</tr>
<tr>
<td>Location of Procedure</td>
<td>LaT 330</td>
</tr>
<tr>
<td>Approval Signature</td>
<td><em>(If required. See section #9 of this template)</em></td>
</tr>
</tbody>
</table>

#2 THIS STANDARD OPERATING PROCEDURE (SOP) IS FOR A:

- [ ] Specific laboratory procedure or experiment  
  Examples: synthesis of chemiluminescent esters, folate functionalization of polymeric micelles, etc.
- [x] Generic laboratory procedure that covers several chemicals  
  Examples: distillation, chromatography, etc.
- [ ] Generic use of specific chemical or class of chemicals with similar hazards  
  Examples: organic azides, mineral acids, etc.

#3 PROCESS OR EXPERIMENT DESCRIPTION

This SOP outlines the procedure necessary to seal a quartz tube for high temperature, inorganic synthesis in a tube furnace. Other non-trivial skills needed are glove box/tube furnace operation and quartz tube carbonization. **Using a hydrogen torch is especially dangerous, read this SOP very carefully.** It is highly recommended that the user has seen this technique done before attempting.

<table>
<thead>
<tr>
<th>Frequency:</th>
<th>□ one time □ daily □ weekly □ monthly</th>
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<td>☒ other: As needed</td>
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<tr>
<th>Duration per Expt:</th>
<th>10 minutes; or _______ hours</th>
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#4 SAFETY LITERATURE REVIEW & HAZARD SUMMARY

1. **Hydrogen combustion with pure oxygen burns at approximately 3200°C and should be handled with extreme care!** Quartz has a higher melting point than steel, so extreme caution should be used when handling freshly sealed quartz! Volatile chemicals and chemicals at risk for emitting gas at high temperatures should not be sealed in quartz. SDS for all chemicals being used should be reviewed beforehand.

2. Lab goggles and latex/nitrile gloves must be worn while handling chemicals during preparation. A welding mask/welding goggles and a thermally resistant glove/mitt should be worn to handle the quartz tube when hot. The other hand should have a latex/nitrile glove (as opposed to another thermal resistant glove/mitt) to avoid clumsy and dangerous handling of the hydrogen torch. The hydrogen tank also presents an oxygen displacement risk as well as being highly flammable.

#5 STORAGE REQUIREMENTS

Gas tanks must be properly secured to the bench top via the appropriate gas cylinder bracket. Make sure to close the main valves on each tank fully to avoid filling the lab with hydrogen and oxygen gas after use. The control knobs on the torch should be opened fully after use to run the hydrogen and oxygen out of the lines and then should be fully closed again.

#6 STEP-BY-STEP OPERATING PROCEDURE

Steps to include in your procedure:

1. Don personal protective equipment.
   - ☒ appropriate street clothing (long pants, close-toed shoes)
   - ☒ gloves; indicate type: Heat Resistant
   - ☐ safety goggles ☐ safety glasses ☐ face shield
   - ☒ lab coats
   - ☒ other: Welding Mask or Welding Goggles

2. Check the location and accessibility of the safety equipment that serves your lab:

<table>
<thead>
<tr>
<th>ITEM</th>
<th>STATUS</th>
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<tbody>
<tr>
<td>Laboratory Fume Hood/Glove Box or other Ventilation Control</td>
<td>Location: LaT330</td>
</tr>
<tr>
<td>Eyewash/Safety Shower</td>
<td>Location: LaT330/Outside Door</td>
</tr>
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</table>
3. After the reaction mixture has been measured, transfer the mixture into a closed-bottom quartz tube that has 15 to 20 cm extra space above the reaction mixture and has been carbonized. Place the quartz tube into the schlenk adapter and tighten it in. Make sure the valve controlling the hook-up being used is closed then turn on the schlenk pump. Twist all of the unused valves on the schlenk to purge any air from the system and fill the dewar flask containing the moisture trap with liquid nitrogen. VERY SLOWLY open the valve to the quartz tube to avoid any reaction mixture traveling up the tube and into the schlenk system (there should also be a plug of glass wool beneath the valve to help prevent this). When the valve is fully opened, leave the schlenk pump to run for the needed time to attain a pressure of 5 μatm or less in the system (manipulating each of the valves may be necessary to get all the air pockets out of the system, and sometimes it is wise to let the pump run over night if moisture is suspected to be in the sample).

4. When the desired vacuum has been obtained, the torch must be lit: Open the main valve on both oxygen and hydrogen tanks to allow flow of gas to the knobs on the torch. Turn the hydrogen knob on the torch about a quarter turn and the oxygen knob about a half turn. Light a lighter away from the gas flow coming out of the torch and slowly raise it to the flow until it ignites. The torch is sometimes very loud, so be prepared. The desired flame should be relatively loud with a coherent, blue, inner cone that doesn’t exceed a few cm in length. A feel for the exact intensity for the torch is obtained through completing the procedure a few times and is very important. Too strong of a flame will lead to a hole being punched in the tube too early, releasing the desired vacuum. Too weak of a flame will struggle to get the quartz hot enough to be pliable. At this point, a heat resistant glove is put on the user’s weak hand, the torch is held in the strong hand, and a pair of welding glasses or a welding mask must be worn due to strong UV emission during heating of the quartz. The tip of the cone in the flame should barely be touching the quartz, and it should never be left in one spot for more than a second. The goal is to evenly heat a ring of quartz (that is clean and above the carbonization to avoid any defects in the seal and several centimeters away from the schlenk hook up to avoid destroying the rubber gasket that makes contact with the quartz tube with the heat) to the point where it all becomes pliable at the same time. This can be accomplished by using the gloved hand to slowly rotate and gently pull down on the tube while maintaining the red-hot quartz along the whole circumference with the torch. Eventually the ring will become pliable enough that the tube will twist shut at the heated point. When this starts to happen, the focus of the torch needs to be taken off the bottom half of the twist and focused more on the top half of the twisted quartz. This will help avoid the common problem of having a blowout in the top of the tube where the quartz becomes too pliable and the vacuum pulls a hole in the top of the tube. This will most likely happen to the schlenk side of the twist when this is done properly, but that is fine because we only care about maintaining the vacuum in the bottom part. Proper execution should result in an airtight seal on the bottom half with a thin piece of glass connecting both halves. Once the thin strand of quartz is severed with the torch, the sealed tube should be transferred to the concrete tube rack. **The area close to the ring should never be touched, even with a glove! Most gloves still burn/catch fire on contact!** Do not touch the part of the tube in the schlenk for several minutes after sealing is completed, and be very cautious of burns when removing the tube. When the business half of the tube is cool enough, use a pair of pliers to carefully break off the sharp piece of quartz that likely remains on the top.

5. Dispose of hazardous solvents, solutions, mixtures, and reaction residues as hazardous waste. See EH&S Hazardous Waste Program

http://www.ehs.niu.edu/ehs/chemical/waste.shtml

6. Clean up work area and lab equipment.

There really isn’t any mess made, but make sure the torch and the tubing is wrapped around the gas tank to keep it out of the way.

7. Remove PPE and wash hands.
#7 WASTE DISPOSAL

There is no chemical waste generated from this procedure, but make sure any excess glass is put in the SHARPS container.

#8 TRAINING REQUIREMENTS

General Training *(check all that apply):*

- General Safety & Emergency Preparedness
- Chemical Safety for Laboratories
- Radiation Safety
- Biosafety training
- Other: __________________________

Location Where Records Maintained: ___________

Laboratory-specific training *(check all that apply):*

- Review of SDS for other chemicals involved in process/experiment
- Review of this SOP
- Other: __________________________

Location Where Records Maintained: ___________

#9 PRIOR APPROVALS

Prior approvals are required by the following University Committees: