Standard Operating Procedure: TUBE FURNACE OPERATIONS

Laboratory Information

Department:	
Principal Investigator(s):	
Laboratory Manager/Supervisor (or designated laboratory-specific chemical hygiene officer):	
Laboratory **emergency contact** (name and phone):	
Laboratory phone:	
Designated area (s) of these procedures (building and room):	

This SOP is intended as an initial resource and reference regarding the topic. It is not a substitute for hands-on training and supervision by experiences laboratory personnel. This SOP is not complete until: 1) Clear and detailed instructions are written so as to ensure safe handling of any hazardous materials and safe performance of procedures, and 2) the SOP has been reviewed, approved, and dated by the Principal Investigator and/or Laboratory Supervisor. A hard copy of this SOP must be placed in the laboratory specific Chemical Hygiene Plan/Laboratory Safety Manual and made available to laboratory personnel.

Purpose

This SOP is designed to function as a starting point to illustrate the hazards associated with- and the best practices for operating laboratory-grade tube furnaces. It is not designed to furnish details for operation of a specific model of tube furnace or for the execution of a specific process/recipe. This SOP does not cover the use of high-pressure tube furnaces.

Groups must conduct a PPE assessment and ensure that furnace operators use appropriate PPE to always protect themselves.

Personnel conducting these procedures shall be informed and trained on the following:

- The chemistry and reaction processes associated with the heating procedure to be performed and should conduct a risk analysis to ensure that risks are minimized, especially in case of a fault.
- The compatibility of materials used to contain specimen(s) and process gases at the proposed process temperatures.
- Any necessary treatment of any exhaust/reaction products from the heating procedure.
- The limitations of the tube furnace and various components such as furnace tubes, sealed reaction vessels, and others.
- How to safely handle/operate compressed gas supplies.

Definition of Terms

Furnace: Any electric-powered heater used to heat materials to high temperature, generally in a controlled manner. In the context of laboratory use this commonly includes box furnaces, muffle furnaces, and tube furnaces. See figures 1-4 below.

Tube Furnace: A furnace having a cylinder as its specimen- and/or reaction-chamber. The cylinder, or tube, is often readily removable and can be sealed at both ends, allowing controlled-atmosphere reactions to be done. The furnace control system allows temperature 'profiles' or 'recipes' to be executed (temperatures and their durations, as well as their rates-of-change are fully controlled). Numerous sizes and varieties exist, including multi-zone tube furnaces that have different temperature regions along the length of the furnace tube.
Common uses for materials synthesis include annealing, oxidation, diffusion, doping, crystal growth, nanotube growth, etc. See figures 2-4 below.

Tube or Furnace Tube: In this context the furnace tube is the reaction chamber where specimens and/or specimen reaction ampules are placed and heated. It is literally a long cylinder, commonly made of Quartz or Alumina (Al_2O_3). It allows the exclusion of air from the reaction (at minimum), and allows a region of controlled atmosphere (humidity, noble- or reducing gas, etc.) to be maintained throughout a desired process, as well as a region of controlled temperature. It also physically separates the reaction volume from other furnace components such as the heater elements, enclosure, and control electronics.

End Cap: A device used to seal the ends of the furnace tube, allowing specimens to be heated in a controlled atmosphere and exhaust gas flow to be controlled/managed. End caps are commonly water-cooled because they are typically made of metal (brass, aluminum) and employ O-rings to accomplish a positive seal to the furnace tube. See figure 7.

Boat: A pan or flat surface used to keep a specimen inside the furnace tube from contacting the tube, and/or to keep it horizontal so that it doesn't spill. Often this is used to minimize contamination of - or deposition of material onto - the surface of a furnace tube. Boat materials are chosen to minimize undesired reactions or contamination. See figure 8.

Push rod: A device used to move specimens to the desired position inside the furnace tube (often the center). A rod of sufficient length is necessary. Consideration should be given to materials to avoid contamination of the furnace tube when the push rod is inevitably dragged across its inside surface during specimen insertion/removal operations. For example, the use of a steel rod with an alumina furnace tube virtually ensures contamination of the tube with transition metal- and carbon contaminants. This is often undesirable. In this example the use of an alumina push rod is preferred.

High-Pressure Tube Furnace
A tube furnace that has a reaction chamber designed to withstand heat and high pressure. These tubes can be filled with compressed gas to hundreds of psi (rather than being designed to flow gas through them at essentially ambient pressure or a few psi above). The furnace tubes used in these applications are commonly made of a nickel-based superalloy. The alloy does not experience brittle fracture and explosion when it fails or is over-pressurized. Instead, its ductility and tensile properties cause it to swell and eventually crack, resulting in pressure relief (a leak) without explosion. This SOP does not cover the use of these types of tube furnaces.

Hazards Related to Tube Furnaces

Operations involving tube furnaces include work performed in numerous hazard categories. The risks are varied and will depend on the process performed. Some are obvious and some are less so. Research groups should perform a comprehensive hazard assessment based on all elements of their proposed operation to determine the risk of exposure to the hazards proposed below, and to discover and understand potential others not mentioned here. The list below will offer some assistance. Possible hazards include:

Burns: Furnace tubes, furnace cabinets, handling tools, specimens, and carriers may be extremely hot. Radiation/glare from very hot objects can pose a hazard to the skin and eyes.

Sharp edges: Furnace tubes, be they quartz or alumina, exhaust tubing, gas supply lines, etc. may have very sharp edges.

Gas cylinder operations: Pressurized cylinders represent substantial hazards during handling and use.

Exposure to gases: Gases may be inert, asphyxiant, oxidizing, toxic, etc. It is possible to be exposed to process/purge gas if a leak occurs. Compatibility of plumbing (including the furnace tube) shall be considered. Gas leaks can occur in gas line connections, furnace tube end caps, etc.

Pressurization: When heated, the pressure inside sealed specimen ampules may rise dramatically, potentially creating a severe risk of breach. Furnace tubes should not be pressurized.

Vacuum: Some furnace tube operations include evacuation to remove air and contaminants prior to desired purge and process steps. This means that an implosion risk exists, especially if there are flaws in the furnace tube or if it is struck by some object during the procedure.

Fire: Working gases may be flammable or may support combustion. Specimens may thermally decompose and catch fire or create smoke – especially if a leak occurs.

Decomposition products/reaction products: Reaction products could enter the room if a leak occurs. This can be caused by a failure of gas line connections, furnace tube end cap leaks, etc.

Inhalation of dusts: Tube furnaces are commonly insulated using refractory-metal-ceramic-fiber-based insulation materials. If these materials are disturbed, they readily create fine dust, which are possible respiratory hazards. Lab personnel should avoid generating and inhaling these particles.

Electrical hazards: Furnaces are electrically operated devices. Heater elements are exposed inside the enclosure. Good furnace design should make it difficult to come in contact with electrical conductors during normal operation; however, it may be possible to come into contact with these items when working with a furnace during maintenance.

Quenching: (In this context, quenching is used to describe a heat-treating method where an object is rapidly cooled to obtain certain material properties.) The use of a tube furnace of the size and type typically encountered in a research laboratory to perform quenching is extremely risky. Tube furnaces with small diameter tubes (6-inches to 2-inches or less) are typically used in these environments, making specimen handling “at temperature” a very awkward endeavor - so much so that the tube furnace should not be considered an appropriate tool for this operation. It is generally not possible to remove furnace-tube end caps during a process without a high risk of burning oneself and/or breaking the furnace tube.

Specimen Ampules: Sealing specimens in ampules is a specialized operation involving the use of (typically) quartz tubes, torches to heat and “pinch off” the quartz tube ends, and gas-filling “jigs” to fill them with a desired reaction

gas (see Fig. 6). Numerous hazards are associated with handling ampules, heating them with torches, evacuating them, using compressed gases to fill them, and heating the resulting sealed volumes to high temperatures inside a tube furnace. This SOP does not cover sealing of specimens in ampules.

Examples of hazardous materials or processes involved with Tube Furnace operations:

- Oxidizers in combination with elevated temperatures and organic materials
- Process-gas flow rates should be kept low enough to avoid displacing small particles / nanomaterials and creating an inhalation hazard.
- Be aware of auto-ignition temperatures for certain gases/materials such as Hydrogen, Methane, etc.
- Toxic gases shall be treated with utmost care, stored in a gas cabinet, etc.

Controlling the Hazards Related to Tube Furnaces:

In response to the lab's specific hazard assessment, groups can plan how to minimize the risks of working with tube furnaces - especially in combination with their process(es). This includes the use of PPE, the development of specific steps in the SOP, the use of engineering controls such as the layout of the laboratory space around the furnace, exhaust connections, etc. The goal is to reduce the likelihood of exposure and/or injury when something goes wrong. The minimum PPE for use of a tube furnace includes eye protection, protection from burns, and protection when handling furnace tubes (see required PPE). Addressing the list in the previous section yields the following suggestions:

Burns: Handle items only when cool, if possible. Otherwise use appropriate PPE (heat-resistant gloves). Always wear eye protection when working near furnace tubes, handling them, etc.!

Sharp edges: Use leather gloves to protect against cuts when handling cool furnace tubes and exhaust tubing.

Gas cylinder operations: Operators shall be familiar with handling gas cylinders, installing and operating pressure regulators, flow devices, etc. They shall understand the risks associated with use of a particular gas, including the potential exhaust products resulting from the desired process reaction. Training for gas cylinder operations and associated equipment (such as pressure regulators) is recommended.

Exposure to gases: Operators shall exhibit some level of competence in making secure (leak free) connections to gas tubing, furnace tubes, bubblers, exhaust lines, etc. Connections should be robust and well secured using quality, compatible materials and products to ensure their function even in the case of accidental contact or faults/mistakes during operations. In processes where more than one gas or gas-mixing is desired, appropriate check valves should be used to minimize consequences of operator error and to ensure intended gas flow direction.

Pressurization: Calculations should be made to determine pressures that will be encountered when heating sealed specimen ampules, and the experiment designed to avoid breach or rupture of these vessels. A detailed set of instructions for filling and sealing ampules (an SOP) should be developed to assure safe, predictable behavior and results during these operations.

Vacuum: The use of furnace tubes that can support atmospheric pressure without collapse is essential if evacuation is a desirable step in the process. Laboratory layout should be designed to minimize the risk of accidental contact with the evacuation system and furnace tube (ample space, etc.). Protective eyewear should ALWAYS be worn around an evacuated furnace tube.

Fire: Connections should be leak free. The experimental design should take into consideration what reactions will occur during the process. This should include the possibility of air ingress (a leak). A plan of action should be

Controlling the Hazards (cont.)

developed to prepare operators for the possibility of a fire.

Decomposition products/reaction products: Operators shall exhibit some level of competence in making secure (leak free) connections to gas tubing, furnace tubes, bubblers, exhaust lines, etc. Connections should be robust and well secured using quality, compatible materials and products to ensure their function even in the case of accidental contact or faults/mistakes during operations.

Inhalation of dusts: Furnace tube installation and removal and furnace maintenance (such as heater replacement) should be conducted with due care to avoid contacting furnace insulation.

Electrical hazards: Electrical wiring should be in good condition and should be inspected regularly to ensure its safety. Cracked or frayed power wiring shall be replaced. Furnace control electronics and consoles should be operated with covers in place. Contact with heater elements should be avoided.
Quenching: The use of a laboratory tube furnace to perform quenching should be considered as use of the wrong tool for the task. See: 'Quenching' in Discussion of Hazards, above.

Specimen Ampules: Sealing specimens in ampules (usually with a specific gas inside) is a specialized operation that warrants development of procedures to perform this safely and consistently. Among other particulars, gas pressures shall be carefully controlled to ensure that the ampule survives the desired heating process in the tube furnace.

Groups should identify hazards involved in their procedures and describe how these hazards will be controlled in this section:

Storage and Handling

Furnaces are often turned off when left in an idle state. Turn off the cooling water if not needed to prevent large spills in case of an unattended leak and to reduce water consumption. Some applications require the furnace to be left at an elevated temperature. Specific procedures should be determined by the individual research groups.

Furnace Tubes
It's generally preferred to store these items where they won't be damaged or contaminated. Furnace tubes that are contaminated from process operations should be stored according to hazard class and compatibility considerations.

Gases

- Gas cylinders shall be stored/operated in an upright fashion, secured at midpoint or above with the appropriate bracket and tether mounted to a secure structure.
- Cylinders shall be stored according to manufacturer's SDS.
- Cylinders shall be stored a safe distance from the point of use (heat sources, furnaces, etc.).
- Cylinders shall be stored away from chemically incompatible materials.
- Cylinders shall be stored away from electric circuits or electrically energized systems.
- Quantities should be kept to a minimum.

Additional lab-specific instructions regarding storage or quantity limits shall be described here:

Work Practices and Engineering Controls

Certain practices, when performed safely and consistently, ensure operational success and personal safety as well as quality results. Practices may include:

- Don't store items on top of the furnace.
- Inspect the furnace regularly (consult manufacturer's guidelines)
- Keep flammable items away from the furnace (paper, solvents, etc.)
- Installation of gas tubing, exhaust piping, and connections should be robust and well secured using compatible, quality materials and parts.
- Before the heating process, verify operation of essential functions that are used to avoid damage (water cooling, for example).
- Verify that safety controls are in place and operating (interlocks, bubblers and liquid level, etc.).
- Employ smart system design and devices to allow some fault tolerance in case of user error or process fault.
- Operators may elect to use a purge gas step to ensure that it's safe to open furnace tubes after the process has finished.
- Furnace tubes that are sufficiently contaminated should be replaced.
- Once a process has been started, it should be monitored until it reaches a steady state (for example to make sure that the reaction isn't likely to "run away"). For long processes, operators should return regularly to check on the operation.
- Ensure that emergency equipment is in place and available before beginning work.
- Use appropriate PPE!

Designated area for work

The work area(s) should contain necessary equipment (or easy access to it), and sufficient controls to operate safely. The furnace should be installed in a compatible space, with sufficient room around it to allow operation and to minimize the risk of accidental contact with plumbing, piping, furnace tubes, etc. Crowding the area around the

furnace is unsafe. The tube furnace should be installed on a sturdy, stable work surface. Ensure that other experiments in the laboratory are not in the space occupied by the furnace and its operation. Keep flammable items and ignition sources away.

Necessary Administrative and Engineering Controls

- The laboratory where the furnace is operated shall maintain a Log sheet to inform others when the furnace is in use and what the process traits are.
- Furnaces should be unambiguously labeled/named when more than one occupies a single lab space.
- The laboratory should have a dedicated exhaust system to remove exhaust gases from the furnace tube.
- When toxic gases are used, appropriate gas handling equipment is essential, including gas cabinets, gas detection sensors, etc.
- When toxic gases are used, the furnace should be located and operated inside a working chemical fume hood in case of a breach or leak.
- When multi-part-gas, multi-step-gas or gas-mixing operations are desired, appropriate check valves installed in gas lines help ensure proper gas flow and help to reduce the likelihood that a user error escalates into a fault or emergency.
- It is often desirable to use a 'bubbler' in the exhaust-end of the gas stream to monitor gas flow and to 'scrub' the exhaust before it leaves via the exhaust line. It is essential that the operator understands the chemistry associated with the process to ensure that this is used when it's necessary to scrub the exhaust gas, and to ensure that a compatible bubbler medium (fluid) is used.
- The use of a mass flow controller can ensure more reliable and repeatable process-gas flow settings and reduce over-use of gas supply.
- A rotameter can be used as a visual indicator to monitor gas flow.
- Gas cylinders should not be placed too close to the furnace. Ideally it is desirable to have access to them if the gas supply must be terminated in an emergency.
- A water-cooling interlock is preferable. It prevents the system from heating without necessary water cooling (end caps). It adds a simple layer of fault tolerance to the process, guarding against a missed operation on the part of the user.
- A device that adds a visual and/or audible indication of water flow can also be helpful.

Additional lab-specific instructions regarding required administrative and engineering controls shall be described here:

Required Personal Protective Equipment

Fully enclosed shoes shall be worn in the laboratory (no sandals, etc.). Long hair and loose clothing shall be restrained, and full-length pants or skirts shall also be worn along with basic PPE to include but not limited to:

- Eye protection – Safety glasses meeting American National Standards Institute (ANSI) standard Z87.1 are required. Eye protection can also protect users from the heat and glare of extremely hot objects. Tinted safety eyewear and full-face shields can offer protection from these hazards.
- Gloves – Gloves are required at all times. Glove choice should balance the need for dexterity and heat resistance. Different gloves may be necessary during various parts of lab procedures (heat-resistant, cut-resistant, etc.).
- Lab coat – preferably a flame-retardant, coat made from non-synthetic material.

Please describe all required PPE for conducting tube furnace procedures:

Description of Procedures

The procedures for individual furnace operation/individual research groups will vary widely due to the variety of equipment combinations available to the researcher (various furnace types and sizes, various temperature controller types and configurations, etc.). This is beyond the scope of this document, so editing this document to suit the specific laboratory is essential. A common sequence of steps will often resemble the following (based on a single temp-zone tube furnace). The material below is simplified because it omits particulars such as details of programming a specific temperature control system and others. This information should be added to this SOP or incorporated into it by reference to instructions for the device.

Step-by-step Procedures

The step-by-step procedure below is divided into six blocks of tasks: Prepare, Inspect, Set up, Run, Repeat, and Finish.

Prepare

- Read SDSs for materials and gases to be used in the experiment/process. Know handling and response protocols for normal use and for emergencies.
- Understand handling and procedures for wastes generated during the experiment.
- Learn how to operate gas-handling controls (cylinder pressure regulators, mass-flow controller systems, etc.). It is essential that users are familiar with the operation of these controls before attempting work.
- Install appropriate gas supply plumbing for the experiment.
- Ensure that sufficient gas supply is available for the duration of the planned process.
- Read and understand how to operate the furnace and know its limitations.
- Understand how to program the temperature controller used on the furnace.
- Know the limitations of the furnace tube that is intended for use (such as upper temperature limit & temperature rate-of-change limits). Furnace tubes have limits to how fast they can be heated!
- Ensure that the various tools/objects needed to handle specimens are available and are of compatible

materials.
- Wear appropriate PPE during the various steps of this procedure.

Inspect

- Inspect the furnace for obvious damage (enclosure, heater elements, insulation, etc.).
- Inspect the furnace tube for damage, unacceptable contamination, cracks, etc. Reject and replace furnace tubes of suspicious or compromised quality.
- Inspect gas lines for proper installation, obvious damage, leaks etc.
- Inspect water-cooling lines for damage, leaks, proper operation, including function of interlocks.
- Inspect exhaust-handling system for damage, leaks, proper function - verify flow!

Set up

- Log the operation so others will know that the furnace is in use.
- Install furnace tube.
- Install one end cap (the order of steps here depends upon the 'perishability' of the specimen - is it air sensitive? Some researchers would do other preparatory steps first, then finish with the specimen insertion and end cap installation. Modify the planned approach accordingly.)
- Insert the specimen(s)
- Install the second end cap.
- Install exhaust line connections, including a bubbler if necessary or desired.
- Install process gas connections if not already done.
- Program the temperature controller (follow its instructions and stay within the limits of the furnace and the furnace tube).
- Purge the furnace tube if necessary.

Run

- Ensure that the exhaust line is open to the laboratory exhaust system.
- Begin process gas flow if not already done.
- Ensure that necessary water flow is turned on.
- Energize/turn ON the heater circuits.
- Begin (RUN) the temperature program using the temperature controller.
- Monitor the process to ensure that it's operating as expected.

Repeat

Temperature controllers commonly retain the programmed temperature profile, allowing repetition.

- Allow enough time for gases to be purged and the system to cool sufficiently, then:
- Turn off the process gas flow.
- Exchange specimens by

Please add additional lab-specific procedural information here:

Please add additional lab-specific procedural information here (continued):

Waste Disposal

These procedures will generate specimens to be used/analyzed elsewhere, however it is possible to be left with specimens and contaminated furnace tubes that must be disposed. These should be handled in the following manner.

- Handle/prepare all waste materials according to their hazard class.
- Place materials into an appropriately constructed and compatible container.

Waste chemicals shall be collected in a secured area. The area shall be free from evidence of spills. A Hazardous Waste label shall be affixed to the collection container and components shall be listed on the label as they are introduced into the container. Do not date containers until the day of scheduled pickup. Additionally, the container shall be marked with the hazard class of the chemical waste (i.e., Ignitable, Toxic, Reactive, Corrosive). When the container is no more than 2/3 full, date the container and submit a pick-up request in the UK E-trax system: https://etrax.chematix.com/Chematix/

Please add any additional disposal instructions here:

Emergency response

A laboratory and/or process-specific plan of action should be developed to increase the likelihood of predictable assessment of- and behavior during- an emergency. This plan should become part of the training procedure.

A well-prepared emergency response will include a plan for emergency shutdown of the furnace and termination of a process. Operators should be trained on the plan. All training should be documented.

Scenarios
When imagining what could happen during a high temperature heating process:

- fire (situation where it is contained within the furnace tube and one where it is not),
- smoke evolution (contained and not contained),
- breach of gas lines, furnace tubes, or specimen ampules (including explosion),
- release of gas or reaction products,
- breakage of furnace tubes, with or without further complication.

Research groups should consider other emergencies unique to their processes and location and document them in this section. The SOP isn't pertinent unless it contains information that is useful to the group.

Emergency equipment must be present before work is begun. Operators should be familiar with locations of all emergency equipment.

- Fire alarm - identify where pull stations are.
- Fire extinguisher of the correct type.
- A chemical fume hood - if available - can be used to remove gas/fumes from the lab space in an emergency by opening the sashes fully and closing the laboratory door. The fume hood must be working properly.

Fire

In almost all imaginable cases, a fire warrants immediate evacuation of the laboratory and activation of the building fire alarm!

- If possible, turn off the power to the furnace when leaving the laboratory. Do not put oneself in danger to accomplish this.
- If possible, shut off the gas supply if it supports combustion. Do not put oneself in danger to accomplish this.
- Activate the building fire alarm and alert nearby personnel to evacuate.
- Call 911 from a safe location to alert emergency responders. Meet responders to inform them of the situation.

If the fire is contained within the furnace tube and it is safe to do so…

- Terminate the heating process using the emergency shutdown plan.
- Shut off the gas supply if it supports combustion.

The next action depends upon whether the fire is extinguished or not:

- If not, consider this an escalation of the situation and follow the instructions for a Fire - above.
- If it does, allow the system to cool down, monitoring for an escalation of the situation.

Note that if the gas supply serves to extinguish the fire (such as Argon or another noble gas), it may be useful to leave it flowing.

Smoke evolution

Presuming that the smoke is contained within the furnace tube and exhausts away without entering the laboratory air, this scenario will require some assessment "on the spot" to deal with.

- Terminate the heating process using the emergency shutdown plan.
- Shut off the gas supply if it supports creation of smoke.
- If safe to do so, monitor for a fire and react accordingly if it occurs.

If the smoke is not contained, follow the steps below in Release of gas or reaction products. Breach of gas lines, furnace tubes, or specimen ampules (including explosion) In this scenario it is unlikely that the operator is able to do anything in the laboratory. Hence

- Evacuate the laboratory, closing the door on the way out if possible.
- Activate the building fire alarm and alert nearby personnel to evacuate.
- Call 911 from a safe location to alert emergency responders. Meet responders to inform them of the situation.

In the case of a specimen ampule rupture that is contained within the furnace tube it may be possible to handle the situation. The key here is that one must be certain that the furnace tube is intact and that therefore no reaction products have been released into the laboratory. Needless to say, this is a difficult determination to make "on the spot."

In case of a simple gas line failure, it may be manageable. Consider how to safely proceed if a process was underway. If it is safe, close off the gas supply and repair the problem.

Release of gas or reaction products

Response to this scenario is greatly assisted if one understands the reaction products of the planned process in advance. This scenario presumes a release into the laboratory. Unless one is completely assured that the release is inert or noble, or that it is safely manageable, users should:

- Evacuate the laboratory, closing the door on the way out if possible. If a fume hood is available, open the sashes to help remove gas from the laboratory. *Do not put oneself in danger to accomplish this.*
- Activate the building fire alarm and alert nearby personnel to evacuate.
- Call 911 from a safe location to alert emergency responders. Meet responders to inform them of the situation.

All situations involving leaks of toxic or life-threatening materials should be considered exceptionally hazardous and should not be handled by lab personnel. In these cases, personnel should:

- Evacuate the laboratory, closing the door on the way out if possible.
- Activate the building fire alarm and alert nearby personnel to evacuate.
- Call 911 from a safe location to alert emergency responders. Meet responders to inform them of the situation.

Breakage of furnace tubes

If the breakage is due to an explosion*, this must be treated as a release of gas or reaction products (see above)*, unless the furnace tube broke during an evacuation step AND no gas is involved.

Handling of broken alumina or quartz pieces presents obvious risks. One must know what contaminants may be on the furnace tube before attempting the following.

- Wear protective eyewear and a lab coat.
- Use leather gloves to protect from cuts and potential poisoning.

- Use a dustpan and broom to carefully collect the broken pieces. *Be mindful that this activity could create a dust hazard (from powder samples) and modify tactics accordingly.*
- Place collected debris into an appropriate container for disposal (plastic pail that can be sealed, cardboard box, etc.). The determination of the container depends upon the state of the furnace tube (what it's contaminated with, if anything).
- Dispose of the debris according to its hazard class.

If the breakage occurs while the tube is installed in the furnace:

- Execute the emergency process-interruption steps if a process was running, including shutting off the gas supply.
- Ensure that the furnace and all parts have cooled.
- Ensure that the furnace is powered OFF.
- Determine what parts can remain inside the furnace without consequence (clean furnace-tube pieces that don't affect the heater elements, furnace insulation, or that don't evolve any contaminants when heated can be harmless).
- Clean up the pieces as described above, manually picking up pieces in or on the furnace - *use PPE!*

Additional lab-specific instructions regarding emergency response shall be described here:

Spill Procedure

Before work begins, lab personnel shall be knowledgeable on clean up or how to react to a spill or leak of any material that will be used. This is part of the hazard assessment process. Spill response procedures are also based on individual comfort level. Only the person dealing with the emergency or the Principal Investigator should decide whether it is too large, complicated, or dangerous to handle safely. Do not hesitate to call for help in an emergency.

Major spills of stock solution:
Leave the area and notify others not to enter. Report the spill to the UK Environmental Quality Management Department (EQMD)at (859) 323- 6280 (M-F 8am-5pm) or after hours by dialing 911 from any on-campus phone or by contacting the UK Police at (859) 257-UKPD (8573).

Minor spills of manageable amount: If necessary, contact EQMD for guidance. Consult manufacturer's SDS for instructions and compatibilities for the chemicals involved (Be aware of any materials such as paper towels or water that could be incompatible with the spilled chemical!

Additional Lab Specific Instructions or Notes on Spill Response:

Response procedures in the case of an incident or injury:

Before work begins: Personnel shall be trained on response procedures such as contact information, emergency resources available, and location of a phone, fire alarm, evacuation route, etc.

Exposure to Process Gas/Reaction Products/Exhaust
Move to an area of fresh air. Know the symptoms of exposure, asphyxiation, etc. and respond accordingly. Keep in mind that the victim's judgement may be impaired. Do not hesitate to get assistance from a lab mate. If incident occurs during regular work hours, inform the PI or supervisor. Seek medical assistance (see below). In case of life-threatening situations (impaired or loss-of consciousness, difficulty breathing, etc.) call 911 immediately! Stay with the victim. If possible, ask another person to meet responders so that they can locate the injured party quickly.

First Aid for Burns
Get help from a lab mate. If no one is available to help, call 911 immediately. Seek medical care without delay. Keep the victim calm and seek medical assistance for the victim (see below). If possible, ask another person to meet responders so that they can find the injured party quickly.

First Aid for Cuts (primarily due to handling furnace tubes, etc.)
Clean and bandage the wound if possible. If it is during regular work hours, inform the PI or supervisor. Seek medical assistance (see below).

UK Employees:

- After receiving first aid (refer to the chemical's SDS), report the occupational exposure to a hazardous chemical to UK Worker's Care at 1-800-440-6285.
- If needed, an appointment will be made for the employee at UK UHS.
- For severe emergency or injury, call 911 or proceed to the UK Chandler Hospital Emergency Department

UK Students:

- Call UHS at (859) 323-APPT to report an occupational exposure to a chemical hazard.
- If needed, an appointment will be made for the employee at UK UHS.

Visitors:

- Proceed to Chandler Hospital Emergency Department or Primary Care Provider, as needed.

For severe emergency or injury, call 911 or proceed to the UK Chandler Hospital Emergency Department

Additional Lab-Specific Emergency Procedures, including any material-specific First Aid treatments (e.g., the use of Calcium Gluconate for skin exposure to Hydrofluoric Acid):

Personnel Training

- Prior to conducting any work with (name of the chemical), designated personnel shall be provided training specific to the hazard involved in working with the substance.
- The PI shall provide his/her lab personnel with a copy of the SOP and provided access to a copy of any related chemical SDS provided by the manufacturer. Any further training materials shall be documented and stored in the lab's Laboratory Safety Manual and available to internal UK or external oversight agency inspectors.
- The PI shall ensure that his/her lab personnel have completed the initial and the consecutive annually required Chemical Hygiene trainings and Hazardous Waste training.

The undersigned have read and understood the content of this SOP for:

FURNACE TUBE OPERATIONS

Lab personnel performing procedures with this agent (typed name)	Date of hands-on training for this agent	Name of person who provided the agent specific training (typed name):	Signature of Lab Personnel

SOP Reviewed and Approved by:

PI: (typed name)	
PI: (signature and date)	
Lab Manager/Supervisor (typed name)	
Lab Manager/Supervisor (signature and date)	

Figure 1: Box or Muffle Furnace

Figure 2: 3-zone tube furnace

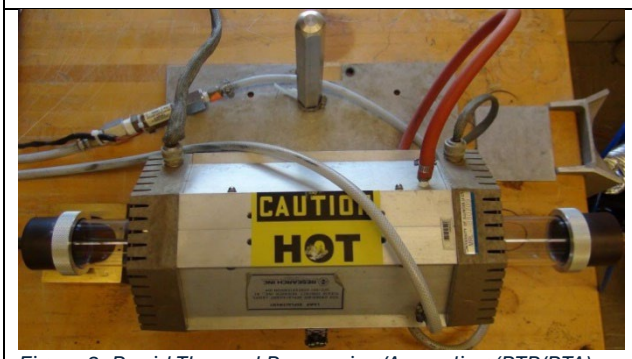

Figure 3: Rapid Thermal Processing/Annealing (RTP/RTA) tube furnace with quartz furnace tub installed

Figure 4: High Temperature tube furnace with an alumina furnace tube installed

Figure 5: Controls for a 3-zone furnace--three temp controllers

Figure 6: Sealed specimen ampule (~ 4"in length)

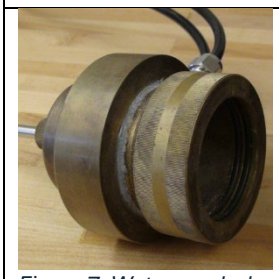

Figure 7: Water-cooled end cap

Figure 8: Boat inside furnace tube