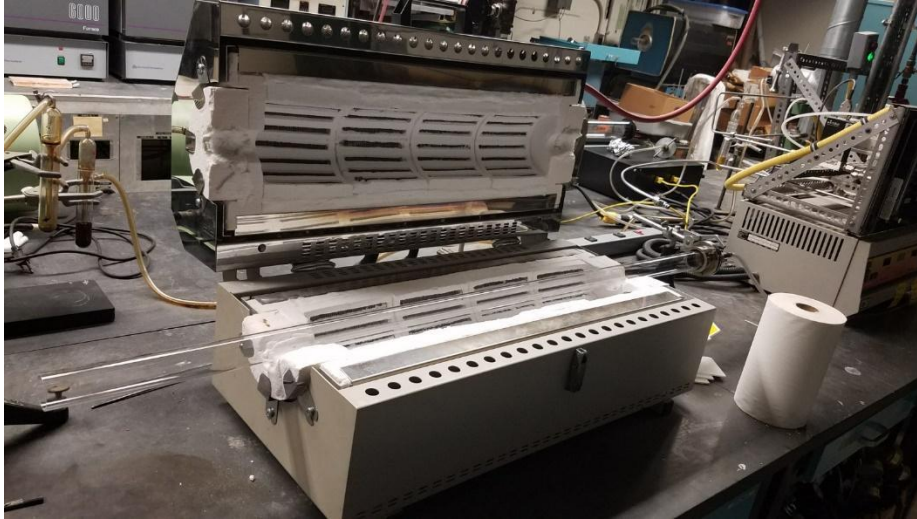


### Tube Furnace Standard Operating Procedure

- i. You must be trained and authorized to work with the tube furnace. Talk to Steve before embarking on using the tube furnace.
  - ii. Wear gloves at all times when handling the tube furnace and its components. If you touch it with finger oils, it will explode like a halogen bulb when the furnace turns on.
1. Get tube for tube furnace. It may be inside a cardboard mailing tube in the back corner of the furnace lab. Ensure that the furnace is not hot before opening by checking the attached temperature controllers on the right side. Check the endcap insulation to ensure that is not heavily cracked or damaged.

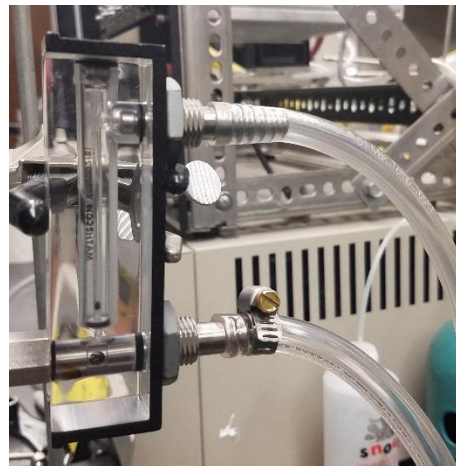


2. Use a hex-key to undo the gas input to the tube right side (metallic fixture)



3. Check the o-rings for cracks or kinks and clean the parts with ethanol if dirty.

4. Apply a small amount of vacuum grease (enough to feel, not to see) to the o-rings and metal parts and then reassemble the fixture, but do not tighten more than needed to keep the fixture together (very loose).
5. Attach the fixture to the tube, sliding the tube past both o-rings gently and then finger tighten. **DO NOT OVERTIGHTEN OR JAM THE GLASS AGAINST THE STEEL FIXTURE.** Sliding just past the o-ring will be sufficient.
6. Place the tube into the furnace and slide your sample (on a boat/holder) into the tube center region with a long rod that will not scratch or damage the tube. Close the furnace.
7. Gas flows from left to right and through the bubbler. **Ensure that there are no kinks or snags in the exhaust line and that the exhaust is running before opening the gas.**
8. Attach the outlet line on the left side of the tube. Select your flow gas from the available cylinders ( $N_2$ , Ar,  $O_2$ , Ar- $H_2$ ). Gas pressure should be  $\sim 10$ PSI, and not exceed 20PSI). Attach the small plastic tube from the cylinder to the bottom of the flowmeter beside the control panel and open the cylinder to the desired pressure.



9. Bubbler (behind the furnace) should be producing 1-5 bubbles/sec (if bubbler is too aggressive, reduce flow rate) (if bubbler is too slow increase flow rate or oil will get sucked into the gas line).
10. Allow the gas to run for roughly 5 minutes to ensure that the line, tube, and exhaust are purged from air.
11. Flip the green switch to turn on the tube furnace temperature control
12. Once proper airflow is established set up your desired temperature profile with the program control (page button) on the right most temperature controller (consult the manual if you are unsure)
  - a. First press -> SP (Set Point deg C)
  - b. Second press -> SPrr (SP ramp rate deg C/min)
  - c. Third press -> t1 (Dwell time)



The tube will cool from the gas flow after the program is executed. If you require a controlled cool please consult Steve for additional options. Be cognizant of the melting point of your boat such that you do not melt your boat with your set point temperature.

13. Flip the yellow switch on the left side of the control panel to output power to the furnace and press the function button (curved arrow) to initiate the program.

- a. Press the button one time during operation to check run time
- b. Hold the button during operation to pause the program
- c. Press the button during paused operation to resume
- d. Hold the button during paused operation to reset the program (do this step each time you run a new program)
- e. The right temperature controller reads the center temperature and the central tube temperature controller reads the right tube temperature.



14. Allow your program to run. You may leave if it is a long process, but check in periodically to ensure nothing has gone awry (tube has broken, line has come loose, endcap insulation is breaking off)

15. Once your dwell time has been completed flip the yellow switch off and allow the furnace to cool back to room temperature while the gas is flowing. If the gas flow through the bubbler seems to become much slower, increase the flow rate to prevent oil suction. Do not open the furnace until it is below 150C if you are in a rush. Do not touch the tube!

16. Once the furnace has cooled, close the gas cylinder, open the furnace, detach the left tube fixture and retrieve your sample and boat.

17. Switch off the control panel, ensure that the gas cylinder is closed, leave the left side gas outlet fixture off.