

Standard Operating Procedure for Ragauskas Lab Ovens

5 Laboratory Ovens:

- 3 Lab Bench thermal ovens – 475 A, 475 K, 475 M
- 1 Vacuum oven – 475 Q
- 1 Muffle oven – 463

Overall Safety Equipment to use:

- Heat resistant gloves
- Safety glasses or shield
- Laboratory coat
- Footwear which covers toes

Hazards

- **Electrical:** these are electrically powered ovens which may require up to 2 kW of power. This power can be lethal or may lead to fires if not handled correctly. Avoid touching any exposed wiring, do not let the ovens get wet, keep the area around the ovens clean and dry and inspect the external wiring regularly. Finally, do not attempt repairs on these ovens while power is still available to it.
- **Fire:** the temperatures generated can quickly cause a fire inside the oven and in the surrounding laboratory. Correct use of these ovens and prudence in choice of materials to put in the ovens will prevent fires from starting.
- **Chemical:** Oxidation, melting, violent reactions, out-gassing of your specimen or part may create chemical hazards such as toxic, or corrosive, or flammable gasses or liquids. Reactions with the materials used in the inner walls of the furnace might also occur. Think carefully about what you will be placing in these ovens so that potential chemical hazards do not become real chemical emergencies.

Safe Material Handling

- **It is very important not to put materials in these ovens that might damage the ovens or create a hazardous situation. Before you put anything in the oven make sure it is safe to heat to the desired temperature.**
- **Combustion:** since all heating will be in air, some materials such as paper, polymers, epoxies and even some materials when in powder form may catch fire or even explode.
- **Charring:** even if a combustible material does not actually catch fire it may smolder and char, giving of noxious gasses and blackening the oven

- **Melting:** if a polymer, glass, ceramic or metal melts in the oven it may permanently damage the oven's lining. In liquid form it may even attack the lining, melting holes in it and leaking onto the electronics below. Please remember that two materials placed in contact may have significantly lower melting temperature that either would have separately.
- **Oxidation:** most materials placed into the oven will oxidize. If you cannot tolerate any oxidation then you should place your sample in an air-free container such as an evacuated and close quartz tube.
- **Thermal Expansion:** A solid specimen being remelted in a crucible will expand while still solid and may crack the crucible. Once the material has melted it may leak out, damaging the oven.
- **Fumes:** these ovens are not vented directly to a scrubber or to the roof. Do not heat materials in these ovens if they will produce dangerous or annoying levels of such gasses.
- The use of **secondary containment** such as a larger crucible will prevent damage to the oven and it will protect your material.

General Operating Procedures:

1. Inspect the Oven.

Before using the furnace, inspect it and its surroundings for the following:

 - a. Make sure the electric power connections are made correctly and that the power cable is not damaged.
 - b. Make sure that the area around the oven is clear. The oven should have at least 6-inches clearance around it and there should be no paper or other flammable materials on or near it.
 - c. Inspect the inside of the oven to make sure it is clean and that no one has left their specimen or any other fixtures in it.
 - d. Do not use the oven until all necessary corrections have been made.



2. Oven in Use Notification

- a. Post a sign providing:
 - i. Your name, the times and dates you will be using the oven,
 - ii. the temperatures you will be using,
 - iii. the type of materials you are working with and
 - iv. a phone number, office number and/or email address where you can be contacted in case anything goes wrong.
- b. List any special precautions or comments that someone rescuing your specimen might need to know who is not familiar with your project!!!

3. Turning on the Thermal Ovens

- a. Turn on the oven by pressing the main power switch in the lower right.
- b. When the self-test is complete the display will show the current set point and temperature. At this point the controller is not only ready but it has already started heating.
- c. Some lab ovens are set and a standard temperature is maintained (If uncertain ask team members) 475A temperature is controlled by a knob and monitored by the thermometer in the oven.
- d. Press and hold the up or down arrow buttons to increase or decrease the temperature set point. Turn knob up (higher numbers for increased temperature) for 475A.
- e. When oven is at desired temperature, prepare to load the sample.



4. Loading the ovens

- a. **Before you put anything in the oven make sure it is safe to heat to the desired temperature.**
- b. Also consider the fact that your specimen is in contact with other materials and that additional reactions, possibly even melting, may occur.
- c. Make sure you have everything your are going to need: safety equipment, tools, etc.
- d. Make sure there is a clear and safe place to put the part when you eventually take it out.

- e. Once you have made these preparations use the following procedure to load your specimen.
- f. **Protective equipment** such as gloves, aprons, face shields and long tongs are available and are located near the ovens. Use what you think is appropriate for the materials and temperatures you are working with.
 - i. Picture Example of gloves:



- g. Open the oven's door.
 - h. Load your specimen or part. Make sure it is not touching the heating elements or an electrical short might occur when you close the door.
 - i. Close the door. The oven will start heating back to the set point.
 - j. Clean up the area around the oven and store the protective equipment properly.
5. Removing samples from oven
- a. Before you start to take anything out of the oven make sure you have everything you are going to need (PPE), especially if the specimen is still hot.
 - b. Make sure there is a **clear and safe** place to put your sample, or that your quench tank is ready, or if you drop the part that you have in place a plan for dealing with the situation.
 - c. Once you have made these preparations use the following procedure to remove your specimen.
 - d. **Protective equipment** such as gloves, aprons, face shields and long tongs are available and are located near the oven. Use what you think is appropriate for the materials and temperatures you are working with.
 - e. Open the oven's door.
 - f. Remove your specimen or part and, if still hot, carefully place it on a **heat resistant surface**.
 - g. Close the door. The oven will start heating back to the set point.
 - h. Clean up the area around the oven and store the protective equipment properly.
6. Finishing Up
- When you are completely finished using the oven, do the following:
- a. Turn off the oven.

- b. Clean up the inside of the oven.
- c. Make sure all of your specimens, parts, fixtures, etc. are removed and use compressed air to gently blow out the finer particles.
- d. If the oven is still hot you may have to wait a while for it to cool first.
- e. Clean up the area around the oven, including anything you dropped on the floor.
- f. Put all tools and protective equipment back in their proper places.**
- g. Remove your "Oven in Use" card and dispose of it.

7. Summary

- a. Use proper PPE
- b. Know your materials/samples placing in oven
- c. Label your samples – Notification card outside oven, labels on samples
- d. Check the oven before and after for damage/problems
- e. If you have questions on using any of the ovens, ASK!

8. Vacuum Oven Operating Procedure



- a. Place samples in oven
 - i. Check cold finger liquid volume
 - ii. Turn on cold finger element
 - iii. Turn on vacuum pump
 - iv. Open/close tubing valves
 - v. Close Gas and Vacuum knobs
 - vi. Press gently around glass window area to ensure a tight seal (pressure needle should be rising)
 - vii. Set temperature
- b. To remove samples:
 - i. Open Gas and Vacuum knobs to release pressure
 - ii. When falls to ~0 PSI on the gauge, open door
 - iii. Turn off the vacuum pump, cold finger
 - iv. Remove samples using proper PPE
 - v. Turn off the oven

9. Muffle Furnace Operating Procedures

- a. Location: Room 463, Barnstead International Type F62700
- b. Same as the general operating procedures and hazards however it is important to be more aware of how the higher temperatures can affect your sample, sample containers, and handling procedures.
 - i. Operating Temperature Range is 204 – 1000 C. (3 Hours +: 204 – 871 C; <3 hr: Max operation temperature is 1000 C, Ideal is 871 – 1000 C)
 - ii. Make sure proper PPE for high temperatures is used at all times when removing hot samples from the muffle furnace.
 - iii. Muffle refers to the ceramic collar envelops the furnace area.
- c. Do not use the oven in presence of flammable or combustible materials – fire or explosion may result. This device contains components which may ignite such material.
- d. Basic Operation:
 - i. When the controller is turned ON it will perform a short self-test and then display the measured value.
 - ii. To view the setpoint, press and release the UP or DOWN buttons. Continue to press until the desired setpoint value is displayed and then release the button. A few seconds after the button is released, the controller will accept the new value and revert to the home display.
 - iii. If oven temperature goes over a predetermined set-point, an alarm will sound.
 1. To set the deviation high alarm, press the SCROLL button until ldHi appears on the display. Press the UP or Down buttons to select the OTP value you desire. Recommended is 20 C above your working temperature to provide protection for your workload.
 2. In addition, this oven features a mechanical OTP relay device which disconnects power from the elements in an alarm condition.
 - iv. Further ramping program capabilities are described in the operating manual located next to the muffle furnace in room 463.
 - v. Furnace sample loading procedures:
 1. For best results, use center 2/3 of chamber.
 2. If heating a number of small parts, spread them throughout the center 2/3s of the furnace chamber.
 3. Keep objects away from the thermocouple.
 4. Use insulated tongs and mittens when loading and unloading the furnace.
 5. Always wear safety glasses.
 6. Use the hearth plates supplied to protect bottom of the chamber.
 7. Do not exceed a load of 25 pounds in the furnace chamber.
 - vi. Furnace unloading procedures:

1. Use insulated tongs and mittens when unloading.
 2. Always wear safety glasses
 3. Be prepared and have available an appropriate surface to place the samples on.
- vii. Cleaning
1. Run furnace up to 871 C when empty occasionally to burn off the contamination that may exist on the insulation and elements. Maintain 871 C for at least 4 hours to insure complete ashing of foreign materials.
 2. Wipe the exterior surfaces with lightly dampened cloth containing a mild soap solution.
- viii. If you will be using the furnace again within a few hours, it is recommended to leave the furnace on and operating at a reduced temperature (ex 260 C). This should help to increase the heating element life.

