Furnace B Training Notebook

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Before you begin...

- Complete the required safety training modules on UC Learning
  - Laboratory Safety Orientation (Fundamentals) 2013
  - Hazardous Waste Management
  - Compressed Gas Safety
- Submit a copy of your Training Transcript to Lab Manager
- Review the MSE Policies and Regulations
- Fill out the MSE 150, 250, 309 FAU Authorization Form with PI signature
- Provide your ENGR username to Lab Manger to set up Faces account
- Arrange a time for training with Lab Manager
- Schedule your reservation on Faces for your training
Tube B Furnace Operation

I. Startup
II. Furnace Configuration
III. Initial Tube Assembly
IV. Sample Loading
V. Final Tube Assembly
VI. Preparation
VII. Measurement Program
VIII. Start Measurement
IX. Sample Unloading
X. Cleanup
I. Startup – 1/1

1. Sign-in at the entrance of MSE 150

2. Sign-in on the instrument sheet

- Current Furnace Temperature
- PV Window
- SV Window
- Setting Temperature or State (End, Hold)
- Increase or End Program
- Decrease or Start/Hold Program
- Cursor Position or Edit Program
II. Furnace Configuration – 1/1

1. Furnace B (OTF-1200X) accommodates up to a 2” diameter quartz tube
2. Identify whether you can use an inert purge gas (i.e. Argon) or require a vacuum
III. Initial Tube Assembly – 1/2

1. Remove any dust or particulates on the outside and inside of your quartz tube with the provided air-gun

2. Place clean tube on provided tube stand

3. Insert the inner-flange on both ends of the tube first

4. Insert the o-ring, spacer, and o-ring on both ends

5. Slide and adjust the o-rings until the outer o-ring is about 3/8” from the edge of the tube
III. Initial Tube Assembly – 2/2

5. Slide the inner-flange against the o-rings until the inner-flange is slightly protruding past the tube

6. Unhook the latch and open the tube furnace

7. Place tube on tube furnace and center
IV. Sample Loading – 1/1

1. Orient the alumina foam block so that the hole is facing outward

2. Insert the alumina foam block and carefully push the foam block into the inlet side until it is positioned adjacent to the hot zone using the provided hook

3. Carefully insert your sample into the tube and center using the hook

4. Orient the alumina foam block so that the hole is facing out again and insert the into the other end of the tube toward the outlet side

5. Carefully push the foam block until it is positioned adjacent to the hot zone using the provided hook
V. Final Tube Assembly – 1/2

1. Identify the appropriate outer-flanges for the Purge Gas or Vacuum setup

2. Attach outer-flanges onto the inner-flanges and tighten screws using the hex key provided

3. Tighten the screws a little at a time and alternate among them

4. Holding onto the inner flange while tightening the screws help prevent it from becoming crooked
5. Unscrew and re-align the o-rings if the flanges are not parallel with the tube

6. Tighten the screws until the gap between the flanges are \( \approx \frac{1}{4} " \) wide

7. Check that there is a gap still between the tube and the outer flange when tightened

8. Attach quick-disconnect lines to the inlet and outlet ports for Purge Gas setup and just the inlet port for Vacuum setup
1. If using Purge Gas setup, continue to **Step 2**, else skip to **Step 7** for Vacuum setup

2. Fully open the valve to the Ar gas tank

   The pressure reading should increase if there is still gas

   If near empty, inform Lab Manager

3. Check regulator is set to 2 psi

4. Open the needle valve on inlet side slightly

5. Check rotameter or bubbles emerging from the DI Water Scrubber for desired flowrate

6. Close the furnace lid and lock, go to **VII. Measurement Program**
VI. Preparation – 2/2

7. Fully open the valve to the Ar gas tank

The pressure reading should increase if there is still gas

If near empty, inform Lab Manager

8. Check regulator is set to 2 psi

9. Turn off the needle valve on the inlet and outlet sides

10. Close the furnace lid and lock

11. Turn on the vacuum pump (requires prior approval first)
VII. Measurement Program – 1/4

1. Turn **ON** the furnace at the front panel if **OFF**

2. The furnace is in the normal state if the SV indicates a flashing **End**

3. Sketch the temperature heating program separately to determine the appropriate values to enter into the controller (see example below)

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Max Temp (&lt; 1 hour)</td>
<td>1200 °C</td>
</tr>
<tr>
<td>Max Continuous Temp</td>
<td>1100 °C</td>
</tr>
<tr>
<td>Normal Heating Rate</td>
<td>$\leq 10$ °C/min, $\leq 60$ °C/hr</td>
</tr>
<tr>
<td>Max Heating Rate</td>
<td>$\leq 30$ °C/min, $\leq 180$ °C/hr</td>
</tr>
</tbody>
</table>
II. Measurement Program – 2/4

4. Enter the input data of your heating program into the prompt (see example)

5. Press ← once to display C01 on PV

6. Set to 20 °C by using keystrokes ← to move cursor and ↑ ↓ to change value

7. Press Set key to complete input and display t01

8. Set to 240 min by using keystrokes ← to move cursor and ↑ ↓ to change value

9. Press Set key to complete input and display C02

10. Set to 450 °C by using keystrokes ← to move cursor and ↑ ↓ to change value
11. Press **Set** key to complete input and display t02

12. Set to 360 min by using keystrokes ← to move cursor and ↑↓ to change value

13. Press **Set** key to complete input and display C03

14. Set to 450 °C by using keystrokes ← to move cursor and ↑↓ to change value

15. Press **Set** key to complete input and display t03

16. Set to **-121** using keystrokes ← to move cursor and ↑↓ to change value

This last entry is used to issue a Stop Program command to stop the furnace and allow it to cool back to room temperature naturally.
VII. Measurement Program – 4/4

17. The table below shows the summary of all input

<table>
<thead>
<tr>
<th>Prompt</th>
<th>Input Data</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>C01</td>
<td>20 °C</td>
<td>Initial temperature</td>
</tr>
<tr>
<td>t01</td>
<td>240 min</td>
<td>Time to ramp temperature from 20 °C to 450 °C in 1st segment</td>
</tr>
<tr>
<td>C02</td>
<td>450 °C</td>
<td>Target temperature of 1st segment</td>
</tr>
<tr>
<td>t02</td>
<td>360 min</td>
<td>Time to hold temperature at 450 °C in 2nd segment</td>
</tr>
<tr>
<td>C03</td>
<td>450 °C</td>
<td>Target temperature of 2nd segment</td>
</tr>
<tr>
<td>t03</td>
<td>-121</td>
<td>Program End: Furnace will cool down naturally</td>
</tr>
</tbody>
</table>

18. Wait until **End** shows on SV window again

19. Confirm the program first before running

20. Press ← key to check program

21. Press Set key to cycle and check the program until **-121** is shown again
1. To start and run the program, press and hold the ↓ for about 2 seconds until run is displayed on SV window

   Note: Holding ↓ too long may start the program then immediately place the system in hold by accident

2. To hold the program, press and hold ↓ for 2 sec until -PE- flashes on SV window

3. To continue the program from a hold, press and hold ↓ for 2 sec until run flashes on SV window again

4. To stop the program, press and hold ↑ for 2 sec until End flashes on SV window
IX. Sample Unloading – 1/1

1. The program is completed when the normal state shows *End* flashing with current temperature in the PV window.

2. For the Vacuum setup, continue to **Step 2**, else skip to **Step 7** for the Purge Gas setup.

3. Slowly open the needle valve on the inlet side to vent in Ar.

4. Allow the pressure to rise close to Atmospheric pressure, checking the physical gauge.

5. Slowly open the needle valve on the outlet side to prevent over-pressurization of tube.

6. Shut off the vacuum pump.
IX. Sample Unloading – 1/1

7. Close the valve to the Ar tank first then close the needle valve

8. Remove the outer-flanges by loosening the screws

9. Open up the furnace and remove the alumina foam blocks with the provided hook

   Note: Remove blocks with the hook in the up position and lift the foam block up while sliding out of the tube

10. Clean the hook first and push or pull the sample out of the tube
X. Cleanup – 1/1

1. Remove the o-rings, spacer, and inner-flanges from your tube

2. Clean all items and place them back into the designated drawer space

3. Return your own tube, o-rings, and alumina blocks back to your drawer

4. Close the furnace tube and turn OFF

5. Confirm the gas tank valve is fully closed

6. Sign-off on the instrument sheet and on the entrance sheet