Furnace B & C OTF-1200X Training Notebook

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Before you begin...

- Complete the required safety training modules on UC Learning
  - Laboratory Safety Fundamentals
  - 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 Manager to set up Faces account
- Arrange a time for training with Lab Manager
- Schedule your reservation on Faces for your training
Furnace B & C OTF-1200X Furnace Operation

I. Startup
II. Initial Tube Assembly
III. Sample Loading
IV. Final Tube Assembly
V. Turning On Purge Gas
VI. Measurement Program
VII. Start Measurement
VIII. Sample Unloading
IX. Cleanup
I. Startup – 1/1

1. Sign-in on the appropriate instrument sheet located on the table: Furnace B or Furnace C

- 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. 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 following on one end of the tube in order
   1. Inner-flange
   2. O-ring
   3. Spacer
   4. O-ring

4. Repeat Step 3 for the other end of the tube
II. Initial Tube Assembly – 2/2

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

6. Confirm that outer o-ring is less than ~ 3/8” or ~ 10 mm from the edge of the tube to prevent damage to tube when sealing

7. Unhook latch and open tube furnace

8. Place tube on tube furnace and center
III. Sample Loading – 1/1

1. Orient 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 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.
IV. Final Tube Assembly – 1/2

1. Identify the appropriate outer-flanges for Furnace B or C

DO NOT MIX AND MATCH DIFFERENT FLANGES!!!

Furnace B

Furnace C
2. Align the inner-flange to match desired orientation of outer-flange and tighten screws using the hex key provided a little at a time alternating among them.

3. Holding onto inner-flange while tightening the screws help prevent it from becoming crooked.

4. Unscrew and re-align o-rings if flanges are not parallel.

5. Tighten the screws until the gap between the flanges are ~ ¼” or 5 mm wide.

6. Attach quick-disconnect lines to the inlet and outlet ports.
V. Turning On Purge Gas – 1/1

1. Turn the switch towards the desired purge gas: Argon or Air

2. Turn the rotameter valve to adjust desired flowrate

<table>
<thead>
<tr>
<th>SCFH</th>
<th>mL/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>47.6</td>
</tr>
<tr>
<td>0.5</td>
<td>238</td>
</tr>
<tr>
<td>1.0</td>
<td>476</td>
</tr>
</tbody>
</table>

3. Close the furnace lid and lock
VI. 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)

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### Parameter

<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 , ^\circ \text{C/min} ) ( \leq 60 , ^\circ \text{C/hr} )</td>
</tr>
<tr>
<td>Max Heating Rate</td>
<td>( \leq 30 , ^\circ \text{C/min} ) ( \leq 180 , ^\circ \text{C/hr} )</td>
</tr>
</tbody>
</table>
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
VI. Measurement Program – 3/4

11. Press \textbf{Set} key to complete input and display t02

12. Set to 360 min by using keystrokes $\leftarrow$ to move cursor and $\uparrow \downarrow$ to change value

13. Press \textbf{Set} key to complete input and display C03

14. Set to 450 °C by using keystrokes $\leftarrow$ to move cursor and $\uparrow \downarrow$ to change value

15. Press \textbf{Set} key to complete input and display t03

16. Set to -121 using keystrokes $\leftarrow$ to move cursor and $\uparrow \downarrow$ 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
**VI. Measurement Program – 4/4**

17. The table below shows the summary of all input for the example:

<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
VIII. Sample Unloading – 1/1

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

2. Turn the rotameter valve to shut off purge gas

3. Remove the outer-flanges by loosening the screws

4. 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

5. Clean the hook first and push or pull the sample out of the tube
IX. 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 rotameter valve is fully closed

6. Sign-off on the instrument sheet and note the tank pressure

**IF TANK PRESSURE FALLS BELOW 200 PSI CONTACT THE LAB MANAGER**