Furnace B Training Notebook

Lab Manager: Dr. Perry Cheung

MSE Fee-For-Service Facility

Materials Science and Engineering

University of California, Riverside
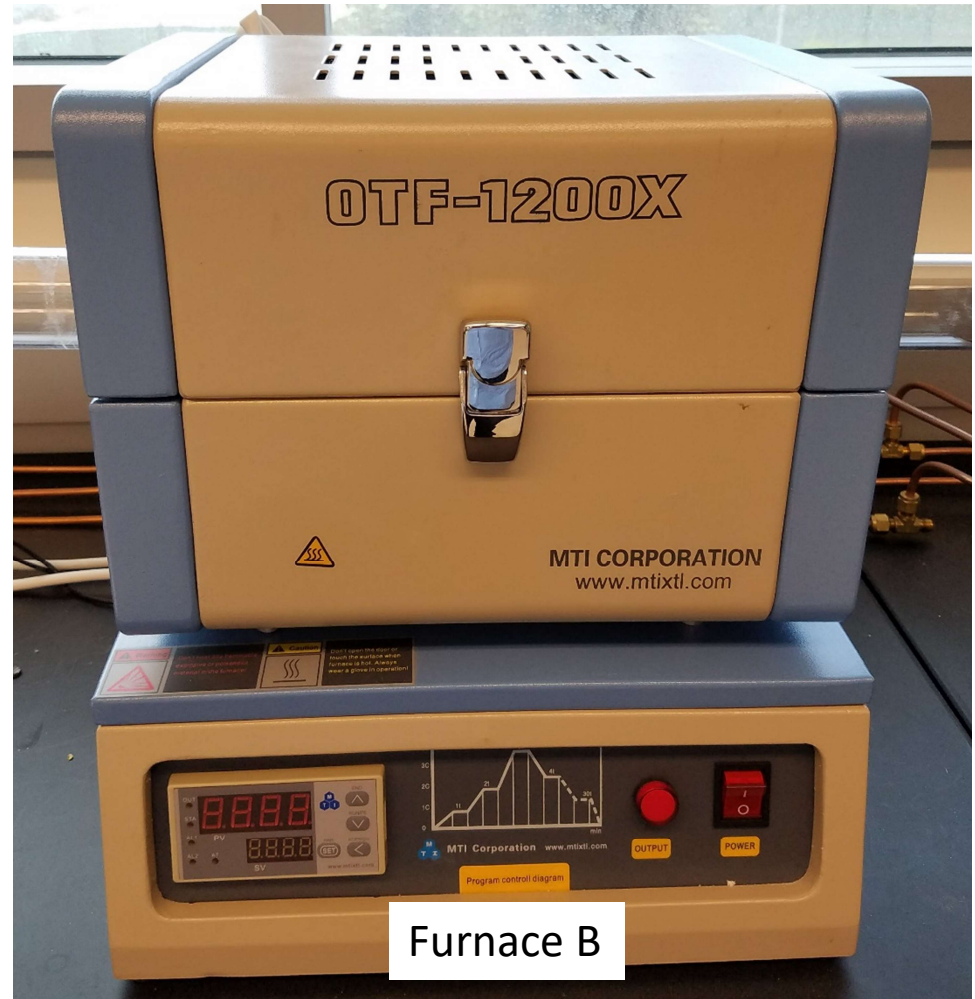
April 21, 2017 (rev. 2)

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
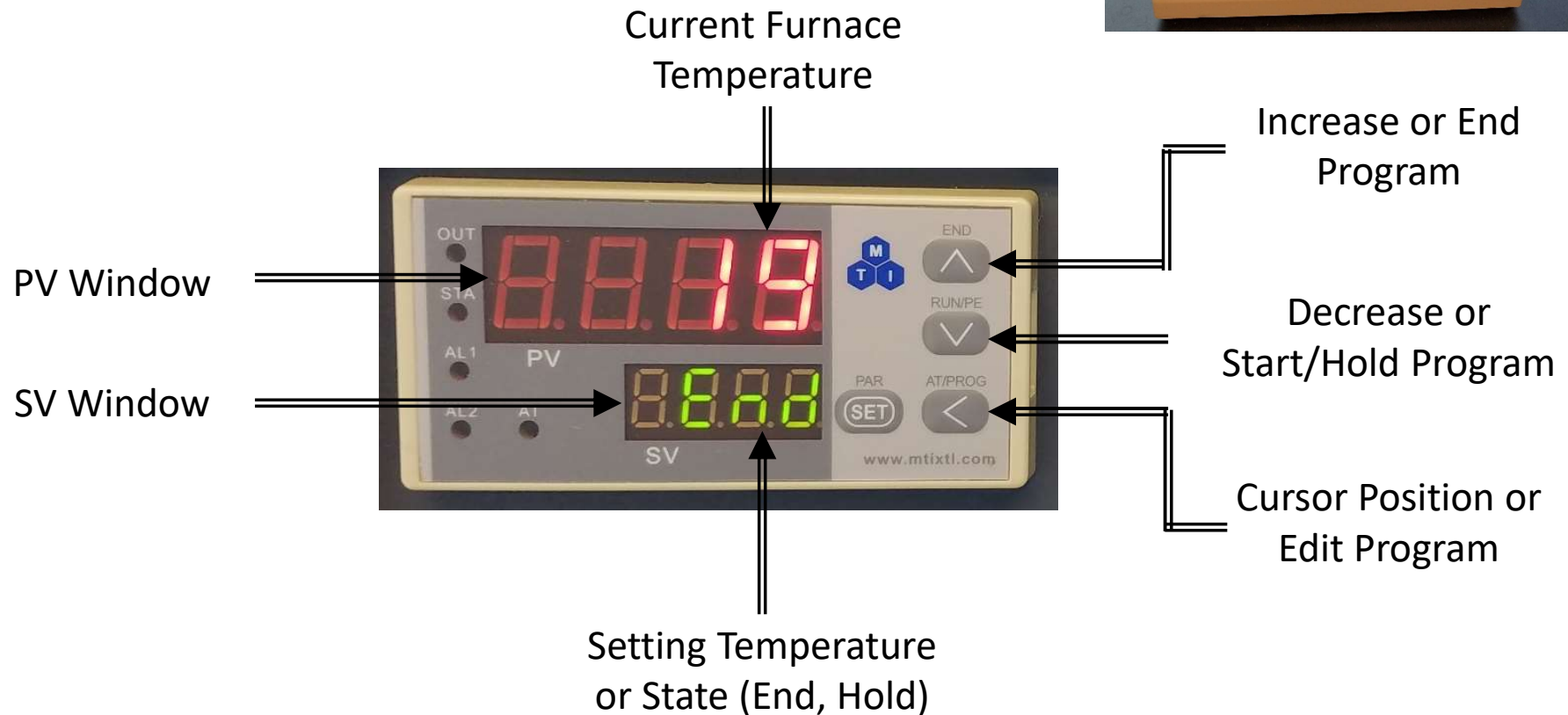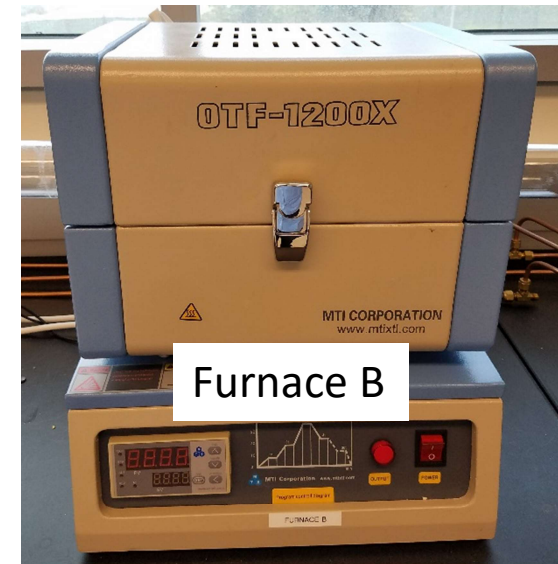
❑ Schedule your reservation on Faces for your training

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

Furnace B

I. Startup – 1/1

1. Sign-in at the entrance of MSE 150

2. Sign-in on the instrument sheet

Furnace B

Current Furnace Temperature

Increase or End Program

PV Window

Decrease or Start/Hold Program

SV Window

Cursor Position or Edit Program

Setting Temperature or State (End, Hold)

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

Furnace B
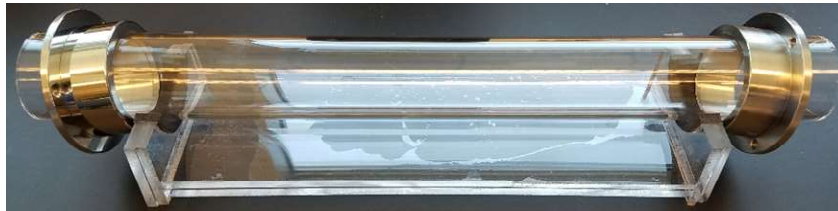

Purge Gas Setup

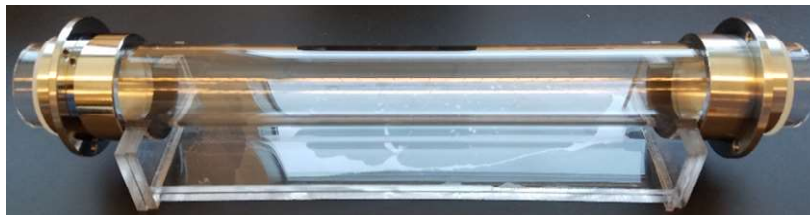Vacuum Setup
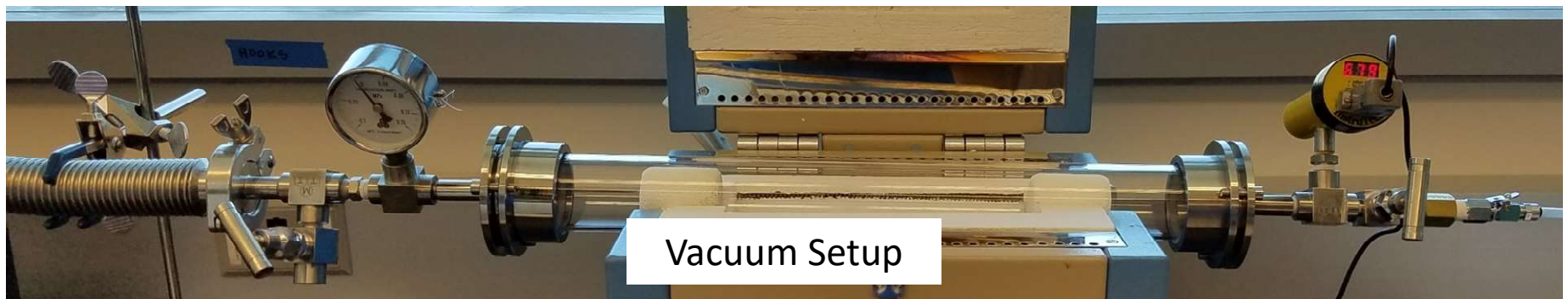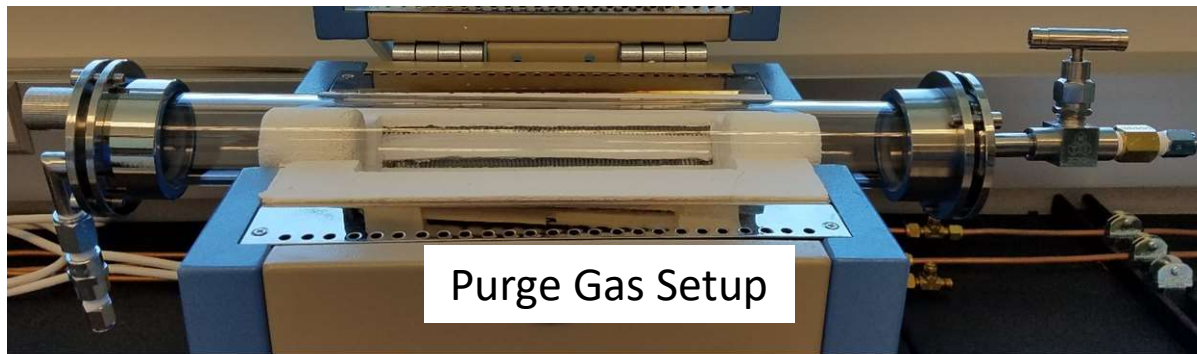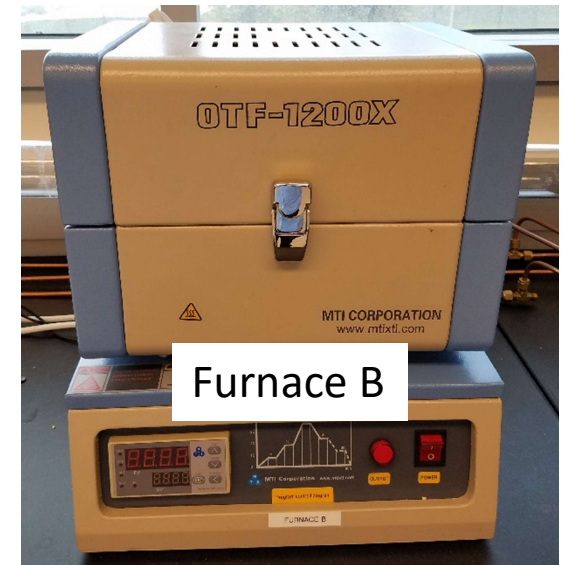
III. Initial Tube Assembly – 1/2

1. Remove any dust or particulates on the outside and inside of your quartz tube with the provided air-gun
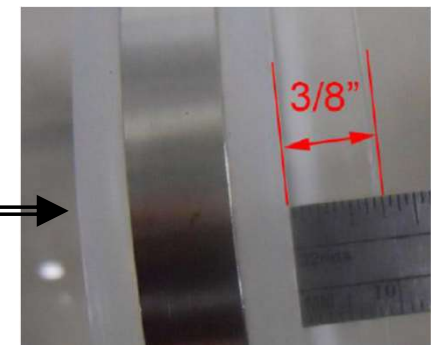
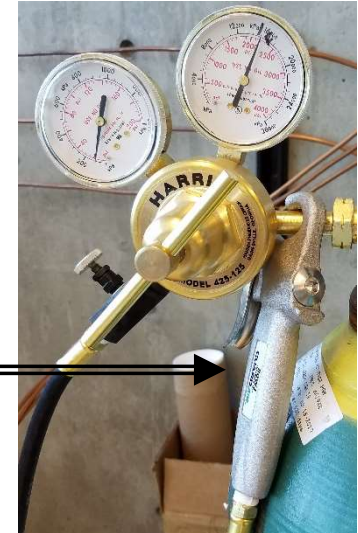2. Place clean tube on provided tube stand
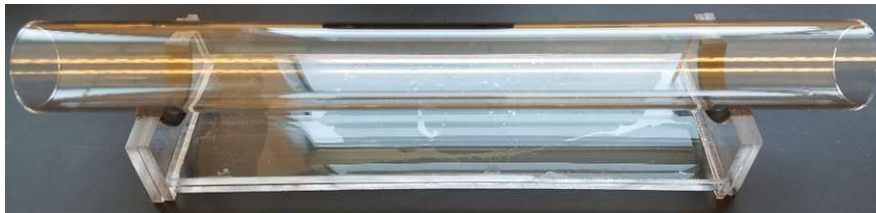
3. Insert the inner-flange on both ends of the tube first

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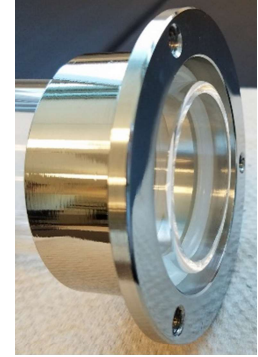

5. Slide and adjust the o-rings until the outer o-ring is about 3/8" from the edge of the tube
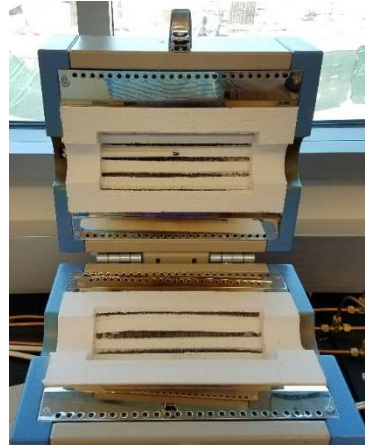

3/8"

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
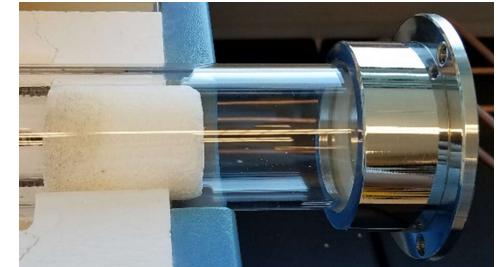

6. Unhook the latch and open the tube furnace

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
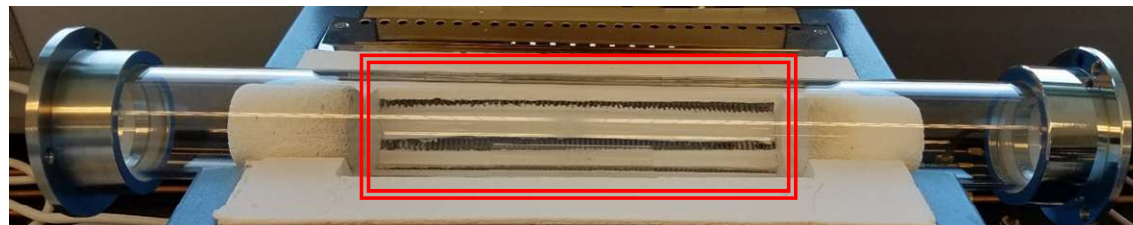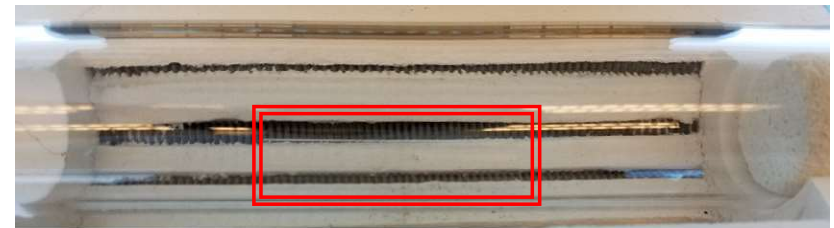
Hot Zone

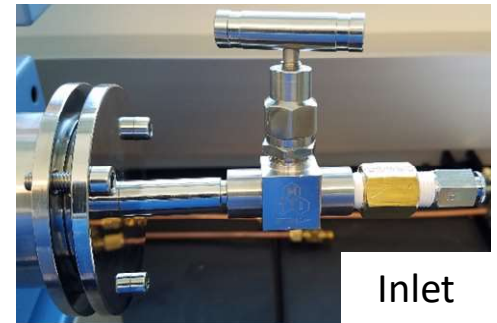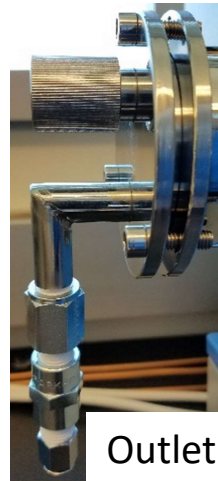# V. Final Tube Assembly – 1/2

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

Inlet

Outlet

Vacuum Setup

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

Outlet

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

Inlet
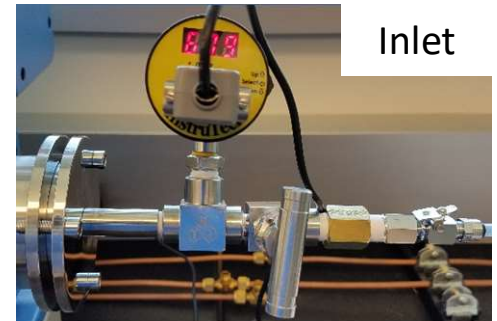
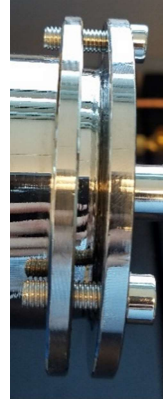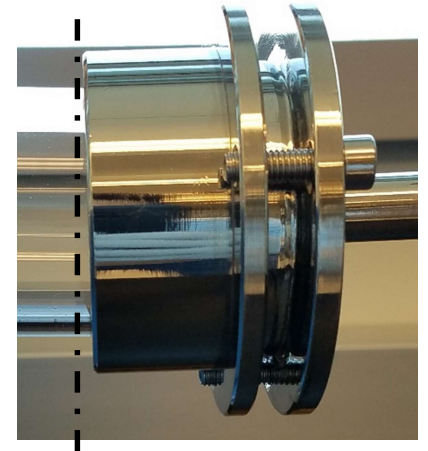# V. Final Tube Assembly – 2/2

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 ≈ ¼" wide

7. Check that there is a gap still between the tube and the outer flange when tightened
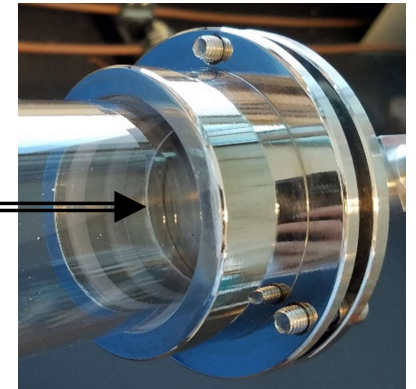
8. Attach quick-disconnect lines to the inlet and outlet ports for Purge Gas setup and just the inlet port for Vacuum setup

VI. Preparation – 1/2

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

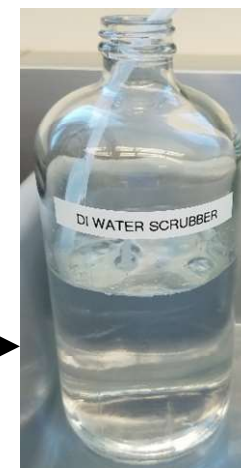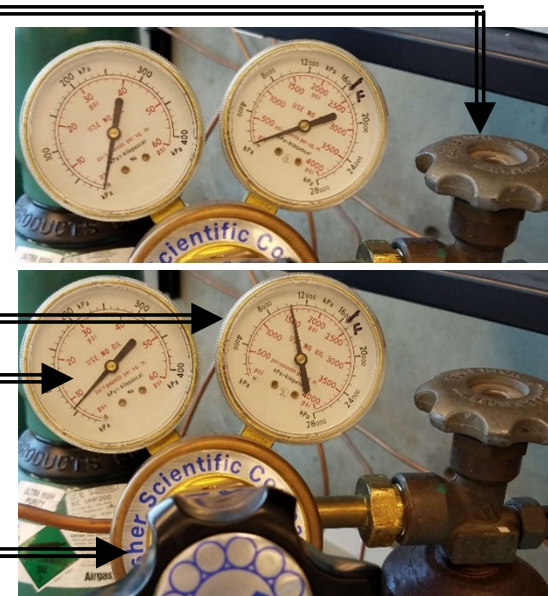8. Check regulator is set to 2 psi

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**)

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

On

Off

POWER

2. The furnace is in the normal state
   if the SV indicates a flashing **End**
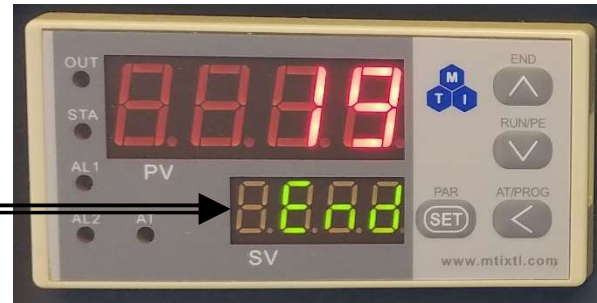
3. Sketch the temperature heating program separately to determine the
   appropriate values to enter into the controller (see example below)
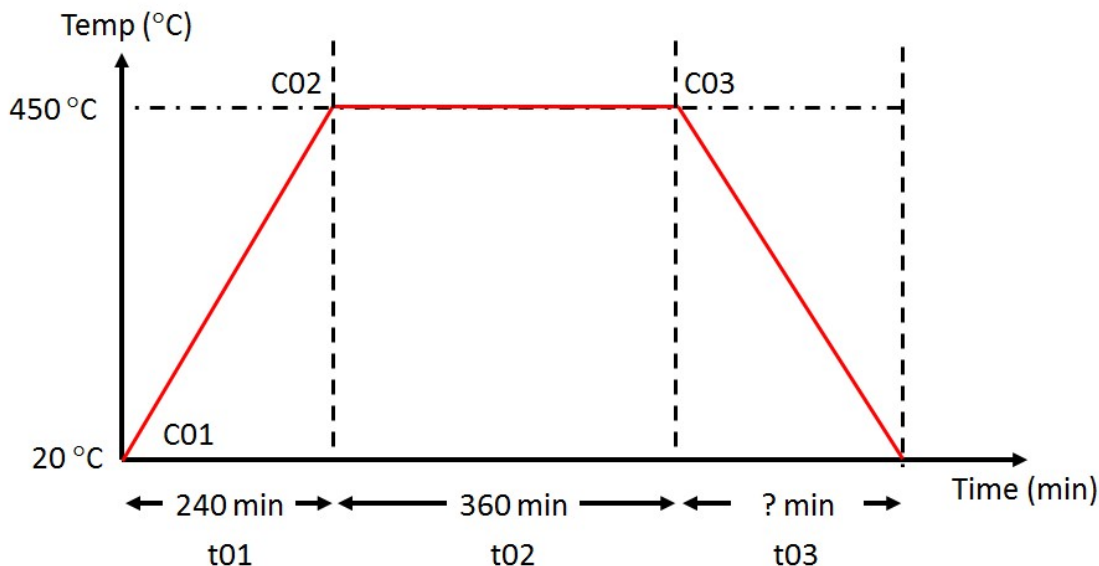
Parameter	Value
Max Temp (< 1 hour)	1200 °C
Max Continuous Temp	1100 °C
Normal Heating Rate	≤ 10 °C/min ≤ 60 °C/hr
Max Heating Rate	≤ 30 °C/min ≤ 180 °C/hr

Temp (°C)

450 °C

C02

C03

20 °C

C01

← 240 min → ← 360 min → ← ? min →   Time (min)

t01   t02   t03

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

17. The table below shows the summary of all input

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

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
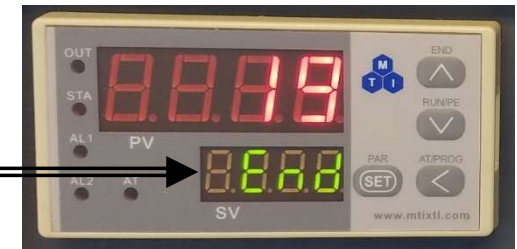
VIII. Start Measurement – 1/1

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
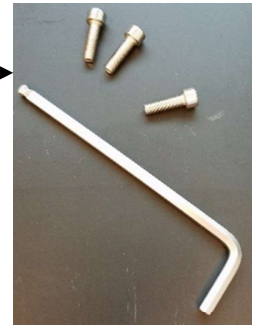

6. Shut off the vacuum pump
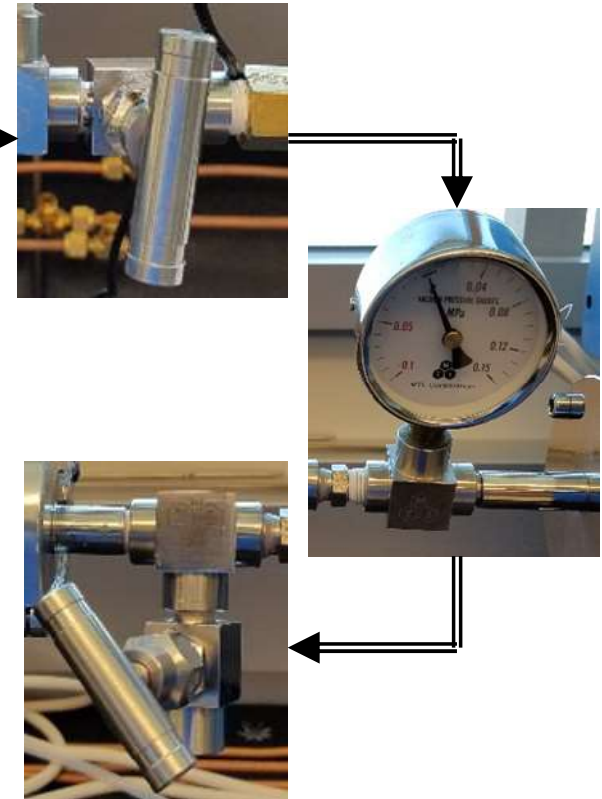
IX. Sample Unloading – 1/1

7. Close the valve to the Ar tank first then close the needle valve
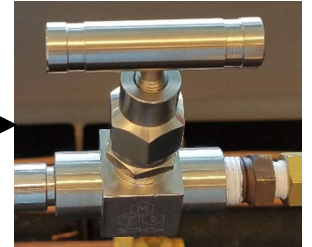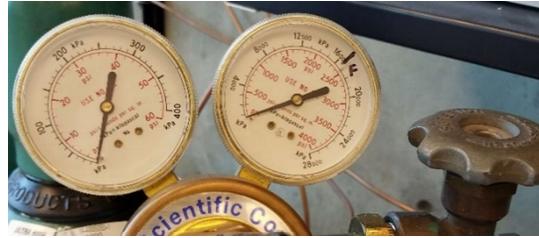
8. Remove the outer-flanges by loosening the screws

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

On

Off

5. Confirm the gas tank valve is fully closed

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