Horizontal Furnace Standard Operating Procedure - Quick Check List

Loading

1. Load boat into tube and place in furnace
2. Close clam-shell
3. Connect gas input line
4. Start gas flow
5. **Turn off outputs**, turn on main controller power
6. Open EZ-Zone, connect to COM2
7. Create profile on all 3 zones/devices.
8. Start profile on all 3 zones/devices.
9. **TURN OUTPUTS ON!!**
10. Update profile info in Sticky Notes.
11. Close sash, label sample and leave any required warning signs.

Unloading

12. **Turn off output switches**
13. Open clam-shell to cool faster
14. ***Turn off gases***
15. Disconnect gas input line
16. *Use gloves* to remove hot samples
17. Inform next user furnace is available

BE SAFE! Use Thermal Gloves.

Note: Each lab member should have their own set of furnace tubes and glass/quartz boats. Make sure to note the material of your boat and tube (pyrex vs. glass). **If you're not 100% confident which material you have, ASK!** Pyrex should not be used > 300^{o}C.

Note: Profile's are saved to the device, but it can only remember 4. If you've previously set up a profile, check the Sticky Notes on the Desktop to see if your profile has been updated.

This furnace has been tested at 10^{o}C/min to 1000^{o}C.

Full set of guidelines are saved on the group Wiki and Desktop of Controller Computer with filename 'Horizontal Furnace Standard Operating Procedure'

Horizontal Furnace Standard Operating Procedure

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Safety

- Use thermal gloves when handling tube. Hot glass looks the same as cold glass.
- Whenever clamshell is open, output switches must be OFF/DOWN.
- Label all samples and post appropriate warning signs
- Hydrogen gas is flammable and explosive. Check flammability limits and dilute with He whenever possible.
- Check valves and a 3 way valve separate oxygen lines from H2 lines. Do not adjust gas manifold without consulting Megan or Daniel.
- High currents used to power furnace. Report any frayed wiring to Megan. Turn off main power (down) and unplug (the plug is yellow and across the room above the 5890 GC)
- Do not use broken tubes. Have glass shop grind them down and create a bevel edge (on quartz) or flame polish (for Pyrex)
- When in doubt, turn output switches off (down) to avoid run away temperatures.

Procedures

Loading your sample

1. Load your catalyst into your boat(s).
2. Carefully slide the boat into your horizontal furnace tubes. Check the material of both the boat and tube. Pyrex should not go above 300^{o}C!
3. Open the furnace clam-shell and place the tube inside with the boat(s) centered as much as possible.
4. Insert your Teflon plug into your tube and attach the plastic tubing labeled ‘Gas Input’. Use a wrench to tighten it ¼ turn past finger tight.
5. *Leave 3 fingers worth of room between the side of the furnace shell and the end of the Teflon plug to ensure the Teflon and orings do not melt.*
6. Start your gas flow.
    a. Valves on the side panel in the vertical position are ON, and are OFF in the horizontal position.
    b. If you are flowing H2, the 3 way valve marked ‘S’ needs to be pointed DOWN towards the red tape marked H2.
    c. If you are flowing air, the 3 way valve marked ‘S’ needs to be pointed UP towards the green tape marked OX.
    d. Use the corresponding rotameter control the gas flow. Be sure to regularly check the flow rates as they occasionally drift over time.
7. Flip 3-output switches down to avoid any initial heat when the power is turned on.

8. Flip the power switch up on the right side of the control box. The lights on the controllers should turn on.

Setting up the controller

9. Make sure the USB marked 'HF-Controller' is plugged into the right hand computer, labeled 'Controller'
10. Open 'EZ-Zone Configurator'. It is on the taskbar with a big red EZ and green flag.
11. Check 'Configure a device while communicating with it' and select 'Next'
12. Select 'COM2' for the Communications Port and then hit 'Next' This is important… if you chose the wrong port, you may end up adjusting Megan's furnace, and she'll be very angry.

Creating a profile

13. 3 Controllers will appear for each zone on the furnace. You will need to repeat the following steps for each device / zone. Select the first device and hit 'Next'
14. Select 'Profile' to expand the section.
15. Choose Profile 1, 2, 3, or 4.
16. Select the 'Step Type' and input the required information.
    a. For ramping steps select 'Time'. Input the final temperature you want to ramp to in 'Target Set Point Loop 1'. Input the amount of time to reach that set point in Hours, Minutes, and Seconds. *Note: Inputting values for 'Rate' will not substitute for the ramp time. You must calculate and input the time for ramping.*
    b. For soaking steps select 'Soak'. Input the soak temperature in 'Target Set Point Loop 1' and the length of soak time in Hours, Minutes, Seconds.
17. Select 'Next' to set up the next step.
18. Add a final step with step type 'End' to stop the profile. Nothing else (temperature or time) needs to be set for the 'End' step. The controllers are set up to return to the 'Closed Loop Set Point' whenever a profile is not running, which allows it to cool automatically.
19. Select 'Back' to repeat for the other zones.

Starting a profile

20. Before beginning a profile, all the set points need to be the same on all 3 zones.
    a. Setpoints are listed in green on the bottom on the controller interfaces, process values are in red on the top. If the setpoints are not the same, manually adjust the setpoints using the up and down arrows on the controller interface and allow a few minutes for them to ramp to the same temperature.
21. On the EZ-Zone software, select the device and hit 'Next'.
22. Select 'Operations' to expand then menu.
23. Select 'Profile Status'
24. Type in the profile number that you've set up for your heat treatment in 'Profile Start'
25. Use the drop down menu under 'Profile Action Request' and choose 'Profile'. This starts the profile immediately.

26. Select 'Back' and repeat this for the other zones. The timing will be off by however long it takes you to start them… so be quick!
27. **FLIP ALL OUTPUT SWITCHES UP!** Otherwise you won't be heating.
28. Update the Sticky Note profile information with your name and date.

While it is running…

29. The controller interfaces should be flashing between 'rP1' and the process value whenever it is ramping temperature. This just indicates that the controller is ramping up the temperature.
30. Whenever the controller is outputting power to the furnace, the red LED lights under the interface and above the output switches will blink.
31. Check the gas flow rates in case of drift.
32. Check setpoint vs. process value
    a. Is it significantly different? >5 ^{o}C? If yes,…
    b. Is the process value oscillating?
        i. If yes, check the PID values. The controllers are set to Auto-tune and change parameter while the temperature increases, but occasionally it can run-away from itself. Open EZ-Zone, open the device with the oscillations, select 'Setup', select 'Control Loop', input '36' for 'Heat Proportional Band', input '172' for 'Time Integral', and input '0' for 'Time Derivative'.
        ii. If no,…
    c. Is it outputting power (red LEDs lighting up) but not increasing temperature? If yes and it is not delayed heating (give it a few minutes to respond), turn all outputs off and contact Megan.

Unloading sample

33. **Flip output switches down** to turn off power output to the furnace.
34. Open clam shell to allow for faster cooling.
35. **Turn off gases** on the hood side panel.
36. Disconnect gas input tubing from Teflon plug.
37. Wear termal gloves! Lift tube from furnace and carefully slide boat out of tube.
38. Inform next user the furnace is available.

Default Values

39. Default ramp rate = 3^{o}C/min
40. Closed loop set point = 20^{o}C
41. Proportional Heat Band = 36
42. Time Integral = 172
43. Time Derivative = 0
44. Low Set point = 20^{o}C
45. High Set point = 1000^{o}C
46. COM2

Notes

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