

Ammonia Treatment Instructions

Page 1 of 5

Original version by Douglas Call
Updated January 21, 2009 by David Jones

Department: Chemical Engineering

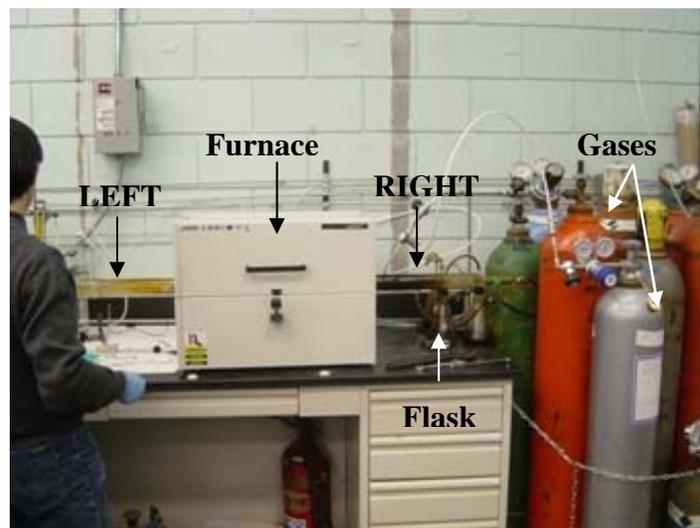
Location: Fenske 102 (access thru room 103)

Contact: BP Matthew Holbrook
161 Fenske
bmh200@psu.edu
Office: 814-865-9734; Mobile: 484-201-7400

Getting Started: Call or email contact above to schedule a time to use the furnace 9 AM - 5 PM. The Logan group has a gas cylinder with 5% Ammonia in Helium (150 ft³) stored in 102 Fenske where treatment is performed. Use this gas mix for treatment. When this cylinder is empty, email David Jones (dwj123@psu.edu). The cylinder costs ~\$230 and requires about 7-10 days for delivery. The Argon gas used for part of the procedure is for general use in the lab and we do not need to purchase a new one when it is empty.

Treatment Capacity: The furnace capacity is limited, so plan accordingly. For example: 4 large brushes can fit into the furnace at one time, so if you need 8 brushes, schedule two sessions to use the furnace (about 4 hours per session).

IMPORTANT: Check the gas pressure in the Ammonia and Argon cylinders before you begin. This treatment requires Argon with a tank pressure of >200 psi and you do not want to empty the cylinder before you finish the procedure. When you complete the treatment, email David Jones (dwj123@psu.edu) and let him know how much Argon and how much Ammonia gas mix are remaining.



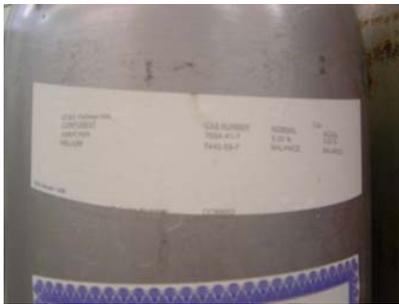
Ammonia Treatment Instructions

Page 2 of 5

Instructions:

1. Before starting, make sure that the **FUME HOOD** located on the right side of the wall as you enter the lab is **ON**. Check by locating the fume hood switch which is below the bench level and to the right of the hood. If it is not on, turn the switch to **ON**.

2. Locate the 5% ammonia gas mix. The cylinder label reads “Ammonia 5%” and “Helium balance”. It is **SILVER** and looks like this:



Make sure that the gas supply is **OFF** and that the **GREEN** knobs connected to the tubing are closed.



3. Argon is the **ORANGE** cylinder. Make sure that the gas supply is **OFF** and that the **GREEN** knobs connected to the tubing are closed.



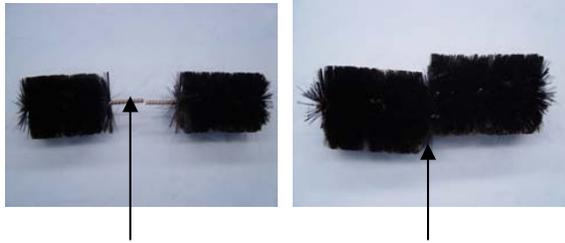
4. Locate a quartz boat and place your samples inside. **DO NOT OVERFILL THE BOAT:**



Ammonia Treatment Instructions

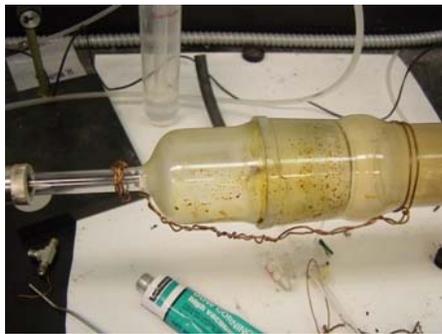
Page 3 of 5

For large brushes do not use the boat, instead, tie them together using titanium wire:



Tie ends together with titanium wire

5. Locate the far left end of the glass extending from the furnace:



Unravel the copper wire, and slide the lid off by **SLOWLY** twisting back and forth. You may need to rotate the lid back and forth in order for it to come off. Once the lid is off, wet a Kimwipe with acetone and wipe to remove the grease. When the lid is removed, insert your samples (boat or large brushes):



Slide the sample all the way until it is in the middle of the furnace. Use a wire rod to

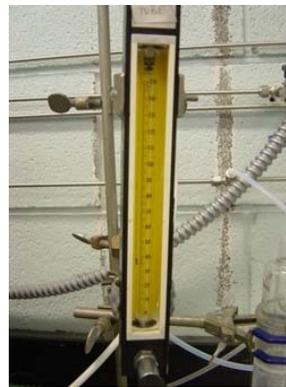
assist. Lift up the lid on the furnace to ensure that the boat is centered:



CLOSE and **LOCK** the lid when done!

6. Re-grease the inlet if needed. Reattach the glass lid. Reattach the copper wire as shown in the previous picture. Make sure that the **WHITE** tube is connected to the top of the glass lid. If a **CLEAR** tube is connected, you need to change it to the **WHITE** tube.

7. After ensuring that the glass lid is on securely, turn on the **ARGON** gas supply. Make sure that the three-way gas valve connecting the **ARGON** line is pointed to the left (facing the furnace). Make sure that the **ARGON** is delivering around **20 psi** by checking the **ARGON** regulator. Check the gas flow meter (see next picture) to ensure that the **ARGON** is and set to **150 cc**. Make sure the silver ball is at the 150 cc mark:



Ammonia Treatment Instructions

Page 4 of 5

Also check the Erlenmeyer flask that sits to the right of the furnace and make sure that there are gas bubbles present in the oil trap.

8. With the **AMMONIA** gas still closed, leave the **ARGON** gas supply on for **20 minutes**.

9. During these 20 minutes, begin programming the furnace. The furnace control box is the **BLUE** box with a #2.



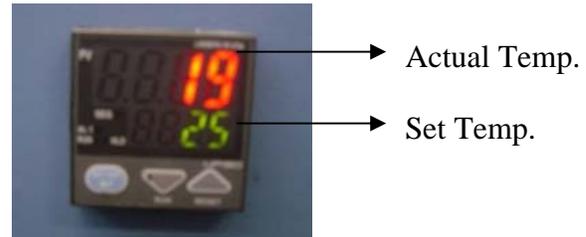
10. Looking at **FURNACE #2**, you will see the following:



The **POWER** switch on the **LEFT** turns the furnace on and off. To set the program for ammonia treatment, use the **RIGHT** control box.

The top number in **RED** is the actual temperature. The bottom number in **GREEN** is the set temperature.

The **RIGHT** control box has a **BLUE** button, a **DOWN** arrow, and an **UP** arrow:



Press and hold the **BLUE** button on the **RIGHT** control box until the display changes to “**node rES**”. Press the **BLUE** button once more until you see “**PrG**”.

Press the **UP** arrow until the display reads “**1**”.

Then use the **BLUE** button to scroll through the menu items until you see **SP1**. This is the first temperature set point. Set it to **700 °C**.

Scroll again using the **BLUE** button until you see “**tn1**”. This sets the rate at which you want to reach 700 °C. Using the **UP** or **DOWN** arrows enter **0.30**, which means 30 minutes.

Scroll again using the **BLUE** button until you see “**SP2**”. This sets the temperature for holding the furnace at 700 °C. Using the **UP** or **DOWN** arrows enter **700 °C**.

Scroll again using the **BLUE** button until you see “**tn2**”. Set for **1.15** hour which will ensure at least one hour treatment time.

Scroll again using the **BLUE** button until you see “**SP3**”. This sets the turn-off temperature. Set it to **20 °C**.

Ammonia Treatment Instructions

Page 5 of 5

Scroll again using the **BLUE** button until you see “**tn3**”, which is the turn off rate. Hold the **DOWN** arrow until it reads **OFF**. Scroll until you see “**A1**”, which stands for the alarm. Set this value to **710 °C** using the **UP** or **DOWN** arrows. This function will shut down the furnace if it gets hotter than 710 °C.

The program is now set.

If the furnace was **ON** when you started entering the program, switch the furnace **OFF** using the switch on **LEFT**. Then switch the furnace back **ON** using the same switch. This ensures the electrical breaker will not be tripped.

Press **RESET** on the **MIDDLE** switch. You will see an orange light go off.

Check to make sure that the **ARGON** gas is still **ON** and that the **AMMONIA** gas is **OFF**.

Press **RUN** on the **RIGHT** control box. An orange light will turn on and you will hear a click indicating that it has started.

The temperature will begin rising.

11. It will take **30 minutes** for the furnace to heat to 700 °C.

12. After 30 minutes, ensure that the temperature displays 700 °C. If it does, turn off the **ARGON** gas feed, and turn on the **AMMONIA** gas feed to a pressure around **10 psi**. Check the flow by reading the flow meter as shown above and make sure the **SILVER** ball is at the **150 cc** mark. Check the Erlenmeyer flask located to the right of the furnace to make sure that there are bubbles in the liquid. It will take a few minutes for the bubbles to appear because

helium is much lighter than argon. **Do not sit until you see the bubbles.**

13. With the **AMMONIA** gas on, wait in lab for **one hour**.

14. After one hour, turn off the **AMMONIA** gas and turn on the **ARGON** gas.

15. The temperature will start to decrease. To ensure the program has ended, turn the furnace completely **OFF** using the switch on the **LEFT** (see picture above).

16. Once the furnace temperature has cooled to <300 °C you can open the lid to allow the cooling process to speed up.

17. Once the temperature is <50 C, remove the lid on the glass tube following the instructions from above. Using a wire rod, reach inside the tube and remove your samples. Be careful not to push your samples in farther. If necessary, the **RIGHT** end of the glass tube can be removed in order to push samples toward the **LEFT** opening.

18. Remove your samples, and leave the quartz boat on the lab bench.

19. Turn **OFF** the Argon cylinder using the knob located on top of the cylinder.

20. Before leaving, make sure that all the gases are **OFF**.

21. Email David (dwj123@psu.edu) and tell him how much **AMMONIA** gas mix and how much **ARGON** gas is remaining when your experiment is over.