Ammonia Treatment Instructions
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Updated January 21, 2009 by David Jones

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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.
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:

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:
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:
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 0.30, which means 30 minutes.

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.
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.