

# Making Solid Ammonia

Solid Polarized Target Group

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## 1 Cleaning and Preparation

Thoroughly clean the surface that will be used to seal the cylinder, both tube rims and the lid. Also clean any utilities that will come in direct contact with the material such as the screen mesh containers and the pestle. The indium used to make the seal should also be wiped down with alcohol.

Wear Cryo-Gloves and safety goggles as appropriate throughout this procedure.

Fill up a large dewar with liquid Nitrogen for use throughout this procedure. Drill very small holes ( $< 0.070$  inches  $\phi$ ) in 2 30ml Nalgene bottles (1 for beads, 1 for scraps) which will allow liquid Nitrogen to keep the collected samples cold in storage and free of any air bubbles where it can be lost.

## 2 Make the Vacuum Seal and Apparatus Setup

Using Indium (99.99% 0.100" dia.) and wrap around the column where the tube lid sits. Make sure it goes all the way around with a slight overlap to prevent leakage and cut accordingly. Note that the overlap as seen in the image is placed where it does not obstruct the screws upon being flattened. Four screws with allen heads should be screwed in from the bottom. Tighten screws in a star pattern, tightening one and then the one across from it a little bit at a time to press the seal evenly.

Use the allen screw drivers to tighten the screws as much as possible and see that the In seal is flattened. There are two ways to set this up so as to facilitate easy removal of this lid at the end of this procedure after forming solid Ammonia:



Figure 1: In Seal After Screw Pressing

- Put two additional screws on the opposing side to screw push against the lid while loosening.
- Simply use a screwdriver to wedge open the lid after all the screws are taken out.

Once the seal has been made place the cylinder inside a plastic cup or any other heat conducting container and stuff it with tissues (Kimtech Science Wipes). This is for keeping the temperature along the length of the cylinder well regulated by providing even thermal contact over the cylinder. Use enough tissue that the metal cylinder is not visible and tight enough that the tissue hold the plastic tube in place. Make sure the metal cylinder is centered. Now connect the top part of the cylinder to the gas system using a clamp.

Note: This picture has liquid nitrogen already poured into the dewar, however, you need not do this until section 4, where it is explained in further detail.

### 3 Test for Leaks and Evacuate Air

Note: All the arrows in pictures denote the direction for increasing pressure or for the opening of valves.



Figure 2: Cylinder Setup and Apparatus Configuration

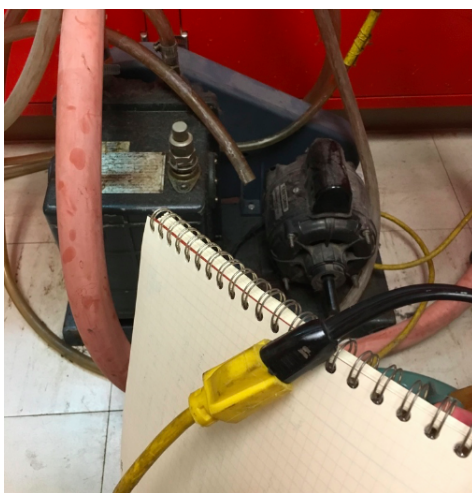


Figure 3: Electric Pump Connector

Turn on the electric pump by connecting the black plug and yellow connector.

In the following sequence, open the two green valves on the vacuum gas system (labeled V and N4) and the pump valve (labeled N2) which will bring

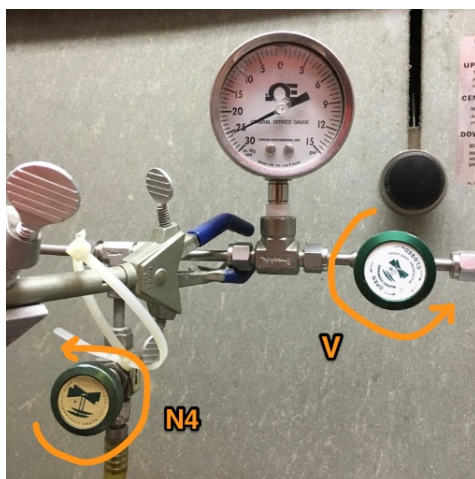


Figure 4: Flow Control Valves

the pressure down to about -29 psi. Close the pump valve (labeled N2) and wait to make sure that the pressure holds. If the pressure doesn't increase after a 2-3 minutes then there is no leak and you may proceed.

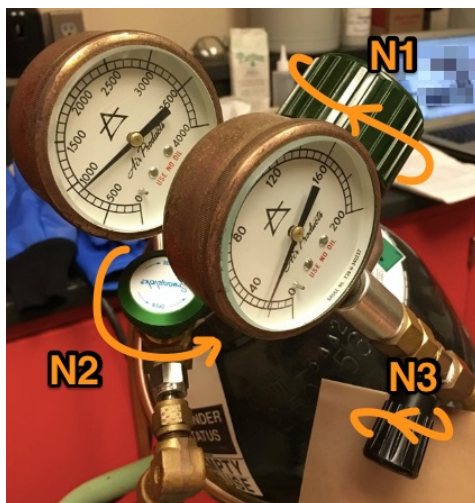


Figure 5: Nitrogen Pressure Controls

After the initial test for leaks fill the cylinder with nitrogen gas, make sure that the pump valve N2 is still closed and open the nitrogen flow valve (labeled N3). The nitrogen flow can best be controlled by first closing the

flow controller (labeled N1) and then opening N3. Then slowly open the flow control counterclockwise until 9 psi is reached on the pressure meter. Once the flow control is set you can just open or close the nitrogen inlet valve, even for future runs. Sometimes the pressure may go very high upon opening N3. To make it drop to 9psi, follow this procedure with 5-10 seconds wait period between each step:

- Close N3
- Open N2
- Close N2
- Open N3 again.

The pressure should drop closer to 9psi. If this does not work accurately, it means that the flow control (N1) setting has been changed and needs to be decreased. Most regulators, including N1, will respond best when the valve (here, N3) is opened completely.

To evacuate the air completely pump out the cylinder contents down to -29 psi by closing N3, and opening N2. Do this 2-3 times, waiting on -29psi and 9psi for 2-3 minutes. On the final iteration pump out for 5-7 minutes.

## 4 Freeze the Ammonia

You can now turn the pump off by simply decoupling the black plug and yellow connector. Fill the dewar under the cylinder with liquid nitrogen up till the lip of the container around the cylinder to create a cooling bath for the cylinder and the ammonia gas that is going to be flowed into it. Ensure that very little liquid nitrogen gets inside this container with the tissue. Make sure the pump valve (N4) is closed and the valve to the cylinder (V) is open to let ammonia gas into the tube.

First close the flow controller (A2), then open the bottle (A1) and open the ammonia inlet valve (A3). Then slowly open the flow control (A2) counterclockwise until 6 psi is reached. In future runs, the pressure may go straight to 6psi just by opening the valves as long as the regulator (A2) hasn't been changed. Keep the valves open and the pressure around 6 psi as the system continues to cool. Keep the liquid nitrogen level at the maximum level at all times and regulate the pressure so that it stays at 6psi. You will

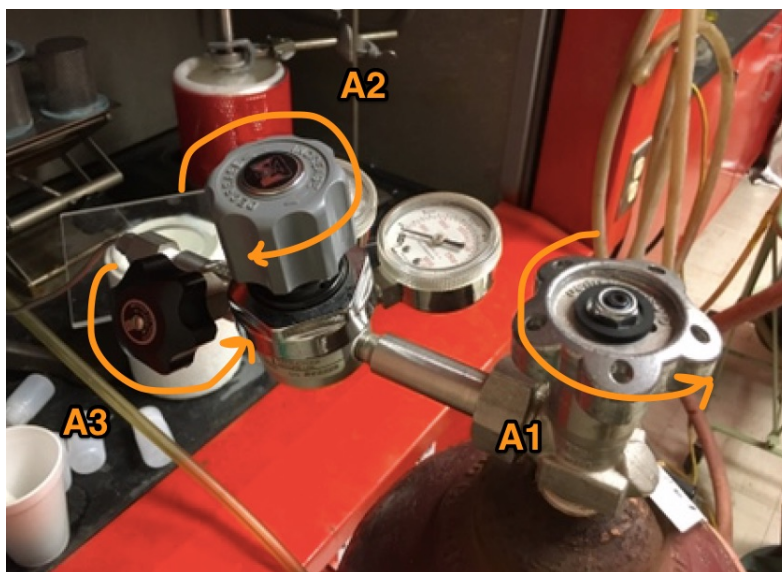


Figure 6: Ammonia Pressure Controls

need to pay attention to these two things every 2-3 minutes. Keep the ammonia open for about 30 minutes after which close the ammonia inlet (A3) and wait for the pressure to drop down to -29 psi while keeping the liquid nitrogen level at the maximum level at all times. The pressure may not drop all the way down to -29psi, in which case observe the lowest point it reaches (-27 to -28psi) and let it stay there for 10 minutes.

In this period prepare the 3 screen meshes and pestle by cooling them in a styrofoam containers with liquid nitrogen. Label the two nalgene bottles with the name ("NH3 Fresh" or "NH3 Scraps") and the date.

## 5 Remove Frozen Ammonia

At this point, make sure that you are wearing the safety goggles and that the Cryo-Gloves are at least on hand if you are not wearing them at some point due to restriction of motor skills.

Close all the valves and open the line at the cylinder. Take the cylinder out of the bath and the container with the tissue in order to unscrew the four screws on the bottom. Loosen the lid a little at a time to push the lid off evenly. Once open and warm enough the ammonia sample will slide

out into the screen mesh. If not, periodically hammer it from the top of the tube with a long screwdriver, however, don't do this too often as the warm screwdriver head will cause parts of it to vaporize. Keep the cylinder over the mesh and liquid nitrogen because there will be small droplets of liquid ammonia melting away from the solid. Keep it submerged in liquid nitrogen and use the pestle to crush the material through the first screen. Cool down the funnel and the bottle using the remaining liquid nitrogen in the dewar. You are to put the material collected in the second mesh in a bottle as good material, and the material collected in the third mesh in separate bottle as scraps. Connect the "NH3 Fresh" bottle to the funnel lid first. Shake the mesh to get the finer pieces to drop through the funnel and into this bottle. Dip it in liquid nitrogen once in a while to get it all to drop into the bottle. Put a lid on this bottle and drop it into the dewar. Repeat this for "NH3 scraps". Take the bottles to lab 28 and store them in storage dewars that have slots available. You can see which dewar has slots based on the PTG Inventory google spreadsheet.