SPTG-TechNote-17002:
Making Solid Ammonia

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1 Introduction

This document describes the process of making of solid Ammonia (both NH_3 and ND_3). This Ammonia will be used by the Solid Polarized Target Group at the University of Virginia in its projects and experiments.

Be aware that there will be exposure to Ammonia and liquid Nitrogen during this procedure; be sure to work in a well ventilated area; avoid contact with liquid Nitrogen and liquid/solid Ammonia; do not inhale gaseous Ammonia; Avoid eye contact.

Disclaimer: The Polarized Target Group at the University of Virginia will not be held responsible from any consequences arising from the use of this document.

2 Preparation

2.0.1 Clean Components

To begin, thoroughly clean the tube rims and lids of the cryostat in which the Ammonia will be made. Also clean any utilities that will come in direct contact with the Ammonia such as the screen mesh containers and the pestle. The indium used to make the seal should also be wiped down with alcohol.

2.0.2 Obtain Liquid Nitrogen

Fill up a large dewar with liquid Nitrogen for use throughout this procedure. Approximately 10 Liters are needed for each iteration of this procedure.

2.0.3 Obtain Bottles

Obtain three 30ml Nalgene bottles with small holes in which to cold store the Ammonia made. Although only two bottles are needed, it is prudent to have an extra bottle in case there is a larger than anticipated yield.

If needed, these bottles can be made by drilling very small holes (< 0.070 inches ϕ) into the bottoms and caps of standard 30 ml Nalgene bottles.

Figure 1: Pestle

Figure 2: Meshes

2.0.4 Create the Seal

Take a strand of Indium wire (99.99%, 0.100" ϕ.) and wrap it around the inner column where the tube lid will sit. Leave a slight overlap between the ends of the wire to prevent leakage, and making sure that the overlap will not obstruct the bolts that will hold the lid and cryostat together.

Figure 3: Inner Column with Indium Seal

Figure 4: Cryostat with Lid Attached.

The lid will be attached to the cryostat by means of four Allen-head bolts, which will be bolted in from the bottom. To ensure an even seal, the bolts should be tightened in a star pattern. Note that this process will flatten the Indium, which is natural and should happen.

In order to facilitate easy removal of the lid at the end of this procedure, put an additional bolt on the back of the opposing flange of the cryostat.

2.0.5 Prepare and Connect Cryostat

Once the seal has been made place the cryostat inside a plastic cup (or any other heat conducting container) and stuff it with tissues (Kimtech ScienceWipes). Make sure to

put a tissue in the bottom of the cup before inserting the cryostat. The tissues will help evenly regulate the temperature along the length of the cryostat within the cup. Center the cryostat in the cup and put enough tissues around the cryostat such that the cup is held in place. Connect the top part of the cryostat to the gas system using a KF-40 clamp and o-ring and, with the help of a stand/clamp, set the cryostat/cup into an empty dewar. Position the cryostat/cup so that it is near the top of the dewar.

Figure 5: Cryostat and Cup

Figure 6: Cryostat/Cup Inside Dewar.

2.0.6 Check for Leaks

Once the cryostat is connected, one must check the system for leaks. To begin with, make sure the bottle containing the Ammonia is closed. Then, open the regulator and all valves except for valve N (see Figure 7). Turn on the electric pump, which will evacuate the system and bring the pressure inside to about -29 psi. Close the valve S2. If the pressure does not increase after about 5 minutes then there is no leak and one may proceed. If the pressure does increase, then there is a leak which must be found and fixed before any Ammonia can be made.

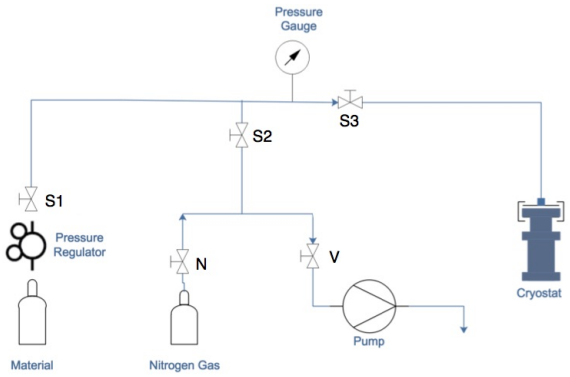


Figure 7: Diagram of System

2.0.7 Purge System

After the initial test for leaks, open valve S2 and close valve V. Open valve N and allow Nitrogen gas to flow into the system until 9 psi is reached. One might have to adjust the regulator of the Nitrogen gas tank (not shown) in order for this to happen. Next close valve N and open valve V, again evacuating to system to about -29 psi. Alternate between filling the system with Nitrogen gas and evacuating it two to three times. On the final iteration pump out for 5-7 minutes before closing valve S2.

Once the system is ready, the pump can be turned off and the Nitrogen gas tank closed.

3 Making the Solid Ammonia

One can now begin making solid Ammonia. Close the regulator and open the bottle containing Ammonia gas. Adjust the regulator until the system is pressurized to approximately 6 psi. At this time the dewar can be filled with liquid Nitrogen. Pour as much liquid Nitrogen as possible without letting it spill into the cup.

For the next 30 minutes, keep the liquid Nitrogen level at its maximum and regulate the pressure so that the system stays at 6psi.

After 30 minutes have elapsed, close valve S1 and the bottle of Ammonia gas. Allow the system to depressurize itself to about -29 psi. During this time continue to maintain the liquid Nitrogen level at maximum. During this period, prepare the 3 screen meshes and pestle by cooling them in liquid nitrogen. Label the Nalgene bottles with chemical formula of the Ammonia and the date.

Once the pressure drops to about -29 psi, wait about 5-10 minutes before collecting the solid Ammonia.

Place the three meshes inside of each other and place them in another dewar, using a specially-made piece of plastic to hold them in place. Fill this dewar with liquid nitrogen.

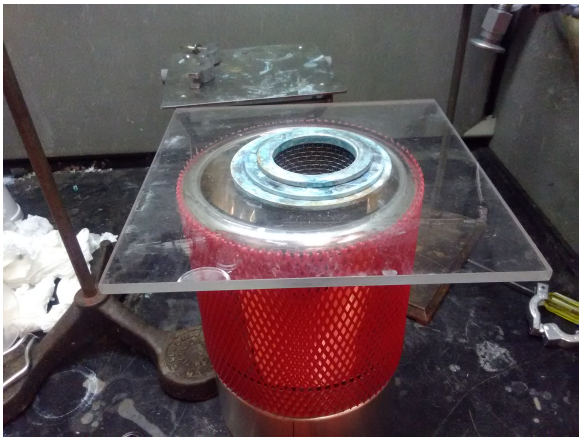


Figure 8: Meshes Inside Dewar

4 Collecting and Storing the Solid Ammonia

Disconnect the cryostat from the system and remove it from the cup in order to remove the lid. Use the bolt on the opposing flange to push the lid away from the cryostat. Once the gap between the lid and the cryostat is wide enough, a screwdriver can be used to pry to lid off.

Keep the cryostat over the meshes to collect the small droplets of liquid Ammonia melting away from the solid. The solid Ammonia will slide out into the screen mesh once it warms up enough.

Once the Ammonia drops out of the cryostat, keep it submerged in liquid nitrogen and crush it through the first mesh with the pestle. Cool down the funnel and the bottles. Attach the funnel to a bottle and submerge the bottle in liquid Nitrogen. Pour the material collected by the second and third meshes down the funnel and into the bottle. During this process, remember to limit exposure of the solid Ammonia beads to the air as it will begin to evaporate. Once the bottle is full, replace with funnel with a cap.

Figure 9: Bottle with Funnel Attached

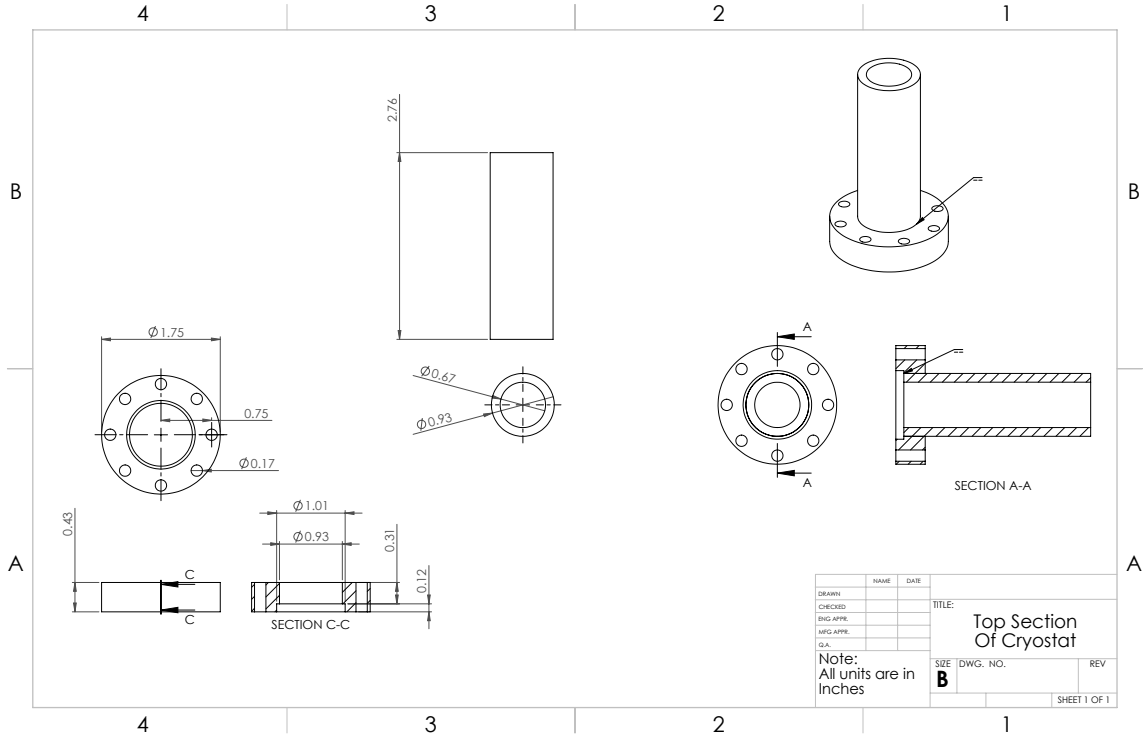
Note that sometimes its is necessary to keep the solid Ammonia collected by the second and third meshes separately. This is because the meshes contain differently-sized Ammonia particles.

Once the material is in bottles, take the bottles to lab 28 and put them in the group's storage dewars. Check which dewars have open slots in the group's inventory spreadsheet. Update the spreadsheet with the location of the new bottles of solid Ammonia.

Continue for cryostat drawings...

Drawings of Cryostat

This section provides detailed drawings of the parts that make up the cryostat.



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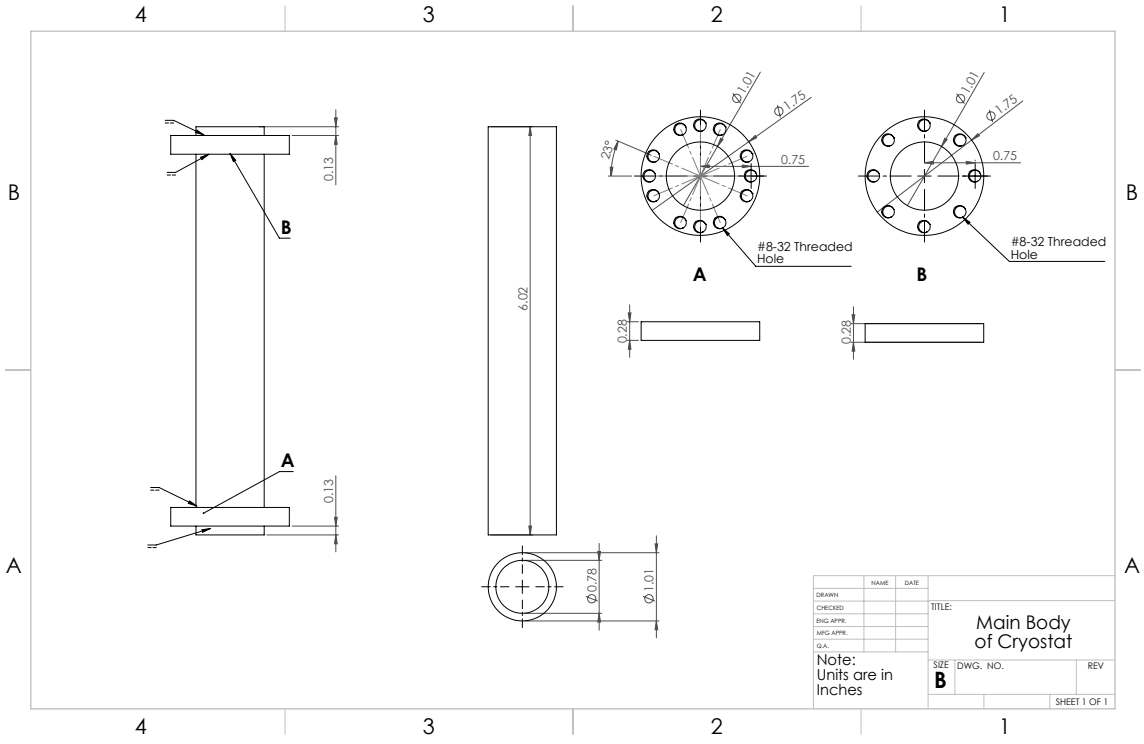

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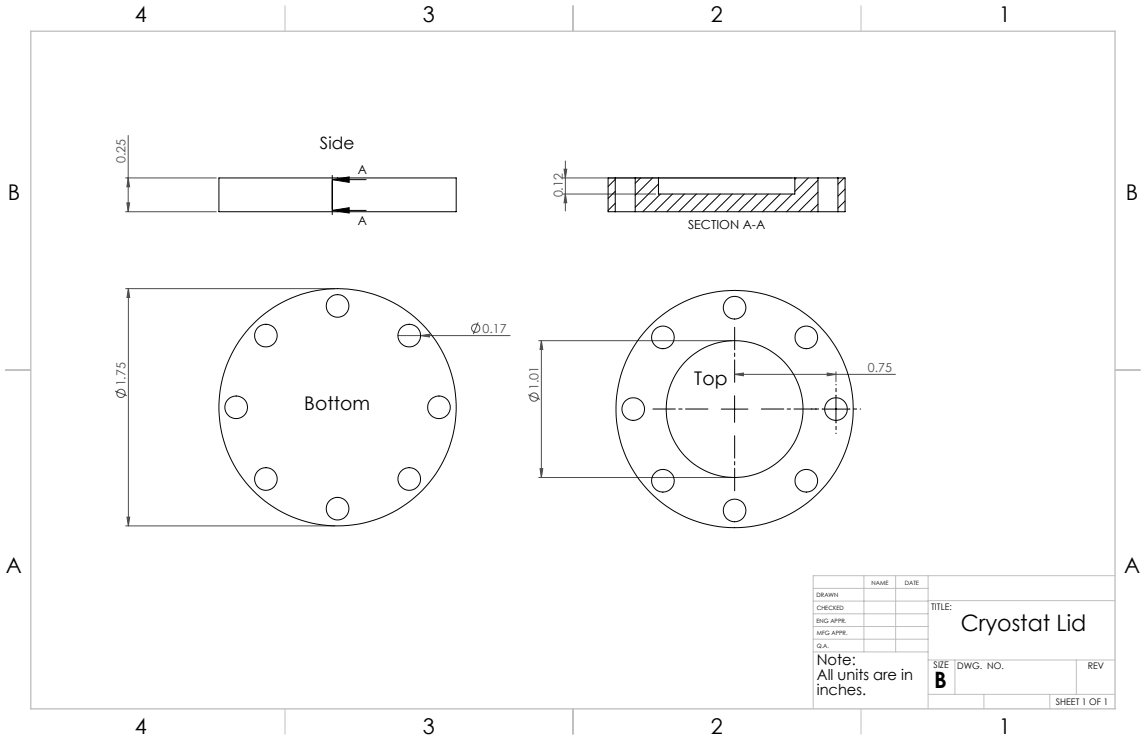

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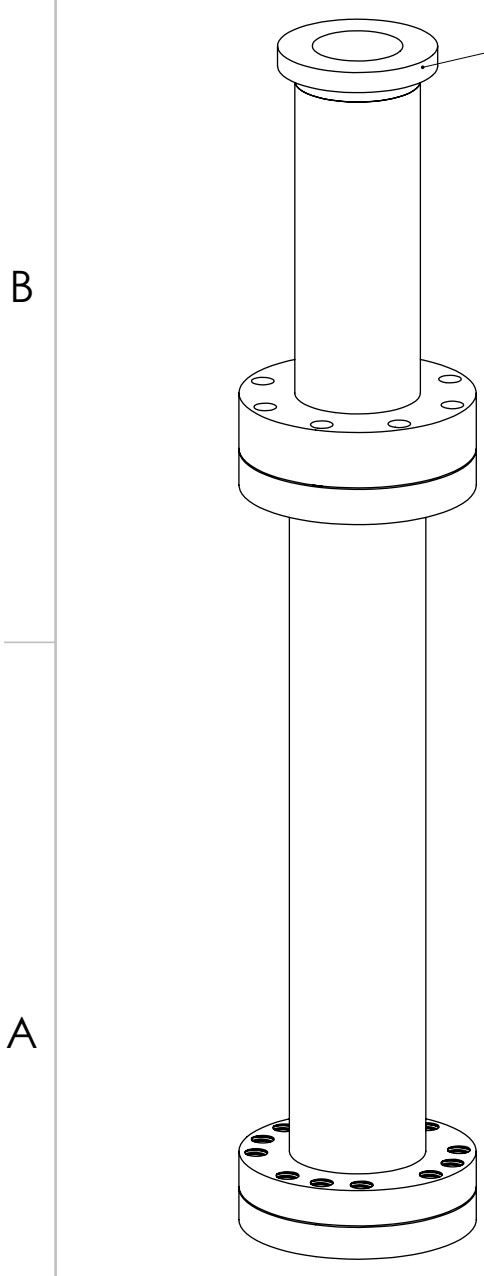

Note:
The different pieces of the cryostat are held together by #8-32 bolts.

	NAME	DATE
DRAWN		
CHECKED		
ENG APPR.		
MFG APPR.		

Cryostat
(Full Assembly)

SIZE	DWG. NO.	REV.
A		
SCALE:1:2	WEIGHT:	SHEET 1 OF 1