



The Triple Point of Nitrogen Protocol: *Preparer's Version*

Introduction

All elements and compounds can exist in any one of various states of matter depending upon the conditions they're subjected to. Phase diagrams are graphical representations of the stability and predominance of each phase of matter as a function of temperature and pressure. Each line of the diagram represents a particular phase transition – but the point at which all lines converge into a single point between the three states of matter is known as the *triple point*. At the specific temperature and pressure of the triple point, the solid, liquid, and gas form of a given substance – and all phase transitions between these forms – are in equilibrium.

Safety Hazards

- Personal Protective Equipment:
 - Cryogen gloves
 - Safety glasses/goggles
 - Chemical & flame retardant lab coat
- Physical Hazards
 - Contains refrigerated gas; may cause frostbite or cryogenic burns
 - Gas under pressure; may cause injury
- Chemical Hazards
 - May displace oxygen and cause rapid asphyxiation

Safety Data Sheet(s)

- [Nitrogen, refrigerated liquid \(Airgas\)](#)

Materials

- Small dewar of liquid nitrogen (4 L dewar with narrow spout recommended)
- Vacuum pump with rubber stopper
- Glass vacuum tube
- Cryogenic gloves
- Ring stand
- Three prong extension clamp

Set Up Reference Photos





Procedure

1. Tightly attach the three prong extension clamp to the upper third of the ring stand. The height of the tube is dependent on preference; 5" to 7" from the top is recommended.
2. Secure the glass vacuum tube to the ring stand by tightening the three prong extension clamp. Be sure the tube is level and straight.
3. Use the small dewar to carefully pour approximately 150 mL of liquid nitrogen into the glass vacuum tube. The tube should be filled roughly 1 ½" to 2" from the top of the tube.
 - a. *Note: Go slowly! The liquid nitrogen will evaporate readily as it is poured out of the cold dewar and into the room temperature glass tube. Pouring in small intervals will prevent splashing and ensure you can maximize the volume of nitrogen in your tube as it progressively cools. You'll notice the nitrogen looks like it's jumping – then settles. Wait until it settles before pouring more until you've reached your desired volume.*
4. Once the liquid nitrogen has settled in the tube (no longer appears to be jumping/splashing), turn on the vacuum pump. The vacuum pump should have a rubber stopper secured to its end that will fit the vacuum tube. Allow the pump to run for approximately 30 – 45 seconds.
5. Gently place the rubber stopper into the top of the vacuum tube. Hold the tube securely in one hand, and press down on the rubber stopper with the other. Hold the stopper in place for a moment to ensure a seal is formed before releasing the tube and stopper.
6. Allow the vacuum pump to run until the nitrogen reaches its triple point!
 - a. *Note: This demonstration will take several minutes to complete. Gradually, the level of the liquid will decrease and solid nitrogen will form and melt periodically. The solid nitrogen is the most visually apparent part of the demonstration, but the liquid level changing as gaseous nitrogen is produced and then condensed (and sublimed and deposited) is also worth noting.*

Tips & Tricks

- For transport safety, place the vacuum tube in an extra cryogenic glove. This will not only cushion it during transport, but serve as a handy holder for the tube when it is still extremely cold after the demonstration has run its course!

Clean-Up Procedures

1. Turn off the vacuum pump and allow the nitrogen in the tube to settle.
2. After the remaining liquid nitrogen has settled, gently ease the stopper out of the vacuum tube. It is best practice to hold the tube with your other hand to prevent tipping the tube and splashing.
3. Liquid nitrogen can be poured out onto the floor (so long as the area is clear of any belongings or people) to instantly evaporate. If there is no clear area to do this, pour the liquid nitrogen back into the small dewar used for transport.
4. The tube will be very cold! Be careful. Allow it to warm back to room temperature before cleaning to prevent any weakening of the glass.
5. Rinse thoroughly with deionized (DI) water and place to dry.