

## Freeze-Dryer Protocol

**Warning:** Liquid nitrogen is potentially dangerous, both due to the possibility of asphyxiation in an enclosed space and the possibility of skin exposure. Information regarding the safe handling of liquid nitrogen can be found [here](#).

### *Materials required:*

Liquid Nitrogen Tank (Airgas, CAT # NI NF10LTC)  
Protective Freezer Gloves

### *Set up for use:*

- Attach freeze dryer metal hose to liquid Nitrogen 230L tank.
- Attach white hose to Nitrogen gas cylinder, which will open valve to primary pump.
- Close green valve
- Aluminum foil to place bell jar on table while loading/unloading sample.
- Switch on right side of freeze dryer should be down, for Normal Cooling Cycle.
- Fill Styrofoam box with liquid Nitrogen, about 1 ½ inch from bottom.
- Separate top and bottom of gold plate and immerse in liquid Nitrogen with forceps.
- When bubbling has stopped, samples can be quickly transferred to slots in gold plate.
- Put gold top plate over samples using forceps.

### *Instructions for use:*

1. Set cooling time to 9999 (equivalent to 999.9 minutes) by pushing small black buttons located below the numbers.
2. Plug in freeze dryer
3. Plug in Varian senTorr Gauge Controller
4. Turn Main Power switch ON, for freeze dryer.
5. Set temperature points: by moving the READ button either left or right to adjust and check the temperature setting.

Cooling	-112 ° F(Set I)
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6. Change cooling time minutes to 0, then push Cycle Reset Button.

Heating	68 ° F(Set I)
Heating	95° F(Set II)
7. Change cooling time minutes back to 9999.
8. Turn on Nitrogen gas.
  - Press Start/Stop/Reset button on white box, to start pumps
9. Once vacuum reaches 0, turn on Gauge Controller by pushing the Emis button.
10. Turn on liquid Nitrogen
11. Press Cycle Reset Button on the cooling cycle to start
12. After the stage reaches -112 ° F (approximately 2 minutes).
13. Turn off gauge controller by pushing the Emis button.

- Turn pump off by pushing the Start/Stop/Reset button on the white box.
14. Release the vacuum in the bell jar by opening green valve.
  15. Hug bell jar and twist off carefully. Place on aluminum foil.
  16. Introduce samples quickly, make sure to wear freeze protective clothing, copper plate **will** freeze skin on contact.
  17. Place bell jar over sample and close green valve.
    - Start up pump on freeze dryer by pushing Start/Stop/Reset button
  18. Push Cycle Reset Button to begin cooling cycle (make sure Liquid nitrogen valve is open).
  19. After cooling cycle and heating cycle have completed and sample is back to or just above room temperature
  20. Turn off liquid nitrogen.
  21. Turn off EMIS on the pressure gauge controller
  22. Vent bell jar by opening green valve, carefully remove bell jar, and remove samples.
  23. Turn off Nitrogen gas.
  24. Turn off main power switch
  25. Store samples in vacuum oven at  $-30$  Hg in. at  $50^{\circ}\text{C}$  until they are ready for loading into sample holder and MIMS.
  26. Unplug the freeze dryer & gauge controller.