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1. INTRODUCTION

This procedure provides guidance for filling the JEOL NMR in the ISU Chemistry Department. The NMR uses a large superconductor magnet that is cooled by liquid helium housed in an interior dewar which is immersed in liquid nitrogen. The liquid nitrogen must be filled regularly (weekly) to maintain proper operation and prevent expensive damage to the magnet.

2. PRECAUTIONS AND LIMITATIONS

- 2.1. Review CSP-0004 Cryogenics prior to using this procedure.
- 2.2. Keep all metal away from the NMR.
- 2.3. Remove all electronic devices (cellphone) and data cards (Bengal card, credit/debit cards) before approaching the NMR.
- 2.4. The area must be kept well ventilated (door open) while dispensing.
- 2.5. Move cryogen tanks with an appropriate cryogenic tank cart.
- 2.6. Wear all appropriate personal protective equipment (safety glasses/lab coat/etc.).
- 2.7. This procedure does not provide guidance for the refilling of liquid helium.

3. APPARATUS AND MATERIALS

- 3.1. Insulated gloves
- 3.2. Safety glasses/goggles
- 3.3. Cryogenic dewar cart
- 3.4. Appropriate transfer line (Tygon tubing)

4. REAGENTS

- 4.1. None

5. INSTRUCTIONS

- 5.1. Open computer program to view nitrogen fill monitor.
 - 5.1.1. Double click on the "Delta" icon.
 - 5.1.2. Click on the "magnet" icon in upper right.
 - 5.1.3. Double click on "Eclipse2-FREE-ECX 300" in the "Spectrometer Control" screen.
 - 5.1.4. Click the "sample" tab on bottom left of "spectrometer control" screen.

NOTE: Percent Nitrogen monitor is in the upper right of the "sample" screen.

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5.2. Position the liquid nitrogen tank.

5.2.1. Using the liquid nitrogen tank labeled NMR in room 152C:

- 5.2.1.1. Set the tank next to but not crossing the yellow tape line on the floor near the NMR.
- 5.2.1.2. Rotate tank so liquid valve faces the NMR.
- 5.2.1.3. Attach Tygon tubing to the “liquid” nitrogen valve on the tank, if necessary.

5.3. Filling the NMR.

- 5.3.1. Remove the small line cap on the top left of the NMR to expose the filling tube.
- 5.3.2. Remove the heat sink cap on the top right of the NMR to expose the nitrogen vent.
- 5.3.3. Vent the liquid nitrogen tank by opening the liquid valve slightly to allow some gas to escape.

Note: *Venting prior to filling is necessary to prevent other gasses from entering the NMR in excessive amounts.*
- 5.3.4. Attach the Tygon tubing to the inlet tube on the NMR with valve slightly open.
- 5.3.5. Open the valve ½ turn more.
 - 5.3.5.1. Gaseous nitrogen should be exiting the vent tube on the right of the NMR.
- 5.3.6. Lift the Tygon tubing between the tank and the NMR to prevent kinks in the Tygon.
- 5.3.7. While lifting the tubing open the liquid valve slowly on the liquid nitrogen tank.

NOTE: *Do not open the valve too much until the tube is completely solid or the tubing will pop off of the NMR.*

5.3.8. When the Tygon tube is solid open the tank valve for a steady flow of liquid nitrogen to the NMR.

5.3.9. Monitor the percent fill of the NMR on the computer until the NMR is 100% full.

5.4. Finishing

5.4.1. When the NMR is 100% full close the liquid nitrogen tank valve.

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- 5.4.2. Allow the Tygon tubing to thaw for 15-20 minutes.
- 5.4.3. Remove the thawed Tygon from the NMR.
- 5.4.4. Replace the inlet cap on the NMR.
- 5.4.5. Check the O-rings on the inside of the heatsink cap and replace if necessary.
 - 5.4.5.1. Replace the heat sink on the NMR.
- 5.4.6. Close the program windows on the computer.