<u>Attachment 4:</u> Cooldown procedure of LakeShore Model HFTTP4

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Center for Functional Nanomaterials Brookhaven National Laboratory	**NUMBER** **REVISION** **2**
Subject: Cooldown procedure of LakeShore Model HFTTP4 probe station using 60 liter liquid helium dewar or 50 liter liquid nitrogen dewar	**DATE** 12/15/2010 **PAGE 1 OF 8**
Approved CFN ESH Coordinator:	**DATE**

<u>Cryogen transport</u>

Take a dewar from the CFN loading dock and carefully roll it to 1L-35 by pulling only. Pushing of storage dewars is forbidden.

<u>Cryogen transfer</u>

Prerequisite: Trained in BNL Cryogen Safety (HP-OSH-025), ODH (TQ-ODH), and use of proper personal protective equipment (PPE): Safety Glasses with side shields or goggles, face shield, cryogen gloves, long cuff less pants, and non absorbent closed shoes. PI will provide equipment specific training.

<u>Emergency Procedures and First Aid</u>

<u>Burns:</u> In severe cases summon medical attention (call x2222) immediately. Flush affected areas of the skin with copious quantities of tepid water. However, a large burn when warmed up will cause the victim much distress and the victim will require powerful pain relief. If there is likely to be a delay in transport to the hospital, then allow the burn to warm up more slowly.

Loosen any clothing that may restrict the circulation of the blood. Do not apply heat to the affected parts, but keep the victim warm. Cryogenic burns will swell and are very prone to infection. Protect the burned area from infection or further injury.

Large Spills of Liquid Nitrogen or Liquid Helium:

Do not enter the 'fog' caused by large liquid nitrogen or helium spills, the oxygen concentration in the fog may be well below that of normal air and hazardous.

For a large spill inside the room, the area must be evacuated. Move away from the danger area, and then call for help (x2222).

Asphyxiation: Never attempt to rescue an injured victim who is in an oxygen deficient environment, you may become the second victim. Call 2222 and wait for the fire/rescue group to arrive.

NOTE.- The procedure below is for **liquid helium transfer**. The procedure is the same for **liquid nitrogen transfer**, except that no transfer line is inserted into the LN_2 dewar. For LN_2 cooling, liquid is extracted from the liquid port using an a appropriate transfer line.

Regular procedures

After evacuating the chamber to below 10^{-3} Torr, use the following procedure to cool the sample and magnet stages. This procedure assumes the probe station and transfer line are at room temperature.

1. Operators should visually inspect the room for any sign of gross gas leaking from tanks and gas lines.

2. Roll the dewar into. Orientate dewar high pressure relief valve (see Fig. 1) away from where people will be standing.

3. Measure helium level in the storage dewar before and after transfer and record He consumption in Helium transfer log book.

4. Use the Z-axis micrometers to raise all probes up 3-4 mm above the sample. Failure to do so will potentially cause damage to the probe tip or scratch the sample surface.

5. Open up the needle valve located underneath the probe station two turns from its closed position (see Fig. 2). This will allow the sample stage temperature to remain higher than the magnet temperature during cooldown to avoid condensation on the sample.

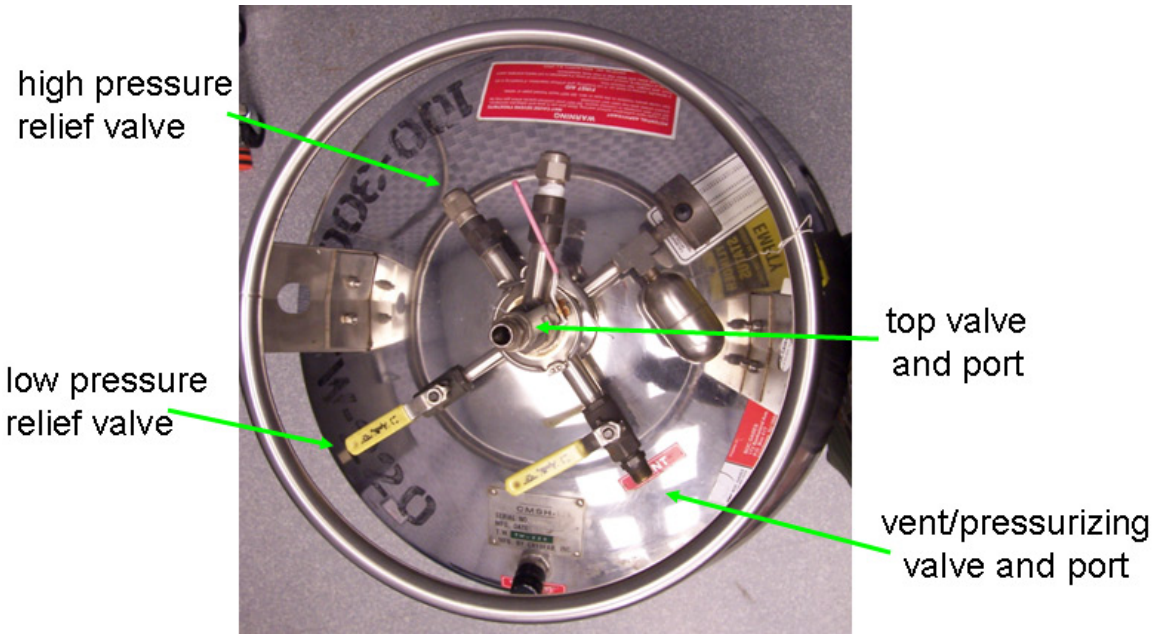

FIG. 1. Top view of liquid helium dewar.

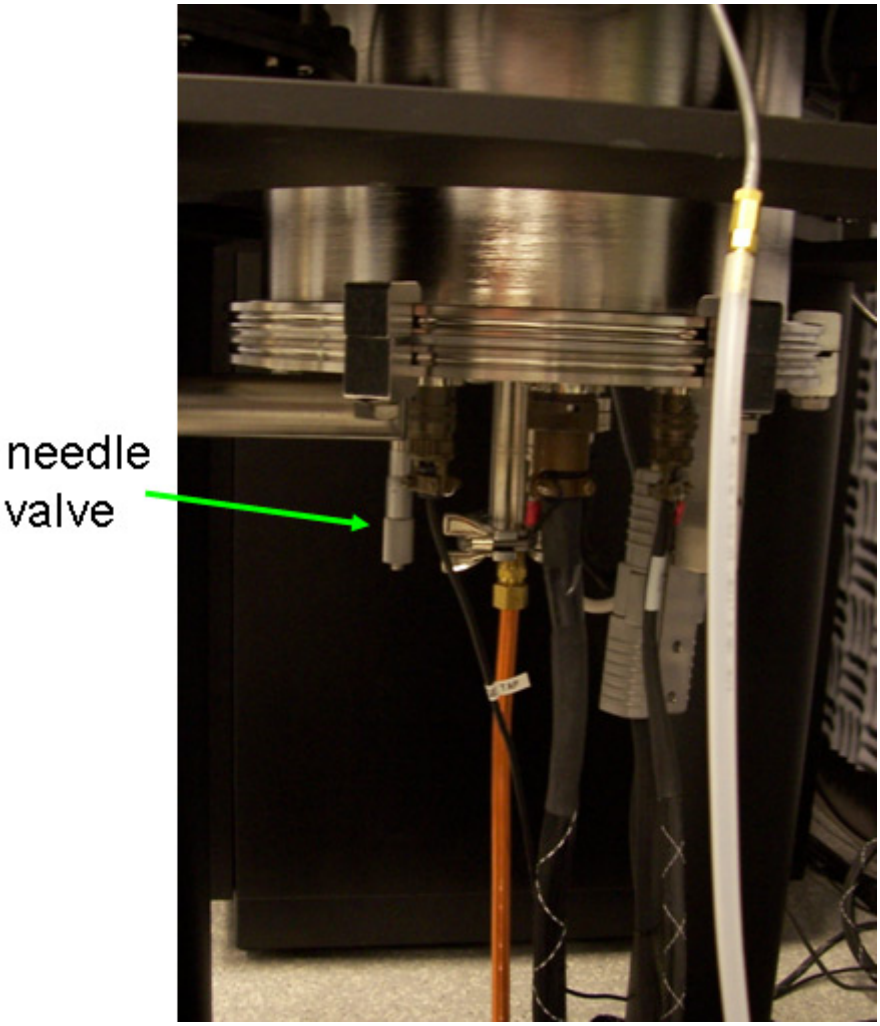

FIG. 2. Bottom of probe station vacuum chamber showing position of needle valve.

6. Open the exhaust valve completely (see Fig. 3).

7. Connect the He gas line to the vent/pressurizing port of the LHe dewar (see Fig. 1). Make sure that the Quick-Connect adapter has been correctly attached. The manometer of the Helios gas line should read below 5 kPa.

8. Prepare the transfer line by placing the compression fitting onto the transfer line leg that goes into the liquid helium dewar, see Fig. 4(a). Push this fitting down to approximately 1 foot above the open end of the leg.

9. Open the foot valve (see Fig. 4) on the transfer line 4 turns. Turn the knob clockwise until it stops, closing it completely, then turn it counterclockwise counting 4 turns open.

10. Remove the plug in the dewar top port. Slowly open the top ball valve to relieve the pressure in the dewar (see Fig. 1).

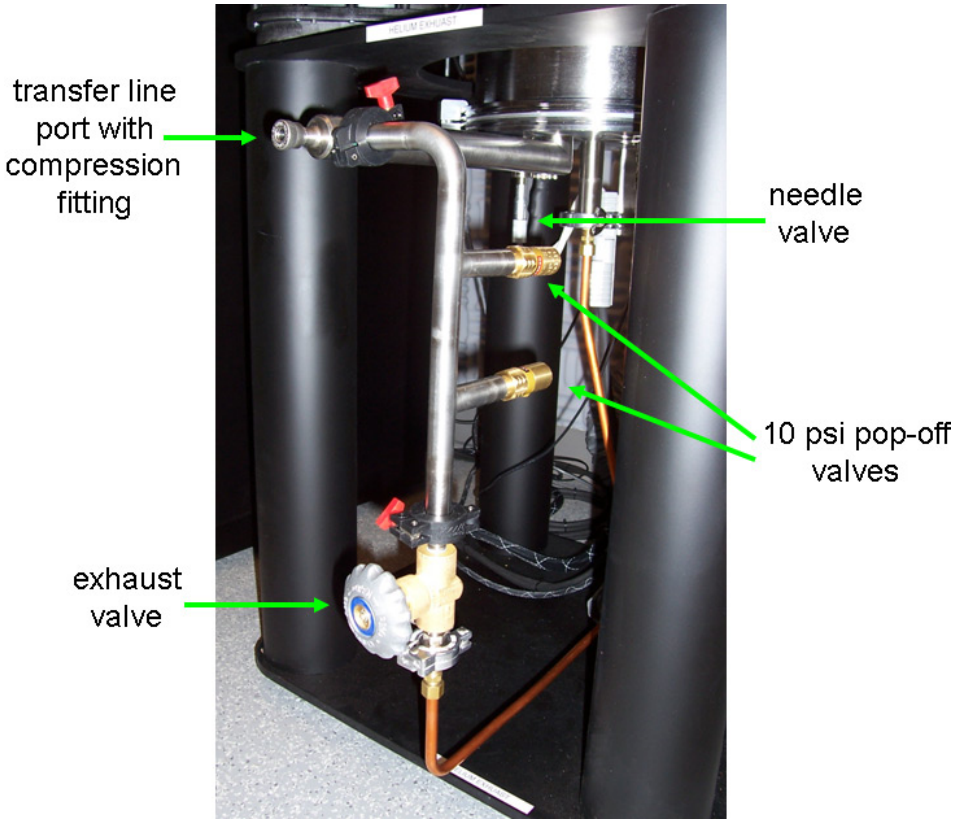

FIG 3. Bottom of chamber showing system valves and ports.

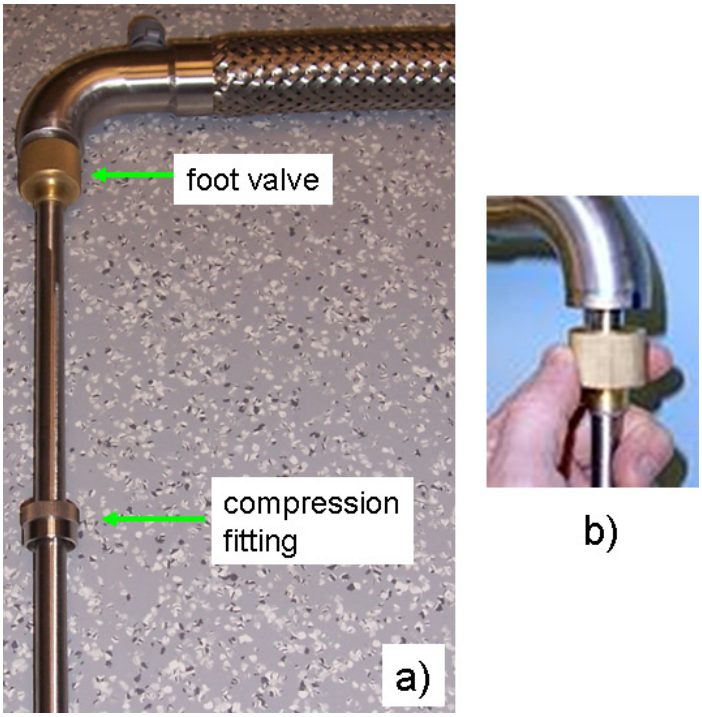

FIG 4. Transfer line detail. a) Foot valve and compression fitting. b) Opening the foot valve.

11. Use long sleeves, cryogenic gloves, and safety glasses with side shields and face shield. Lower the transfer line into the dewar. First insert about 1 foot in and tighten the compression fitting to the dewar top port. Second, release the pressure in the compression fitting O-ring and lower the line until the pressure just starts to rise [see Fig. 5(a)].

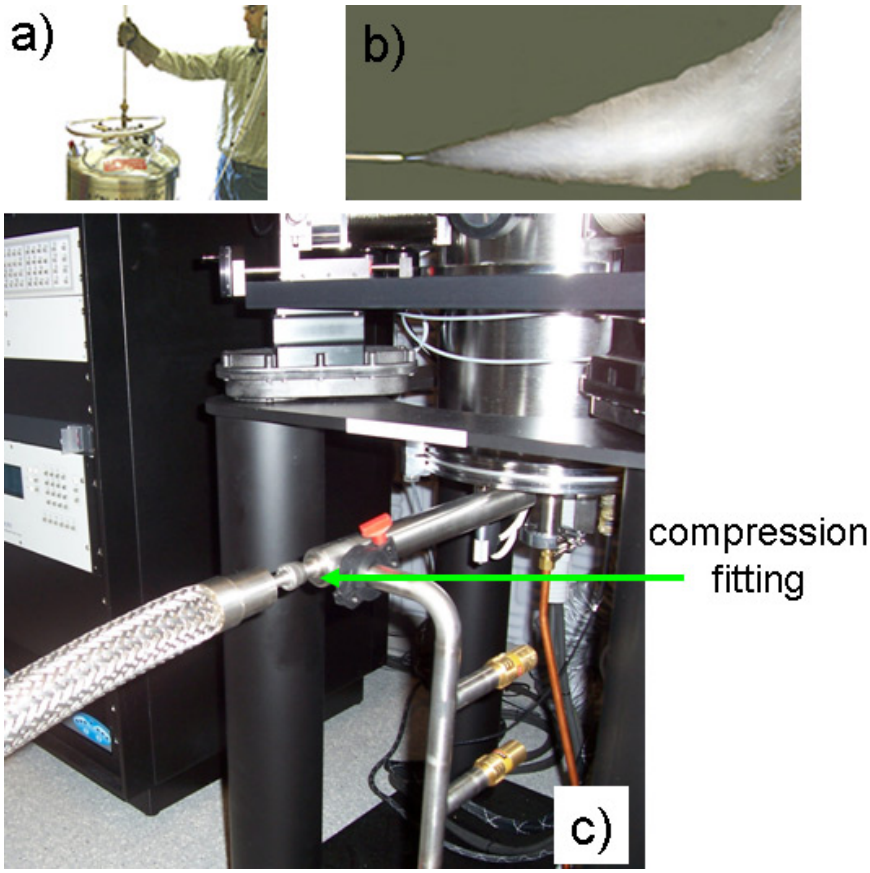

FIG 5. Beginning of transfer. a) Inserting the transfer line into the dewar. b) Indication that the process of precooling the transfer line is complete. c) Inserting the transfer line into port in probe station.

12. Close the low pressure [typically 1 psi (6.89 kPa)] relief valve on the dewar (see Fig. 1). Warning: Do not close or disable the high pressure relief valve (see Fig. 1), otherwise an overpressure hazard could exist. While lowering the transfer line into LHe dewar, monitor the pressure and be sure to keep it below ~16 kPa psi.

13. Continue lowering the transfer line slowly enough so that the dewar pressure does not rise dramatically, until the transfer line leg hits the bottom of the dewar. There is no need to raise the tube slightly from the bottom because the LHe intake is situated on the side of the tube, about 5 mm above its bottom. If the pressure raises above a full turn of the dial, release it by slightly opening the low pressure relief valve (see Fig. 1). Make sure you close this valve once the pressure has lowered.

14. If this is the first LHe transfer (magnet at room temperature), you can insert the other end of the transfer line into the bayonet on the probe station without waiting this part to precool. Otherwise (if the magnet is already cold), you need to wait until you see a continuous plume

of white vapor coming from the free end of the transfer line. Use a clean cloth to wipe any ice crystals from the tip of the transfer line and quickly insert it into the bayonet. Push the transfer line in until it stops, then hand tighten the compression fitting [see Fig. 5(c)].

15. Most helium dewars will not self pressurize at these flow rates so regulated helium gas pressure must be introduced through the vent/pressurizing port on the dewar (see Fig. 1). Try to keep the dewar pressure at ~ 16 kPa.

16. The transfer is started. A schematic drawing of the system configuration is shown in Fig. 6. Start the LabVIEW program (cooldown.vi) that avoids condensation on the sample by (a) keeping the T(sample) 20 K above T(waste shield) if [T(waste)-T(magnet)]>100 or (b) by keeping the T(sample) 100 K above T(magnet) if [T(waste)-T(magnet)]<100. Make sure the sample heater range is set to 50 W.

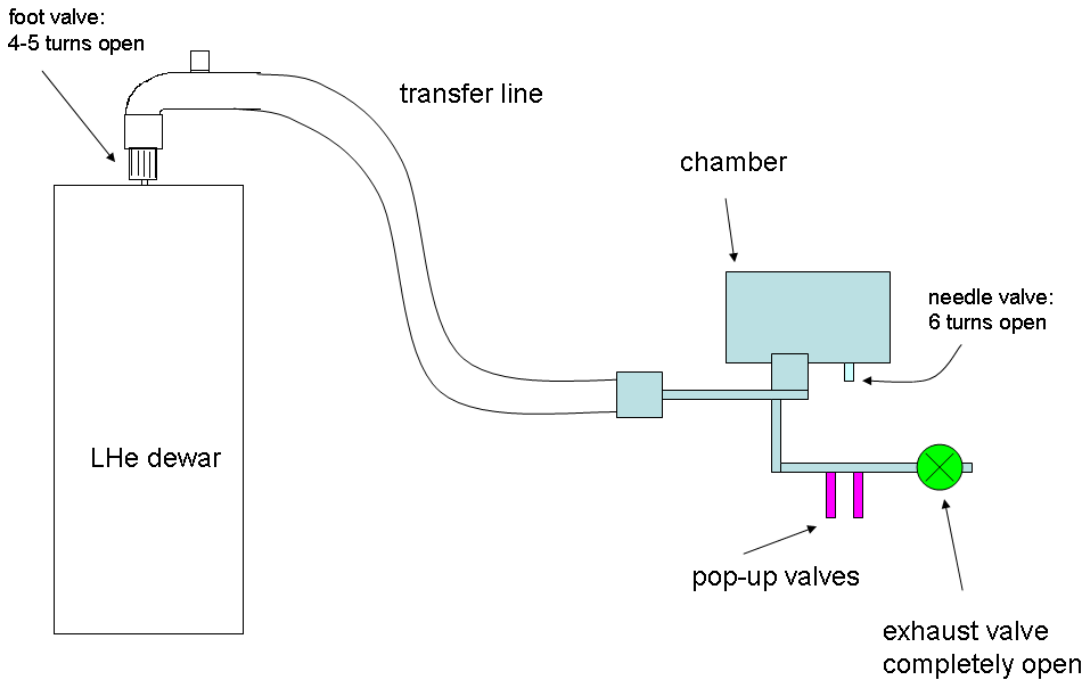

FIG 6. System configuration at the beginning of transfer.

17. Close the main valve to stop pumping, once you are sure that the chamber is cryopumping. You can detect this situation by comparing chamber pressure before and after closing the main valve. If the pressure drops after closing the main valve, then the chamber is cryopumping and it is safe to close the main valve and to turn off the pumping station. Perform this check when the magnet is below 250 K. If the chamber pressures raises once the main valve is closed, repeat the check when the magnet is below 200 K.

18. If T(sample) cannot follow T(magnet)+100K, then it needs more cooling. Open the needle valve in increments of one full and wait ~ 5 minutes to see if is the sample stage has more cooling power that allows T(sample) to reach the right value above T(magnet). After approximately three hours, the probe will cool to its base temperature.

19. When T(magnet)≈10 K, turn off the sample heater. If T(sample) drops too fast, slightly close the needle valve. If it stays above 4 K, then slightly open the needle valve to increase cooling.

20. Allow the refrigerator stages to cool. Normal values are:
    T sample ~ 4.2 K
    T magnet ~ 4.3 K (below 5 K)
    T rad shield ~ 8-10 K (below 15 K)
    T waste shield ~ 12-20 K (below 25 K)

21. Allow about 20 min to let the system (refrigerator and probe arms) to stabilize in temperature.

22. Helium flow and dewar pressure may now be reduced for normal operation. Refer to the following section for more information on operating the probe station once base temperature is reached.

Operating near 4.2 K

The following procedure assumes that the sample and magnet stages have stabilized below 5 K and it is desirable to remain at that temperature while minimizing the consumption of cryogen.

1. Maintain the pressure in the dewar around 14 kPa. Use the 0-5 psi regulator located on He line on the wall behind the pumping station. This reduces the temperature of the cryogen and makes it easier to regulate the flow. Make sure the exhaust valve is completely open.

2. Close the foot valve on the transfer line a quarter turn, wait two minutes to see if the temperature at the magnet stage (Channel B on the Model 340 Temperature Controller) rises.

3. Repeat step 2 until you see the temperature at the magnet stage rise.

4. Open the foot valve approximately 30 degrees at a time and wait one minute to see if the temperature reverses its rise. When the temperature stops rising, turn the foot valve one more quarter turn open.

5. Close the needle valve on the probe station (located underneath the probe station) a quarter turn, wait two minutes to see if the temperature at the sample stage (Channel A on the Model 340 Temperature Controller) has risen.

6. Repeat step 5 until you see the temperature at the sample stage rise.

7. At this point the needle valve can be used to set the sample stage temperature within a range of approximately 1 K from base temperature. It is difficult for the temperature controller to achieve stable control in this range.

Warm up

1. Close the foot valve, close the pressurizing line from the He gas tank end (so you can still use the gauge in the pressurizing line), and slowly open the pressure relief valve in the LHe dewar.

2. Warm up with a heat gun the transfer line fitting in the probe station bayonet until the ice around it is melted. Put on cryogenic gloves and remove the transfer line end from the bayonet.

3. Raise the transfer line above the LHe level and wait until the Dewar pressure drops below 5 kPa.

4. Remove the transfer line. Close the top and the pressurizing line valves on the LHe dewar. Remove the pressurizing line and respective Quick-Connect adapter. Completely open the pressure relief valve.

5. Do not forget to remove from the dewar the transfer line brass or SS fitting for the input port.

6. Start the pumping station and wait until the gauge pumping state gauge reads below the 10^{-5} Torr level. At this point, slowly open the chamber main valve.

7. Warm the sample stage to a setpoint of 310 K. Warm the magnet stage, radiation shield stage, and second shield stage to setpoint of 300 K. Leave the system in this state until all refrigerator stages are at their temperature setpoints and the probe arm is at room temperature. Make sure the sample heater is set to 50 W, the magnet analog output is ~ 100% and that the radiation and waste shields heaters are set to "high" and "on" states, respectively.

8. Once all refrigerator stages are at their temperature setpoints, it is safe to stop pumping. Close the main valve, stop the pumping station and slowly introduce He gas into the chamber until it reaches atmospheric pressure. The chamber can be opened now to remove your sample.

Training and authorization to enter the experiment room

The LakeShore probe station will be operated only by trained users who will have authorization to enter lab 1L-35. Training will be performed by Fernando Camino, Chuck Black, or Chang-Yong Nam. Users will be trained in the general use of probe stations, in basic operation of electronic instrumentation and in the cooldown procedure of the Lake Shore probe station.