7T Superconducting Magnet ^{3}He Insert Manual

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I. Introduction:

This is a user manual to assist in the operation of the ^{3}He insert for the 7T-VF magnet. The insert has a temperature range of 300mK to 300K, however, it is most useful at temperatures less than 50K. To run, the insert must be immersed in liquid helium above the 1K pot inlet (approximately 35% on the level meter). Other than that the only requirements are a rough pump and a temperature controller (found on the associated electronics rack; See Figure 4). While operation of the insert is fairly simple there are multiple modes of operation depending on the temperature range. These different modes can make things difficult, especially when trying to move smoothly from one to another. The following information should help you, but it is not intended to replace the advice of an experienced user. For assistance with this piece of equipment contact Evan Fitzgerald (Rm. A123, x6657) or Dan Dender (Rm. A118, x6225).

II. Sample Loading

a. **Before making any preparations be sure that the sample can/holder will fit into the insert sample space.** Consult the sample mounting webpage for the current dimensions. It is recommended that samples be loaded into cans with an atmosphere of helium gas to conduct heat since the insert relies on conduction of heat through the sample mount below 4K.
b. Once the sample is loaded into a can it can be mounted onto the insert. There are two options for mounts: 1. The original mounting surface of four #4-40 screws on a 1.06" bolt circle. 2. The standard NCNR mount of four #10-32 screws on a 1.75" bolt circle. Note that the standard NCNR mount requires the use of a 1/8" thick adapter, which decreases the available sample space.
c. Once the sample is mounted on the insert the length should be adjusted so the center of the sample is in the center of the beam. The length from the bottom of the insert mounting flange to the beam center is 873mm. Any crystal axes or other information useful for sample alignment should be transferred to the top of the insert above the mounting flange.

III. Insert Preparation

a. The 1K pot line should be checked for proper flow before proceeding, especially if the insert was recently cold. To do this, pressurize the line by connecting helium gas to the 1K pot pumping valve on the top of the insert (See Figure 2). Cover what is normally the inlet, the small hole on the side of insert just above the taper seal, with alcohol and look for a small bubble of gas every few seconds to verify flow. If flow can not be verified contact a sample environment team member before proceeding. If flow is vverified close the pumping valve and disconnect the gas line.
b. Clean the two mating taper surfaces with alcohol and then cover each in a liberal, but uniform, layer of grease. Connect a turbopump to the inserts vacuum valve (a KF 16-25 adapter will be necessary) and open the valve (See Figure 2). Place the vacuum can carefully over the sample and gently

mate the taper seals. Start the pump while holding the vacuum can and slowly rotate the can to spread the grease as the can pulls up on the taper. When the can is snug stop turning it and let it pump out for at least 15 minutes at which time the pressure should be less than $1x10^{-4}$ Torr. At this time close the vacuum valve and remove the turbo pump and KF adapter.

c. Remove the alcohol from around the 1K pot inlet with a small towel if there is any still present and then clean out the 1K pot using the following procedure. Connect a rough pump with a gas inlet to the 1K pot pumping valve and pump/purge the 1K pot with helium gas at least three times before leaving it pressurized with the gas still flowing. **The 1K pot should now remain pressurized with helium gas until the insert is loaded and in an all helium environment.**

IV. Insert Loading

a. If the magnet is above 4K (liquid helium temperature) open the magnet's exhaust valve and begin filling liquid helium through the magnet's fill port. Do not pressurize the storage dewar and move on to step "b" once the exhaust is calm, but steady. If there is already liquid helium in the magnet move on to step "b" without starting a helium fill.

b. If you are filling the liquid helium lower the pressure by exhausting the storage dewar almost completely. If not, open the magnet's exhaust valve. Next unscrew the three nuts holding the clear lid on top of the magnet and remove it right before carefully placing the insert into the hole. Slowly lower the insert into the magnet and mount it onto the three screws. Pay attention to the location of the dark angle in relation to the sample axes, if necessary. With no liquid helium present the insert can be completely lowered in less than a minute. With liquid helium present it will take longer and the vapor will be thick and extremely cold. Low temperature gloves (and safety glasses as with any cryogen fill) are essential in either case. Be careful not to stress the rubber gasket when it is frozen as it will easily fracture.

c. Once the insert is completely loaded secure it in place with the nuts on the three screws. Warm the rubber gasket with a heat gun as necessary before tightening the nuts. Increase the flow of liquid helium back to normal levels or begin a fill when the exhaust has died down to a reasonable level.

d. To efficiently cool the insert, and sample, exchange gas must be added to its vacuum space. To add the smallest amount possible simply purge the open fitting of the insert's vacuum valve with helium gas then quickly cover the fitting with your thumb and briefly open then close the valve. This takes some confidence and a suitably large thumb, but does work well. This will be followed by a sudden increase in helium vapor being exhausted. Continue filling the liquid helium until the magnet is completely full.

V. Cooling to 4K

a. With the insert loaded connect the temperature controller cable labeled "^{3}He Insert" to monitor the sensors. There are three sensors: one on the 1K pot (sensor C, Silicon Diode, 1.4 – 325K), one near the 1K pot on the exchange gas sorb (sensor B, Ruthenium Oxide, 0.3 – 75K) that also functions as the exchange gas sorb heater (Analog Out 1) and one on the ^{3}He pot which is the sample sensor (sensor A, Cernox, 0.05 – 300K). There is also a ^{3}He sorb heater (Analog Out 2) and a sample heater (Heater Out)

b. If there is exchange gas in the sample space all three sensors should be cooling at approximately the same rate, but may be somewhat separated (see section IV-d on how to add exchange gas). To allow the system to cool most efficiently turn on the exchange gas heater by setting Analog Output 1 on the temperature controller to 100% (See Figure 5) and turning the manual switch to "exchange gas heater" (See Figure 4). This will warm the exchange gas sorb and allow the sample to continue to be cooled by the helium reservoir via the exchange gas. Without using the exchange gas heater the sample temperature will cool very slowly below about 30K.

c. When the sample has maintained 4K for a few minutes the exchange gas heater can be turned off by setting Analog Output 1 to 0% (See Figure 5) or turning the manual switch to "temperature sensor" (See Figure 4).

VI. Cooling to Base Temperature

a. The sample temperature should be at 4K before starting this procedure. See section V "Cooling to 4K" if the sample is warmer.

b. Verify that the exchange gas heater is turned off as described in section V-c.

c. Connect a high capacity rough pump to the 1K pot pumping valve on the top of the insert and pump out the line. Open the 1K pot pumping valve and monitor sensors B and C to verify that they quickly drop to 1.4K. If their temperatures do not drop contact a member of the sample environment team. This pot uses an impedance to control the flow of liquid helium into it so **there is no needle valve to adjust.**

d. Note: This system uses a "single shot" method that will sustain base temperature for a maximum of 36 hours before needing to repeat the following steps. This time can be drastically shortened if heat is added to maintain a temperature above base or by various other factors.

e. Once the 1K pot is cold turn on the ^{3}He sorb heater by setting Analog Output 2 to 100% (See Figure 5). This will drive the ^{3}He gas out of the sorb to be cooled by the 1K pot. After ten minutes or so the sample temperature should slowly descend to around 1.4K.

f. When the sample drops below 2K the ^{3}He is condensing and collecting in the ^{3}He pot. The longer you want to stay below 4K the longer you should allow the ^{3}He to condense. One hour of condensing has been shown to give about 24 hours at base temperature and two hours should give the maximum amount of time, 36 hours.

g. After condensing for an appropriate amount of time turn off the ^{3}He sorb heater by setting Analog Output 2 to 0% (See Figure 5). In approximately half an hour the temperature should reach a base temperature around 300mK.

VII. **Temperature Control**

a. T < 1K: When starting from base temperature the sorb can be gently warmed (< 15%) to increase the sample temperature slightly (See Figure 5). Alternately the sample heater can be used at a low power setting (25mW) to control at slightly warmer temperatures, however, this will shorten the time temperatures less than 1.8K can be maintained.

b. 1K < T > 1.8K: The ^{3}He must be condensed and pumped on to work below 1.8K, but it is difficult to make it last for more than a few hours when working above 1K. To work in this temperature range use both the sample heater and the sorb heater. The sorb can be warmed with about 15 – 30% of its heater power to slow the pumping of the ^{3}He and then the sample heater used at a low power to control the temperature of the sample (See Figure 5).

c. 1.8K < T > 4.2K: In this range the cooling from the 1K pot is necessary, but pumping on liquid ^{3}He is not. The sorb heater should be set at 30% or above to provide exchange gas in the ^{3}He circuit. The sample heater can then be used on a low setting (See Figure 5).

d. 4.2K < T > 50K: At temperatures close to 4 K the same method as suggested in "c" should be employed. At higher temperatures the cooling from the 1K pot is not useful to cool the sample, but it us useful in keeping the exchange gas sorb cold. At this point the ^{3}He sorb temperature is not important, so its heater can be turned off. The sample will then be isolated in a vacuum and its temperature entirely dependant on the sample heater.

e. 50K < T > 300K: Everything from the previous step applies here except that at somewhere around 50K is becomes necessary to pump out the exchange gas. The exchange gas sorb will eventually warm up from the heat applied to the sample and start releasing the helium gas. When this happens the sample will be thermally connected to the LHe reservoir, which will burn off a large amount of LHe. To prevent this, close off the 1K pot pumping valve and connect a turbopump to the inserts vacuum valve and pump continuously while at high temperatures (See Figure 5).

f. Cooling from High Temperatures: For temperatures above approximately 50K it is necessary to add exchange gas (if it was pumped out) to cool the sample back down at a reasonable rate. If the exchange gas was not pumped out simply turn on the exchange gas heater by setting the manual switch to "exchange gas heater" and setting analog output 1 to 100%. A setting of 100% will cool the sample as quickly as possible, but a lower setting may burn off a bit less helium and offer a slower more controlled cool down (See Figure 5).

VIII. **Sample/Insert Unloading**

a. If you have any reason to suspect a leak of helium into the insert or the overpressure valve releases during this process immediately connect a rough pump to the insert's vacuum space and contact a sample environment team member. Any liquid helium present will suddenly expand to many times its initial volume and create a dangerous build up of pressure that will release at the weakest joint.

b. Removing the insert is quick and simple. First disconnect the 1K pot pumping line and the sensor cable. Then remove the three nuts holding the insert down and pull the insert out. Be sure to have a pair of cryogenic gloves on and have the insert's cart nearby. After setting the insert on its cart remember to screw it down. Place the clear lid back onto the magnet and screw it down.
c. Use a heat gun to bring the outside of the insert to room temperature. If you have time let it sit for at least an hour to allow the inside to warm as well. If you need to open the insert quickly give it fifteen minutes after warming the outside then move to the next step.
d. Connect helium gas set to 5 psi to the insert's vacuum valve using a hose barb. Hold the vacuum space's overpressure valve closed with a piece of tape. Hold the plastic bin under the insert so that its padding is an inch or two below the bottom of the insert and open the vacuum valve. Slowly increase the pressure until the can pops off into the bin. The pressure necessary to remove the can is normally around 10 psi. Then turn off the helium gas and carefully lower the can off of the insert.
e. The two taper seal surfaces should be cleaned to remove the grease and the can stored in the tube on the insert cart. Keeping in mind any radiation hazards the sample can now be removed.

IX. Drawings & Pictures:

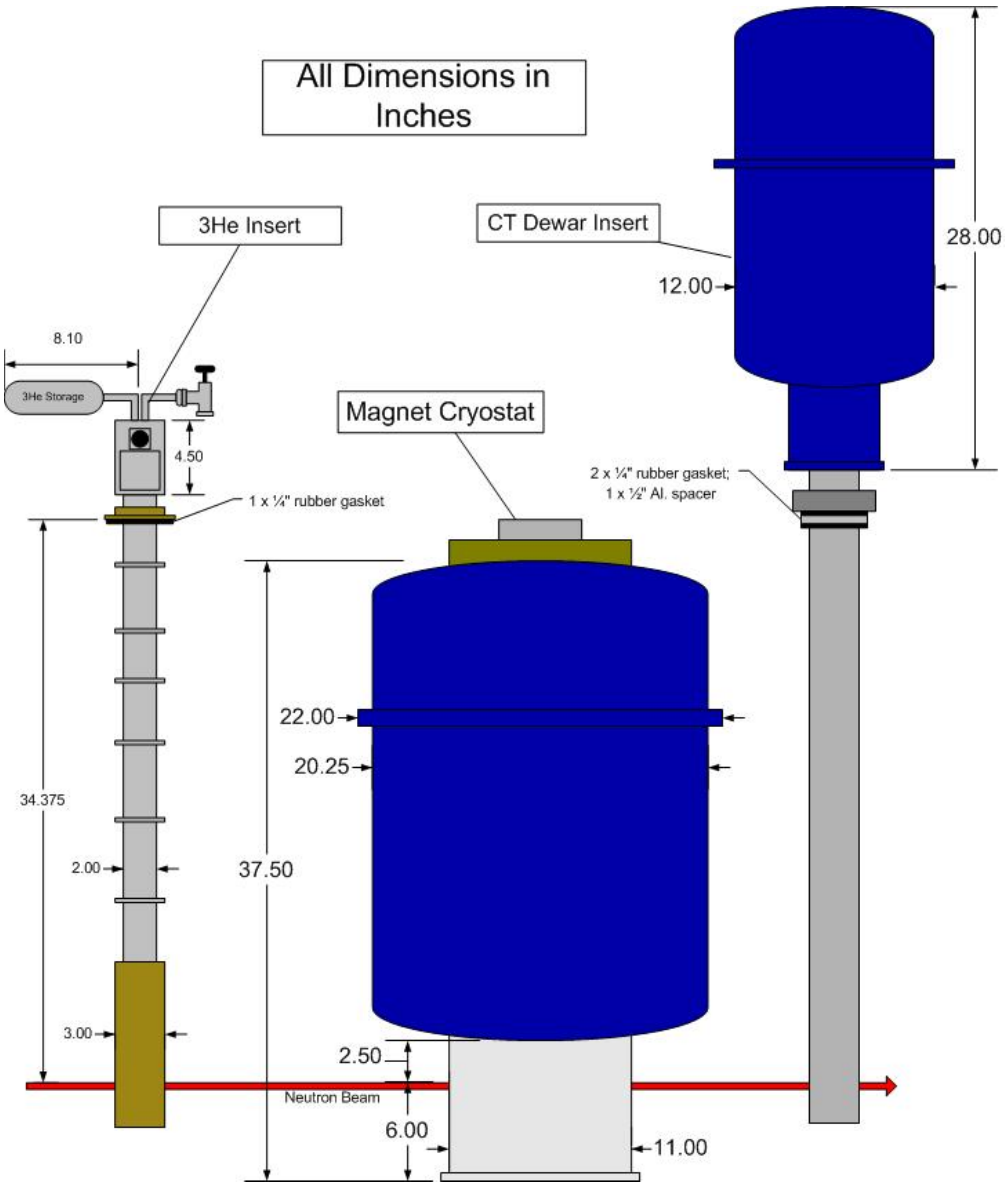

Figure 1: Magnet and Inserts' Outer Dimensions

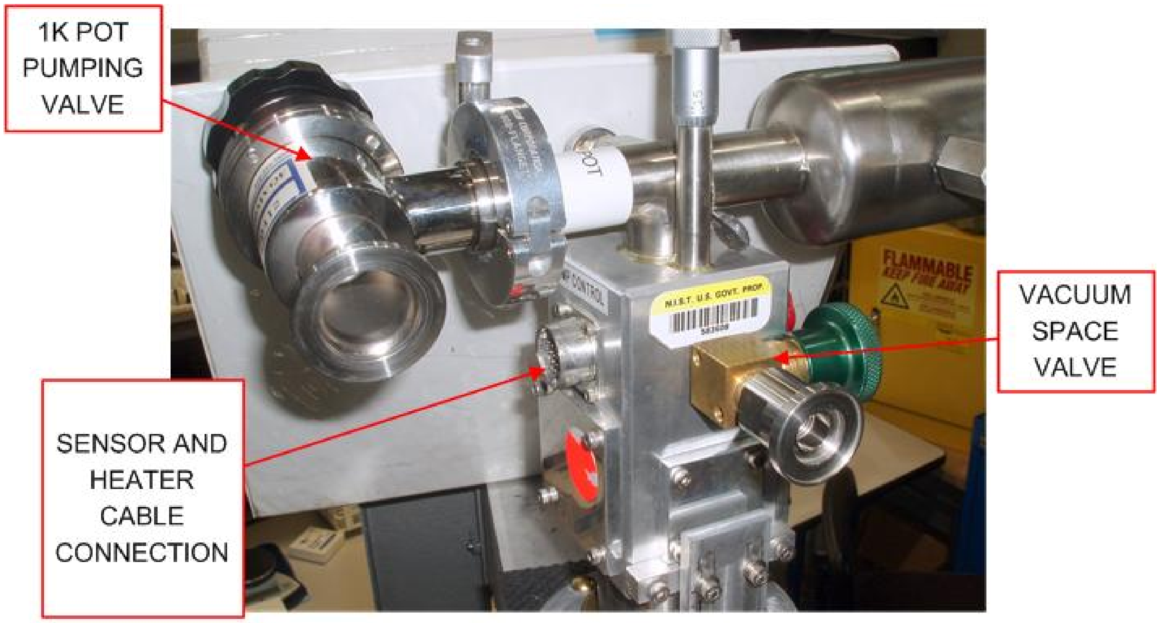

Figure 2: Insert Top Connections

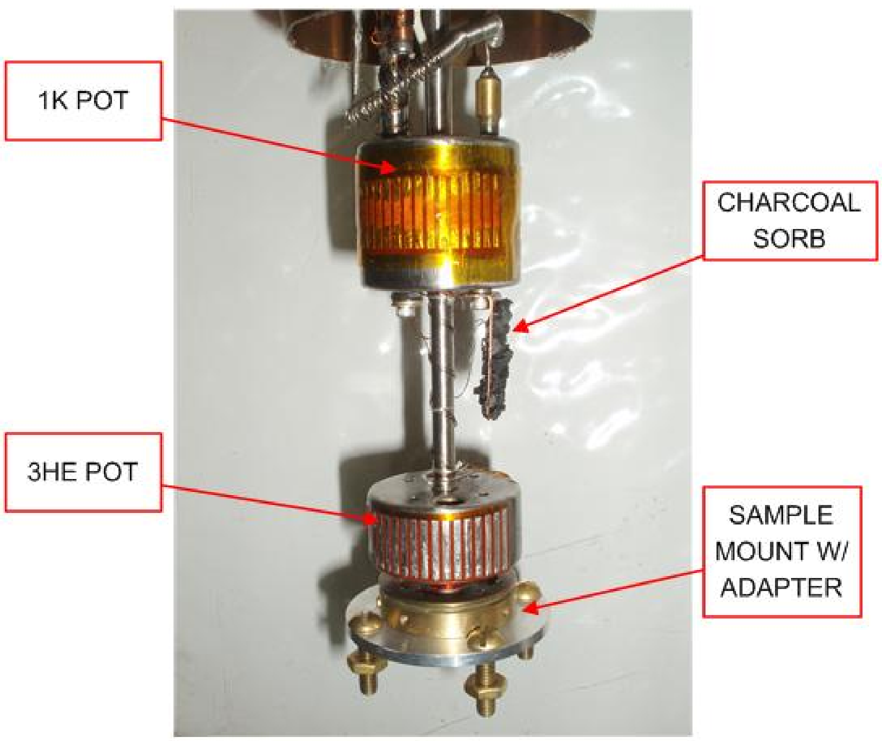

Figure 3: Insert Parts

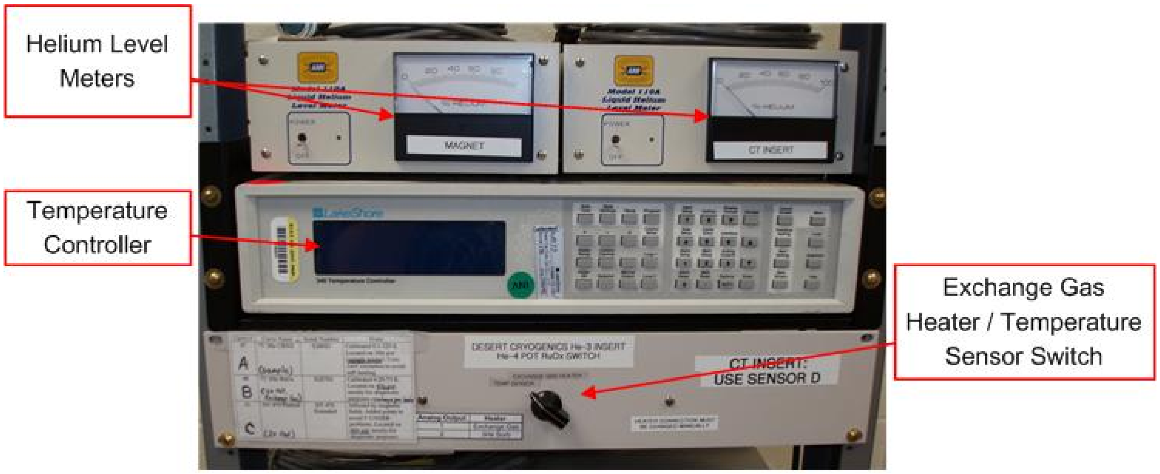

Figure 4: Electronics Rack Setup

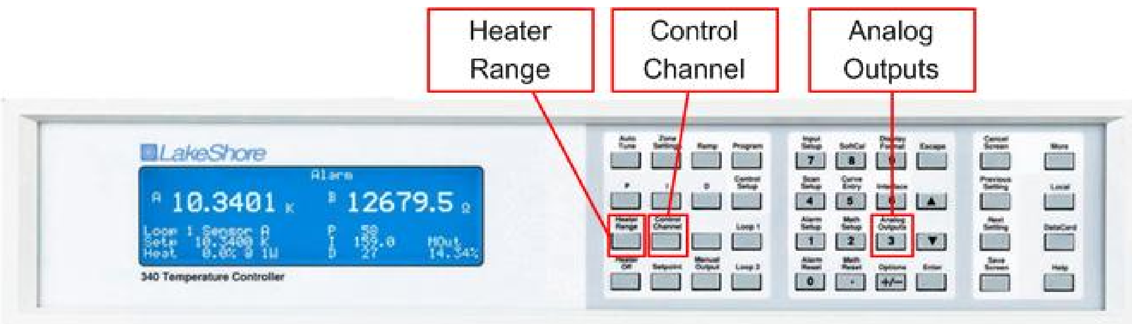

Figure 5: Temperature Controller Front Panel