9T Horizontal Field Magnet Operation Manual

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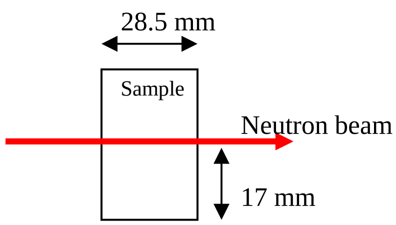
I. Prepping

1. Pump on the outer vacuum space until the pressure is less than $1x10^{-4}$ torr, but preferably just let it pump overnight.
2. Pump out the sample well using a rough pump, then backfill with helium gas. Repeat this three times, the last time leaving the well filled with gas and seal off. NEVER exceed a pressure of 5psi in the sample well space, a slight overpressure (1-2psi) is all that is necessary.
3. Using the rough pump, pump out the annulus with the needle valve closed and purge with He gas to prevent condensation from forming during the cooling process. Repeat the process twice more, the last time sealing the He gas in the annulus. Then do the same thing to the liquid helium reservoir, the last time sealing the He gas in it.
4. Leave the needle valve and annulus exhaust valve closed and open the He exhaust valve. Begin filling the He reservoir with LN_2 through the helium fill port and let the system cool to 77K. The magnet temperature sensors will have a resistance of ~7.6 Ω at this point (~30Ω equals room temp.). After one hour begin filling the liquid nitrogen reservoir as well. When the liquid nitrogen reservoir is full stop filling both reservoirs and check the level in the liquid helium reservoir using a bakelite stick. If there is greater than five inches of liquid nitrogen close the two open valves and let the system sit overnight. If there is less than five inches continue filling another 15 minutes then check again, repeating until there is greater than five inches of liquid nitrogen present.
5. When the magnet has cooled to nitrogen temperature (normally after sitting overnight), the remaining nitrogen in the helium reservoir needs to be blown out. The best way to do this is to blow warm He gas, at about 5 psi, in through the He exhaust port. There is a piece of stainless steel tubing with a Teflon tip that is attached to the magnet cart, attach a rubber hose to the end and place in the He fill port. The nitrogen will spray out here, so you may want to catch it in a small dewar. Remove the large brass relief valve from the He exhaust and replace with a blank-off flange to build up enough pressure.
6. Continue to blow the warm helium through until the magnet temperature has risen to ~90K (9-10 Ω) to be sure all the nitrogen has been removed. You may use the magnet heater to boil off the last bit of liquid nitrogen. The current through the 50 Ω heater should not exceed 1 Amp.
7. Replace the large brass relief valve and then, as in step 3, pump purge the He reservoir with He gas three times, the last time sealing the He gas in it.
8. Now you can fill the helium reservoir with liquid helium. There should be a brass fitting in the small parts box that needs to be put on the end of the transfer line that goes into the cryostat. (This is because our transfer tubes are smaller than the fill port and we don't want He spraying out of that port.) Make sure the He exhaust valve is open. Shortly after starting the fill open the needle valve, begin pumping on the annulus with a rough pump and slowly open the annulus valve. This will pull a steady flow of He gas through the needle valve to prevent it from blocking. Be careful not to overwhelm the pump, obvious by the smell of oil and or loud noises, and avoided by closing down the needle valve almost all the way. The liquid nitrogen should also be topped off during this time.

9. Once the magnet has reached helium temperature (~2.6Ω) you can monitor the progress with the He level sensor. The meter measures in millimeters and the reservoir is full when the sensor reads 240mm or above (anything greater than that is an erroneous reading that is equal to 240mm).
10. Refill LHe when the level gets below 40mm and top-off the liquid nitrogen every time you fill helium.

II. Sample Loading:

1. Note the sample mounting dimensions below and make an appropriate sample holder. These dimensions come from physical limitations in the sample well. The only dimension with little restrictions is the space above the beam because the stick can be moved up to accommodate the sample holder.



Distance to beam from top of airlock = 1363 mm
Distance from beam to bottom of sample well = 17 mm
Max diameter = 28.5 mm
Mounting Surface: 8x M3 tapped holes, 0.15" depth on 24mm diameter and 5/16"-18 tapped hole in center, 0.25" depth.

2. Mount the sample onto the sample stick and mark the clamp location. (Note: the 1363mm distance is correct only when the sample stick is cold, when warm it will be too long by ~4mm.) If the sample is a single crystal, also mark appropriate orientation information. Lightly grease the entire length of the stick to prevent leaks from forming around the o-rings inside the green fitting.
3. Make sure the gate valve is closed.
4. Mount the sample stick in the airlock and tighten the clamp. Make sure the height adjustment clamp is properly tightened so that the stick is secure.
5. Connect a turbo pump to the airlock evacuation valve and pump it out to 1 x 10^{-4} mbar and then close the evacuation valve.
6. Open the gate valve and slowly lower the stick after loosening the height adjustment clamp. When it is in the correct position tighten the clamp and fine tune the height and alignment using a neutron camera.

III. Procedure for Unloading the Sample

1. Loosen the height adjustment clamp.
2. Slowly pull the stick all the way up. If the sample well has been cold, you may have to stop and let it warm up several times along the way. The cold stick freezes the o-rings, but they can be warmed using a heat gun. Do not force the stick through the O-rings, or they may crack and cause leaks and do not overheat them because they will melt.

3. Secure the height adjustment clamp to hold the stick up and close the gate valve.
4. At this point the sample can be removed by venting the airlock. However, if the sample is sensitive to condensation connect helium gas to the airlock evacuation valve and fill the airlock with a foot or two of gas by pinching off the latex tube. Do not over pressure the airlock as there is no relief valve.
5. Allow the sample to warm. There should be no condensation on the airlock when the sample is ready to be removed.
6. Loosen the upper airlock clamp and remove the stick.
7. Cap the airlock with a blank flange then pump it out to 1×10^{-4} mbar and finally close the evacuation valve.

IV. Procedure for Adding Exchange Gas

It may be necessary to add some helium exchange gas to the sample well. This is done once when the magnet is first cooled down. After that, it may be necessary to add helium due to the loss of exchange gas after multiple sample changes. If the sample temperature and the control temperature are not in reasonable agreement, then adding helium exchange gas is a possible solution.

The sapphire windows at the bottom of the sample well are fragile, and easily dislodged by over-pressurizing the sample well! Exchange gas is always introduced in small, controlled amounts.

1. Adjust the regulator on the helium supply to ~2 psi outlet pressure.

2. With helium flowing to purge the line, attach the helium hose to the closed airlock evacuation valve.

3. Pinch off the helium hose tightly approximately 6 inches from the airlock evacuation valve. This trapped volume is the amount that will be introduced to the sample well.

4. Open the airlock evacuation valve to suck in the trapped volume of helium and then close it again. There may be a momentary rush of cold helium exhaust from the helium reservoir when this action is performed.

5. If the sample well is warm and evacuated, add enough exchange gas to bring the pressure up to 10in Hg, which is ***less than*** 1 atm, or 0 psi on the pressure gauge.

6. If the sample and control temperatures do not come into reasonable agreement within 5-10 minutes contact a member of the sample environment team. DO NOT continue to add more helium gas!

7. As the sample well cools, the pressure will drop, due to the ideal gas laws, and the gauge will read 30in Hg (which is the bottom of the gauge), but there is still gas in there. When you add gas, you may not see the pressure rise on this gauge.

8. If you add exchange gas while the sample well is cold notify a member of the sample environment team. What is a reasonable pressure at low temperature can quickly get unreasonable at warmer temperatures, so it is necessary to keep an eye on the pressure as the system warms up.

V. Lakeshore 622 Magnet Power Supply Operation

This supply is setup to be controlled remotely from the SANS instrument control program. However, it may be necessary or preferable on occasion to run the magnet manually. Below are the basics to let you get by even if you have never used this supply before.

1. Connect the leads from the supply to the connections on the top of the cryostat. The polarity does not matter unless you care about the direction of the field through the bore.
2. Turn on the supply and if you are running it remotely connect the RS-232 connection and move to the instrument control computer. The screen should look something like this:

Settings:		Outputs:
0.0000T		0.0000T
Current:	0.000A	0.000A
Compliance:	3.501V	0.165V
Ramp Rate:	0.0050T/s	LVL: 104.2%
Ramp Enable:	<ON>	FLD: 0.000T
Persistence:	<ON>	

3. To run the magnet manually from zero field first turn on the persistence switch heater by using the cursor down arrow until the cursor points to “Persistence”. Then press the up or down key on the number pad to turn persistence off. A small red light below the power button will light to indicate that the heater is on.
4. Once the heater has been on for longer than one minute you can ramp the supply to the desired current to set the desired field in the magnet. To do this move the cursor up to the large number labeled Tesla and set the number by using the right arrow, hitting a number key and finally pressing enter when the correct value is set.
5. When the supply has settled in at the desired current either leave the system as is, non-persistent, or turn off the switch heater, wait a minute and ramp the leads to zero to make the magnet persistent.
6. To then change the field when in persistent mode ramp up the leads by entering the previous set point, turn on the switch heater and set the field to the desired value.

VI. Temperature Control:

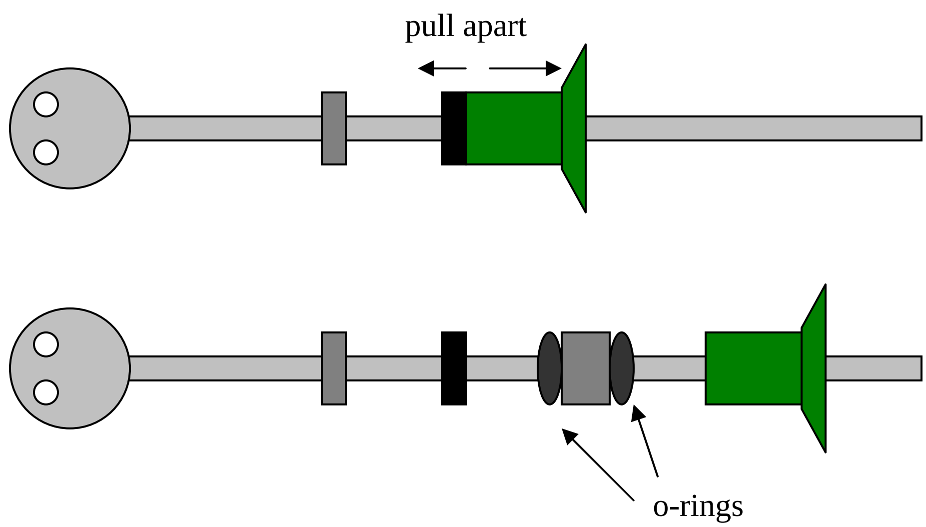
Temperature control is done using a Lakeshore 340 controller that has two sensor inputs and two heater outputs. There are two sensors that are used during operation of the system both of which are Cernox type sensors. The first is located on the VTI heat exchanger and is used for control. The second is located near the bottom of the sample stick and is used for a more accurate reading of the sample temperature. Similarly there are two heaters on the system as well. One is located on the VTI heat exchanger and has a resistance of 50 ohm and maximum current of 1 amp. The other is located near the bottom of the sample stick and has a resistance of 100 ohm and a maximum current of 100mA. The VTI heater is used for normal control of the temperature over the entire temperature range of the system. The sample stick heater, when necessary, is used for fine control at lower temperatures

9. Turn on the controller using the button on the right side of the back of the unit. Verify which sensor, VTI or Sample, is connected to which input, A or B, by reading the labels on the cables and the back of the controller.
10. Plug in the VTI Heater cable single banana plugs into the High and Low plugs on the back of the controller
11. Set the control channel to the VTI sensor by pressing the Control Channel button, selecting the appropriate channel and then pressing Enter.
12. Set the desired temperature by pressing the Setpoint button, then entering the temperature using the number pad and finally pressing Enter.
13. Set the correct heater range by pressing the Heater Range button, selecting a range using the Up or Down Arrow buttons and then pressing Enter. The temperature range you are working in should determine the heater range. The maximum heater range is needed to reach room temperature, but lower temperatures may only require, and may have better control, using a lower range.
14. You know the heater is on by seeing a non-zero percentage of the heater power on the display under the heater range. Keep in mind that if the temperature of the sensor is above the set point the heater will not turn on until necessary.
15. If the temperature is not dropping to your set point it may be necessary to open the cold valve on the top of the cryostat to increase the cooling power. The valve can be opened wide for a quick cool down over a large range, but should always be closed as much as possible once the set point is reached to conserve helium.
16. There will almost always be some difference between the sensors even after staying at one temperature for a long time. The best thing to do is to use the VTI for control and the sample as the actual temperature. This is because the VTI sensor is placed near the heater and controls the temperature of the bottom of the sample well that shields the sample. If a specific sample temperature is needed simply offset the set point on the VTI to compensate.
17. The sample stick heater can also be used if none of the above solutions works. Please contact Evan Fitzgerald (see contacts on last page) before attempting this.

VII. Problem Solving:

If a leak develops in the sample well:

1. Remove the sample stick and cover sample well with blank flange.
2. Remove three bolts on the top of the green fitting and pull apart.
3. Inside are two o-rings, separated by a spacer. Clean with alcohol, then grease the o-rings and reassemble.
4. Make sure the length of the sample stick is cleaned and re-greased before inserting it into the sample well.
5. Follow sample loading procedure as usual.



Note: Possible symptoms of a sample well leak include: stuck sample stick, inefficient cooling in sample well, and increased pressure in sample well.

If the Cold Valve Gets Blocked:

A blockage may occur on the initial cooldown, after refilling cryogens, after an extended period of having the valve closed or after the helium has run dry. The cold valve is blocked if the VTI temperature is not dropping with the needle valve and the annulus valve open. You should also normally be able to feel some flow of helium gas out the annulus valve if things are working correctly. Depending on the severity of the block you may be able to fix the problem with the following steps:

1. Close the cold valve and annulus valve completely.
2. Connect a rough pump to the annulus valve and after opening it pump/purge the annulus with helium gas multiple times.
3. With the annulus pumped out and the pump on open the cold valve and monitor the VTI temperature.

4. If the temperature drops steadily you have removed the block. If it does not drop you should repeat the above steps again. If this fails again continue to the next step.
5. The next option is to raise the temperature of the sample well to 300K and hope that is warms the cold valve assembly enough to melt away the blockage. Once the VTI has reached 300K repeat steps 1 – 4 again.
6. If this does not work the system will need to be warmed by blowing off the cryogens. Contact a member of the sample environment team for assistance before attempting this.

If the Magnet Quenches:

The magnet has quenched when for some reason it goes normal (non-superconducting) while carrying current. Normally the sudden resistance produces enough heat, depending on the amount of current in the coils, to boil off all the helium and warm the magnet to 30 or 40K. The boil off will occur within a matter of a few minutes and is pretty unmistakable. The power supply should also indicate a quench especially if it occurs during ramping. No matter how it happens a member of the sample environment team should be contacted immediately to assist in the recovery as well as determine what went wrong.

VIII. System Specifications:

Sample Probe

Mounting Surface: Gold plated copper (green part is anodized aluminum).
8x M3 tapped holes on 24.0 mm diameter with 0.15" depth.
1x 5/16"-18 tapped hole centered with a 0.25" depth.

Maximum Sample Size: 28.5 mm diameter.

Distance to field center: Top plate surface: 923 mm
Top of airlock assembly: 1363 mm

Field center to bottom of sample well: 17 mm

Sensor Type: Calibrated Cernox
Heater Resistance: 100 Ohm, 100mA (max)
Wiring: User twisted pair: Enameled manganin 1.2mm
Sensor: PTFE sheathed constantan 1.25 mm
Heater: PTFE sheathed Stranded copper 28 AWG

Cryostat

Liquid nitrogen reservoir: Depth: 227 mm

	Volume factor: 1.15 L/cm Capacity: 26 L
Liquid helium reservoir:	Depth: 225mm Volume factor: 1.0 L/cm Capacity: 23.5 L
Heater Resistance / Max Current:	Reservoir: 26 Ohm, 1 Amp Magnet: 50 Ohm, 1 Amp Heat Exchanger: 50 Ohm, 1Amp
Static He boil-off:	0.22 L/hr

Magnet

Central Field constant	0.07565 T/A
Current for 9 Tesla	118.9 Amps
Max field achieved in test at 4.2 K	9.08 Tesla (120 Amps)
Homogeneity over 10mm DSV	2.3×10^{-3}
Homogeneity over 20mm DSV	8.9×10^{-3}
Nominal Coil inductance	32.0 H
Remnant Magnetization	55 Gauss (5.5×10^{-3} T)
Maximum ramp rate	0.18 Amps/sec

Persistent mode switch normal state current	17.9 mA
Switch heater resistance (Pins 1-2)	132 Ω
Switch heater resistance (Pins 5-6)	134 Ω
Switch opening voltage	1.7 V
Recommended switch voltage	2.2 V

Horizontal magnet bore	94 mm
Vertical/horizontal split bore	43 mm
Diameter of stainless outer vessel	254 mm
Overall length	244 mm

IX. Contacts:

Main Magnet Contact: Evan Fitzgerald (x6657, Rm. A123, cell: 240-498-3941)
Secondary Magnet Contact: Dan Dender (x6225, Rm. A118, cell: 443-676-5073)

SANS Contacts:
- NG3: Bryan Greenwald (x5797, Rm. A113)
- NG7: Jeff Krzywon (x6650, Rm.A113)

X. System Drawings/Additional Info.