

Operator's handbook

11T Magnet



Introduction

“11T Magnet” is Dilution refrigerator with built in superconducting magnet. Preparation procedures combine activities which lead to cooling down this system with cryogenic liquids. Main concern is freezing the gases and liquids within the system at lowering the temperature down. That can lead to blocking the capillars, disturb functioning of the needle valves, contamination of the 3He/4He mixture, etc. Thus, the preparation procedures are the cooling system down keeping it at vacuum or properly pressurized conditions. Vacuum purity depends on the refrigerator part considered, from pretty rough in the Main bath to very high in the IVC (Vacuum Chamber). Also, the cryogenic liquids and gases are replaced by each other during precooling procedures. At the end of each precooling step the used liquid has to be completely removed and gas residue has to be pumped out before transferring next liquid with lower base temperature to begin the next precooling step. Therefore, if you will remember to pump the system out and fill it with gases you able to pump out at the end of each precooling step you will never have the problems with 11T Magnet preparation.

Room temperature (Step #1).

Preparation begins with powering up the systems which control 11T Magnet. To perform this step we have to plug in 2 (two) standard 110V cables which are located at the rear side of Magnet cart. No other power sources required.

The image of the cart with electronic devices used for “Magnet” control shown on Fig.1.

1. **Lambda point Refrigerator Control** used for operation of cryogenic system located inside the Main Dewar to cool down the magnet from 4.2 K to 2.2K. It allows rising up the maximal magnetic field from 9T to 11T. Using this control device we can measure the pressure in the pumping line and temperature of the magnet. Also, we can control opening the needle valve of the Lambda point Refrigerator regulating the cooling speed and helium consumption. The opening is ~20% used for stable work. Also, opening can be automatically adjusted for the best performance within predesigned LabView program.
2. **Magnet Power Supply** controlled by predesigned LabView program “Magnet Control” (Fig.4). Before start the program necessary perform 4 steps manually:
 - i. Switch Power supply in to Manual mode (Press **Setting**, Choose the **Manual**, Press **Apply**); Return to main Window pressing **Home**.
 - ii. Set Supply on **Hold** (pressing the corresponding button).
 - iii. Set the ramping rate **Slow**, pressing at the Current and Field indication area, and pressing the **Fast** button.
 - iv. Switch power supply in to USB SCI mode (Press **Setting**, Choose the **USB**, Choose the **SCI**, Press **Apply**); Return to main Window pressing **Home**.

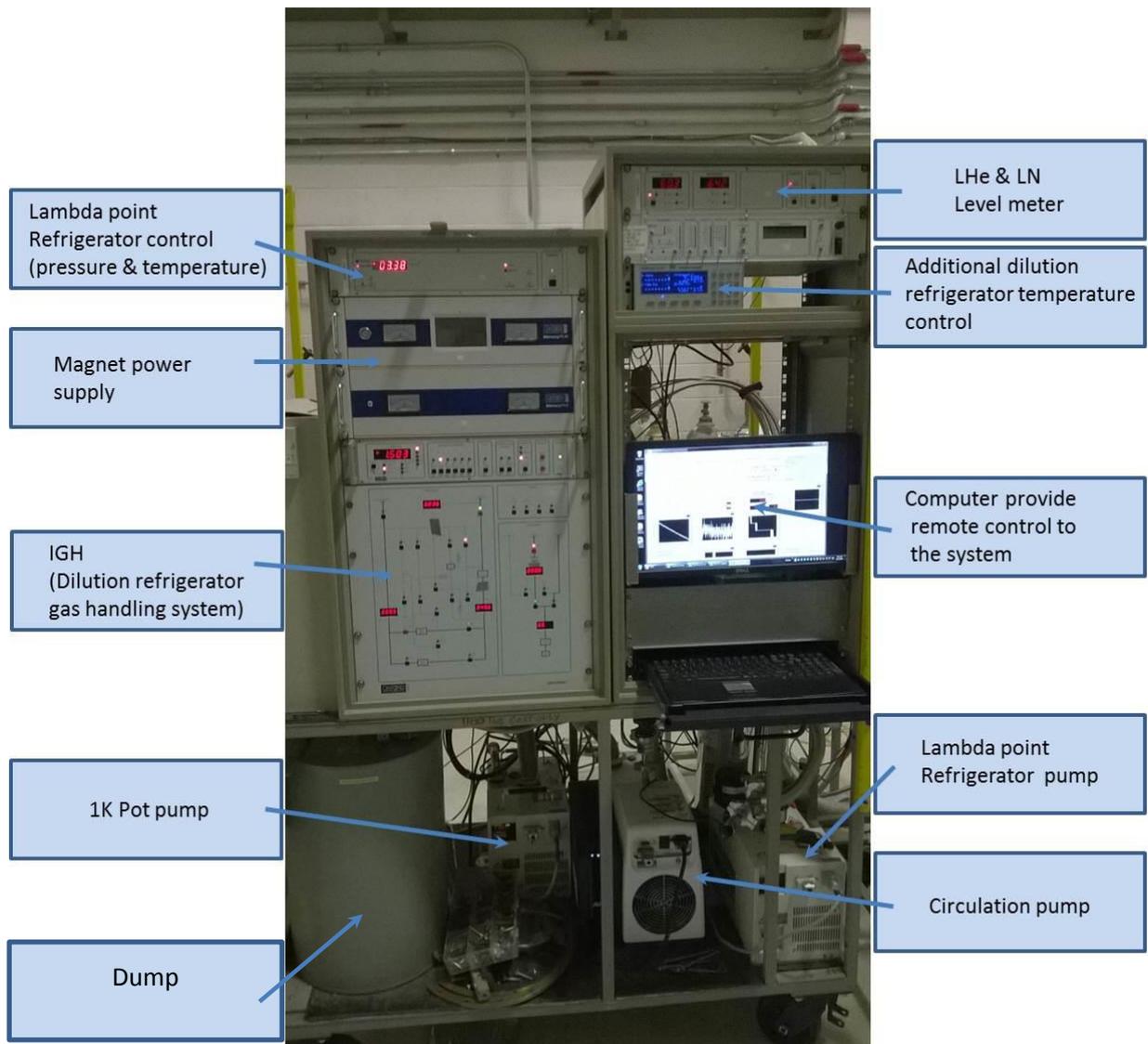


Fig.1

3. **IGH (Dilution Refrigerator Gas Handling system)** can be controlled both remotely and manually. For remote control use program: Gas Handling (Fig.4). Program simulate IGH box, thus, you have to be familiar with main operating regimes of dilution refrigerator to work with IGH within both manual and remote modes. Operating regimes of the Refrigerator will be described in the Operational Manual later.
4. "11T Magnet" uses 3 rough pumps: **1K Pot pump, Lambda point Refrigerator pump and Circulation pump**. All 3 pumps have to be turned on before cooling down. 1K Pot and Lambda point pumps exhausted outside and no problems expected. Circulation pump stays within the closed system and exhaust pressure should be watched when the pump is turned on. The exhaust pressure gauge indicator is G2 on the IGH panel. If the indicator is over ranged: <999.9> open the valve #9, and, if necessary, Dump with manual valve to relief the pressure in the system.

5. **Computer** used for “11T Magnet” remote control is permanently attached to the system. Thus, remote operations do not require any additional cables reconnections. Single exclusion is connection the Magnet to the Neutron Instrument computer. This connection performed with RS232 cable to the computer port: COM #10. LabView program responsible for communication between Magnet and Instrument is: ICE ICP Control (Fig.4).
6. **Additional Temperature Control** used to control and indicates temperatures higher than standard Dilution Refrigerator temperatures: $>1.2\text{K}$. Two channels used. Channel 1(D) indicate the Slug (sample holder) temperature within $40\text{K} < T < 0.05\text{K}$, Channel 2 (C) indicate sample (mixing Chamber) temperature within $273\text{K} < T < 0.3\text{K}$. Channel 2 used for high temperature control. Channel 3(B) indicates 1K Pot temperature.
7. **LHe & LN Level meter** used for measurements of cryogenic liquids level in the 11T Magnet.

Pre-Cooling Time sheet (Step #2).

Generally, it takes 2 days to complete Magnet preparation up to final condensation and circulation stage, which has to be done on the Instrument. This time mostly spend to overnight cooling to nitrogen and helium temperatures. The Pre-cooling strategy is:

1. **Preliminary cleaning** (pumping the inner He3/He4 circulation system with Circulation Pump to the Dump) and vacuum pumping of IVC and Vacuum chamber of 11T Magnet cryostat. It can be done for 5-6 hours, but, preferably, to make it overnight, especially if the cryostat wasn't used for the long time.
2. **Nitrogen pre-cooling**. During this stage the liquid nitrogen has to be transferred to the nitrogen and helium bath of the 11T Magnet cryostat (transfer duration is 2 hours approximately). That step is completed when the temperature of the magnet (measured with Lambda point refrigerator control) and inside the IVC (measured with additional Dil. Fridge temperature control) is around the Liquid nitrogen temperature $\sim 77\text{K}$ ($\sim 80\text{-}100\text{K}$ in reality). There is an overnight step.
3. **Helium pre-cooling**. At the next day the final step of pre-cooling procedure started with evacuation of liquid nitrogen from helium bath. It takes up to 1 hour and this time definitely depends on LN amount previously transferred for nitrogen pre-cooling. Liquid He transfer started and takes 1-2 hours after nitrogen is removed from helium bath. The some amount of He⁴ exchange gas has to be added to the IVC before pre-cooling the cryostat. When temperature inside the IVC reaches approximately 10K the exchange gas should be pumped out from IVC. The helium pre-cooling and exchange gas pumping is overnight procedure.

1 day (Cleaning and LN pre-cooling) - **2 day** (LHe pre-cooling and IVC pumping) - **3 day** (Experiment).

The precooling is the preparation of refrigerator to work with cryogenic liquids. It includes pumping of these liquids through the narrow capillars. This capillars can be easily blocked during cool down process by frozen liquids and gases. Thus, one more important step of pre-cooling procedure is throughput test. Throughput test performed twice: at room and liquid nitrogen temperatures. During

this test we check the gases flow through: 1. Condenser line (He3/He4 system); 2. 1K Pot line; 3. Lambda Fridge line. Test procedure described below.

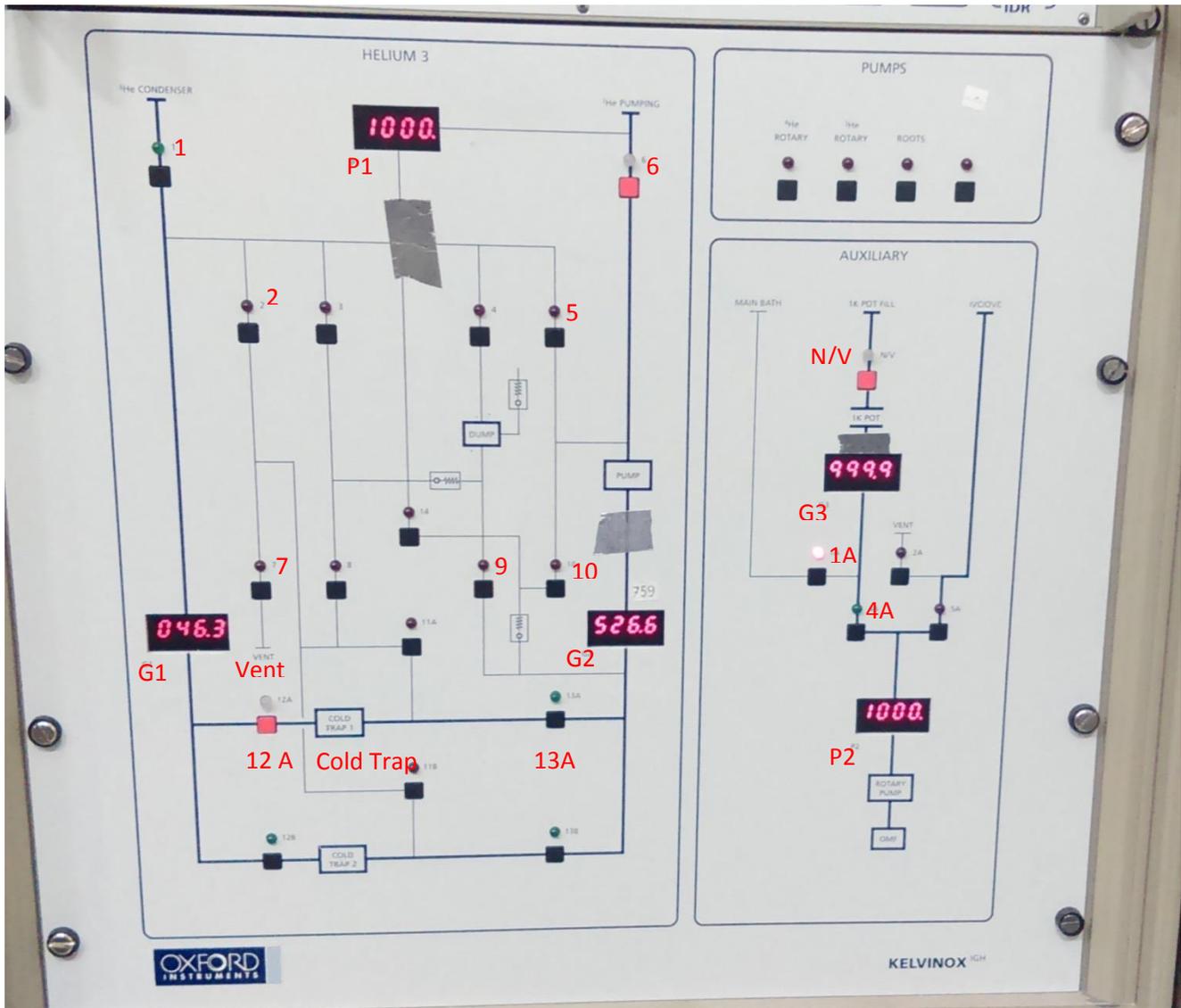


Fig.2

Condenser line:

- Turn on the Circulation Pump Fig.1
- Open Condenser Valve (2) and Still Valve (1) shown on Fig.3.
- Completely Open 6, open 5, and 1 (Fig.2). Pump the He3-He4 system till pressure on the P1 drop down to ~ 0.03 . Pressure on the G2 should be lower than 999.9, otherwise open Valve 9, and, if necessary, Dump Manual valve Fig.1.
- Close 6 and 5 to stop pumping.
- Nitrogen "Cold Trap 1" shown on the Fig.2 and physically located behind the cryostat has to be filled with LN.

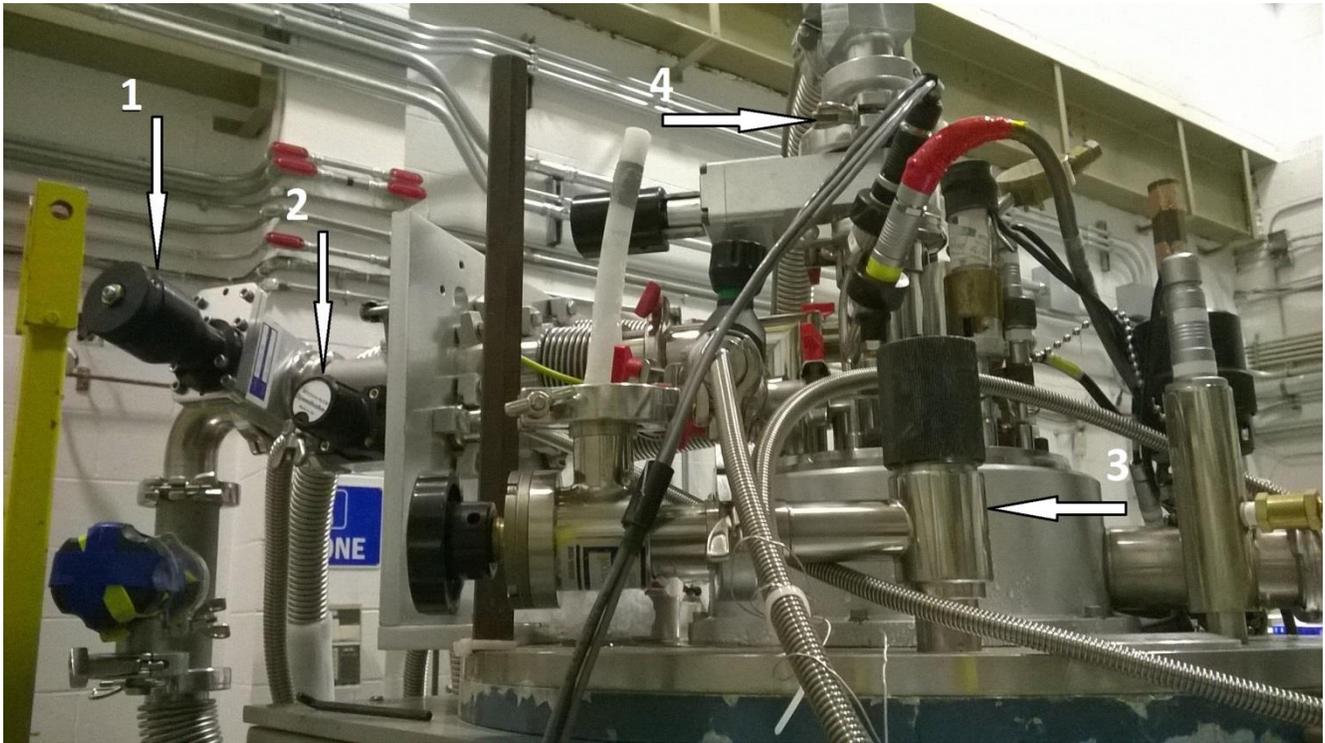


Fig.3

1. Still Valve; 2. Condenser Valve; 3. Vacuum Jacket Valve; 4. Sample load Port.

- Open 13A, and 12A for 10% approx.
- The throughput test is successful when pressure P1 starts slowly rise up(for example from 0.05 to 0.1 within a minute). If the pressure doesn't change within several minutes the condenser capillar is blocked.

1K Pot line:

- Turn on the 1K Pot pump Fig.1
- Open 4A and 1A to pump out the main bath.
- Close 4A and fill the main bath with Helium gas using the exhaust output on the top of the cryostat.
- Close 1A and open 4A to pump out G3 volume.
- Close 4A and open N/V up to 40% to let the Helium gas flow from the main bath to G3 volume.
- The throughput test is successful when pressure G3 starts rapid rise from 0. If the pressure doesn't change within a minute start over and check the needle valve motor rotation. If it got stuck help manually to rotate it CCW.

Lambda Fridge line:

- Turn on the Lambda Point Refrigerator pump Fig.1
- Open two manual valves on the line (at the pump and on top of refrigerator).
- Pressure should be controlled with Lambda Point Refrigerator control.
- When the pressure drop to zero close one of the manual valve and open (~40%) the needle valve using LabView program "Gas Handling".
- The throughput test is successful when pressure starts rapid rise from 0. If the pressure doesn't change within a minute start over and check the needle valve motor rotation. If it got stuck help manually to rotate it CCW.

Troubleshooting:

The test performed at room and N₂ temperatures. The room temperature failure can be caused by capillars blockage by liquids (oil, etc.) or problem with the needle valve motors (1K Pot&Lambda). Currently, oil pumps are replaced with dry ones which significantly reduce the risk of capillars blockage. In the case of blockage define what liquid caused the problem and start pump the system with Turbo pumps, using heating if it's possible. Cleaning the system is a long process which can require several days.

The N₂ temperature failure is caused usually by a leak from outside to the system under test. Try to find a leak, localize it, and replace the leaking part if it is possible. Otherwise the refrigerator has to be warmed up to room temperature to make a repair.

Step by step instruction of cryostat preparation and operation:

- **Preliminary cleaning**
 1. Connect external turbo pumps to Vacuum Jacket line (3) and Sample load port (4) Fig.3. Pump it till pressure goes down to $\sim 10^{-6}$ Torr.
 2. Turn on 1K Pot pump, Lambda point Refrigerator pump and Circulation pump (Fig.1).
 3. Then pump: **1)** Circulation system: open 1, 5, 6(100%) (lowest pressure on P1 is ~ 0.03) **2)** 1K Pot system: open 4A and Manual valve on top of the cryostat. (lowest pressure on P2 is ~ 0.05) **3)** Lambda Fridge system: open manual valves on top of the cryostats and another one placed just before the pump input. (lowest pressure on Lambda Point Refrigerator control is 0). Pump for 10-20 min.
 4. Close valves and make room temperature throughput test.
- **Nitrogen pre-cooling**
 1. The main Bath has to be filled with Helium gas after throughput test. If it wasn't pump it and fill with helium gas up to atmosphere pressure.
 2. Using two liquid Nitrogen dewars start transfer LN to helium and nitrogen volume. Helium port located close to Sample transfer port and Nitrogen port is close to

Vacuum Jacket port. Open the Helium volume (Main Bath) exhaust Valve on top of the cryostat before transfer.

3. Transfer to the Nitrogen volume has to be stopped when LN level is 100% on LN&LHe meter (Fig.1). Transfer to the Helium volume has to be stopped when magnet temperature on the Lambda Point Refrigerator control drop down from 99K to ~85K and kept at this level.
4. Put exchange Helium gas to the IVC through the input on top of the cryostat (20 cm of rubber tube, approx..).
5. Close the Baffle (valve on top of the cryostat, turn clockwise).
6. Leave system for overnight cooling. The next morning Mixing Chamber temperature should be around 100K.
7. Fill the LN trap located behind the cryostat with Nitrogen.

* Transfer tube for the He volume must touch the bottom which is actually is not the bottom of the Refrigerator, but the tube extension. Transfer with the extension tube allows cool down magnet more efficiently. Also, it let you be sure that extension tube isn't blocked. Blockage of the extension tube makes the cool down impossible.

- **Helium pre-cooling**

1. Evacuate liquid Nitrogen from Helium volume. Evacuation procedure is: apply helium gas pressure to the Main bath exhaust; put transfer tube into the Main Bath (be sure the transfer tube firmly touches the extension tube); connect the transfer tube output with the LN volume to evacuate Nitrogen (generally, you can use any volume able to store LN); open exhaust valve to apply Helium gas pressure.
2. Pump out the Main Bath volume from Nitrogen gas after liquid Nitrogen evacuated. To do this turn on the 1KPot pump, open 4A, 1A.
3. Pressure should drop down to several units on P2. It means no liquid left inside the Main Bath.
4. Stop pumping: close 4A and 1A.
5. Fill the Main Bath with Helium gas using the exhaust output.
6. Make Nitrogen temperature throughput test.
7. Set Sorb Pump temperature = 50K.
8. Start transfer LHe to helium volume using Helium port. Transfer tube must touch the extension tube.
9. Leave the system cool down to the Mixing Chamber temperature ~6K.
10. Connect turbo pump to the Sample port 4 (Fig.3).
11. Start pump out exchange gas from IVC.
12. Cooling to ~6K plus pumping are overnight procedures. It is important to close the baffle using valve on the top of cryostat turning it clockwise before the pumping of exchange gas starts. Otherwise, room temperature radiation starts warm up inside fast.

- **Condensation and Circulation of He3/He4 mixture.**

1. Set Sorb Pump temperature on = 0 K.
2. Start pumping 1K Pot: Turn 1K Pump on; Open 4A; Open manual 1K Pot Valve on top of the cryostat; Open N/V (Fig. 2) for ~10%, P2 is approximately 5-8.
3. Check the 1K temperature on Dilution refrigerator temperature control (Cryocon 44). The temperature is approximately 1.2-1.5 K.
4. Open Still #1 and Condenser #2 manual valves on top of the cryostat (Fig3).
5. Begin He3/He4 mixture condensation: Turn the Circulation Pump on; Open 9; Open Dump manual valve; Check the LN trap is filled; Open 13A; Open 1; Open 12A ~0.5%.
6. Initial pressure on P1 is 0.03 approximately. It should start rising slowly (within couple of minutes) after beginning of condensation process. If it doesn't the condenser line can be blocked. The LHe trap prevents the condenser line capillars blockage inside the refrigerator. The physical location of LHe trap is on top of the cryostat.

Troubleshooting:

- a. Pump the mixture back to the Dump using Circulation Pump for 20 min.
 - b. Close valves which separate volumes with mixture from rest of the system.
 - c. Connect external Rough pump to the Vent (Fig.2) which is physically located at the rear side of IGH.
 - d. Open 1 and take the He trap out of the cryostat.
 - e. Using pressure gauge G1 check the pressure rising. It happens due to unfreezing of the gases caused blockage of the trap.
 - f. Clean the system with external Rough pump opening 2, 7, and manual valve at the physical location of Vent.
 - g. Try to find the reason of the blockage and replace the leaking parts.
7. Continue the condensation keeping G1 lower than 100 by controlling 12A opening.
 8. When G2 dropped down to ~100 open 12A completely, close 9 and open 10 to pump out the rest of the mixture from Dump.
 9. Close and open 10 keeping G2 between 100 and 200.
 10. Close 10 permanently when pressure drops lower than 150 at 10 opened.
 11. Now system starts circulate and cool down to base temperature. At this state opened valves are 1, 13A, (12A, 6 opened completely), G1, G2 should drop down to ~40.
 12. Temperature can be controlled using IGH MixChamber indicator and dilution refrigerator temperature control (Cryocon 44).

- Programs to control 11T Magnet.

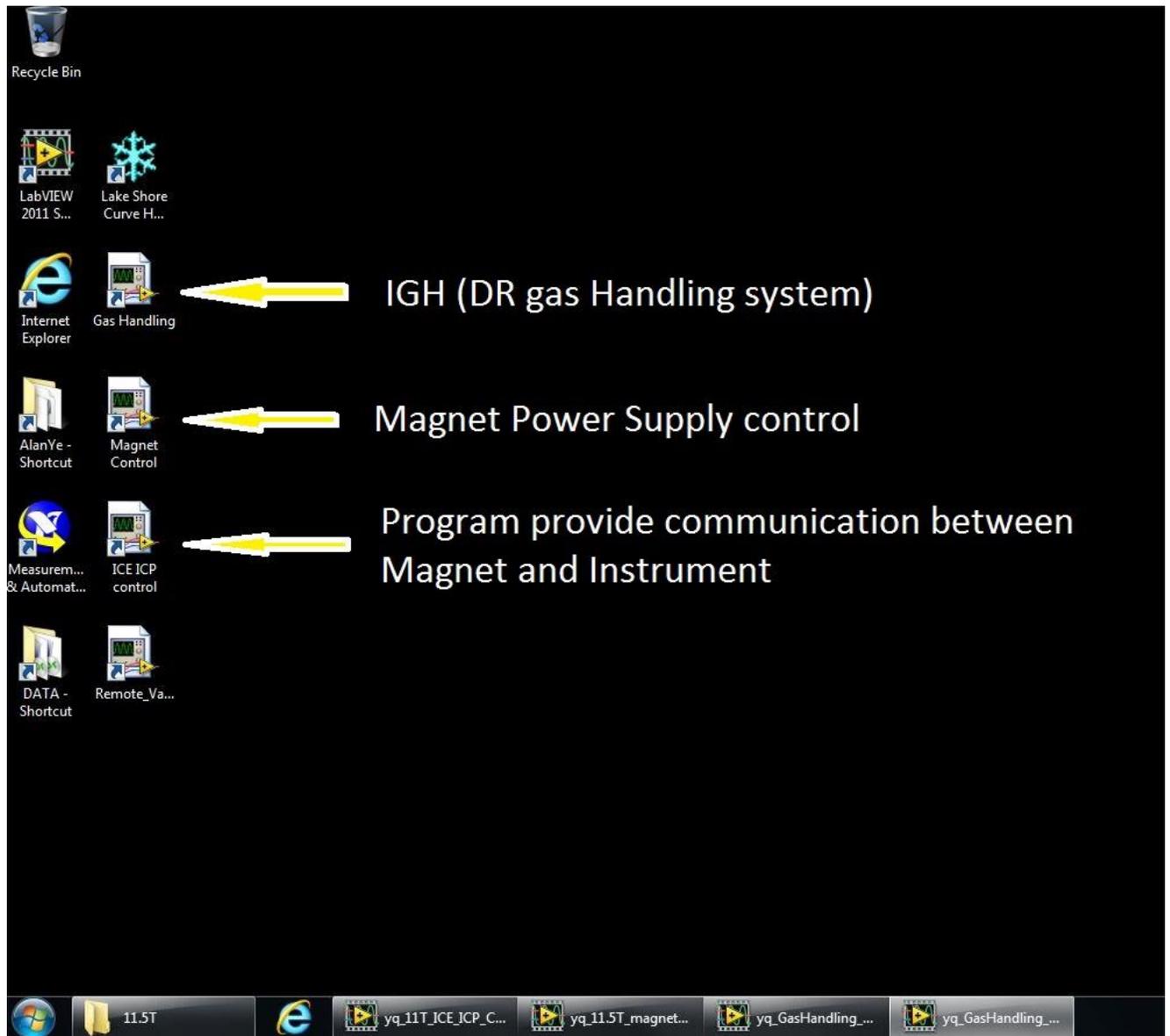


Fig.4

- Basic procedure for Loading and Unloading sample.

I. **Loading**

1. Sample diameter should be less than 1 inch. Use vacuum grease at all screw attachments.
2. Attach slug with torque wrench set to 12.5 N.m. Notice that the thread is left handed. Bottom part of slug is tightened to “finger tight”. Mark the key location on top of the stick.

3. Attach air lock to central port. Open baffles. Pump out the air in the air lock. (NOTE: Turn counter clockwise to open the baffle.)
4. Continue pumping. Open the air lock and push sample stick down. Align the key using marks on top of stick and top of cryostat.
5. Slug drops down when keys have aligned. The distance between the lower edge of the stick head and the top of the sample chamber is ~ 67 mm.
6. Turn clockwise 3 turns then the slug drops to ~ 44mm.
7. Continue turning clockwise until slug breaks loose (~8.5 turns). It's tight.
8. Make 6 more turns the top loading stick to detach slug. Stick can be lifted.

II. **Unloading**

1. Make sure the baffle is open.
2. Slowly push stick to bottom.
3. Turn 6 turns counter clockwise.
4. If it is very tight at this point, wait for about 10 minutes.
5. After ~9 turns counter clockwise slug is loose and ready to be pulled out.
6. Attach clamp to hold in place when out. Remember tip of slug is loose on exit so the stick must be held vertical until sample has been removed.