USER GUIDE

OXFORD INSTRUMENTS CONTINUOUS-FLOW HE CRYOSTATS

Stanford Synchrotron Radiation Laboratory

Structural Molecular Biology/X-ray Absorption Spectroscopy Group

OPERATING INSTRUCTIONS

SSRL SMB/XAS CONTINUOUS FLOW

LIQUID HELIUM CRYOSTATS

Oxford Instruments CF1208

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Section 0. SAFETY NOTE:

Liquid helium and liquid nitrogen, and the surfaces that have been in contact with them, can cause severe burns.

Always wear thick insulating gloves and safety glases and exercise appropriate caution when changing samples, changing dewars, or assembling or dismantling the cryostat.

If affected, please seek prompt medical attention.

Please see Section 10 for lHe supply dewar information.

Section 1. OVERVIEW OF COMPONENTS AND FUNCTION:

The SSRL Structural Molecular Biology X-ray Absorption Spectroscopy (SMB/XAS) group provides Oxford Instruments CF1208 continuous flow liquid helium cryostat systems for SSRL users. These cryostats usually operate at temperatures between 4 and 220 K, maintain the sample in non-vacuum conditions under a carrier gas, and enable rapid sample change and quick re-equilibration at the desired cryogenic temperature (typical total sample change time is about 10 minutes).

The cryostat consists of the following components: a cryostat body and sample probe (see Figure 1, Section 1.8); a LHe transfer arm (see Figure 2, Section 1.8); a LHe dewar (see Figure 4, section 10.2); a cabinet or rack containing a temperature controller (see Figure 3, section 1.8), a gas flow controller, a diaphragm pump, a rough pump, switches, valves and gauges; and a turbo pump system.

The cryostat system can be functionally divided into three separate flow systems (or spaces): the sample space, the vacuum space, and the LHe flow system. Each of these and other aspects of the complete system are described in detail below.

1.1. Sample Space:

The space consists of a 30 mm diameter tube down the center of the cryostat body and a shroud with three or four x-ray transparent windows. The sample probe is inserted into this sample space. An adapter is attached at the end of the sample probe and is designed to hold different sample holders (for solids, solutions, films, *etc.*). The space is filled with He or N_2 gas (depending on the operating temperature) during measurements. The atmosphere of this space is controlled via toggle valves on the cryostat control panel, a rough vacuum pump, and He and N_2 gas cylinders. The sample space is not connected in any way to either the vacuum space or the LHe flow system (except by thermal contact with the heat exchanger, see below).

1.2. Vacuum Space:

This space surrounds the sample space and consists of the area between it and the outer shroud. There is a radiation shield inside this space. Under normal operation, the vacuum space is dynamically pumped with the turbo pump system to assure thermal insulation of the sample space.

When using He gas in the sample space (or if the aluminized Kapton windows of the sample space have a minor leak), sample space gas will diffuse into the vacuum space, thereby degrading the thermal insulation, unless the vacuum space is dynamically pumped with the turbo pump.

1.3. LHe Flow System:

This system consists of the LHe dewar, the LHe transfer line, the cryostat heat exchanger, the gas flow controller, the gas diaphragm pump, and the sensor/heater subsystem (including the temperature controller). The temperature controller, gas flow controller and diaphragm pump are all separate units.

1.4. Transfer Line:

The LHe transfer line is constructed as three separate spaces; an overall vacuum space for thermal isolation, a LHe in-flow capillary connecting the dewar and the heat exchanger, and a return-flow line that acts as a thermal shield for the LHe capillary. The transfer line vacuum space is pumped before use to low pressure ($<10^{-5}$ torr) and valved off. The flow of LHe starts at the bottom of the transfer line storage vessel leg, passes through the LHe in-flow capillary of the transfer line and into the heat exchanger in the cryostat. The return flow connects the exhaust from the heat exchanger to the gas diaphragm pumping system via the barb/polyflo connection near the transfer line needle valve (atop the storage dewar). Care should be taken not to bend to a tight radius or compress the flexible tubing, as this could result in breakage of the internal lines.

1.5. Cryostat Windows:

The windows consist of aluminized Kapton, with the following thicknesses: inner shroud 0.002" or 0.003", heat shield 0.00025", outer shroud 0.002" or 0.003". The window sizes are 20 mm circular on the beam entrance side, and 70 mm circular on the fluorescence exit side. The opening for fluorescence collection is a cone with 90° opening angle. Caution should be exercised in order not to puncture the thin cryostat windows. The white teflon shroud shield should be used to protect the windows when necessary.

1.6. Cryostat Operation:

The operation of the cryostat is based on a continuous flow of LHe through the cryostat heat exchanger by pulling the LHe from the LHe dewar all the way through the system, using the diaphragm pump. The heat exchanger thus removes heat from the column of gas and the metal in the sample space which in turn remove heat from the sample. Coarse temperature control is maintained by adjusting the LHe flow. Fine temperature control is maintained by means of a heater at the cryostat heat exchanger, the current to which is proportionally controlled by means of a Rh/Fe T/C sensor on the heat exchanger and a set point on the temperature controller in the rack.

During the initial cooldown of the transfer line, if the coupling nut on the delivery leg is not tight (see below), the He flow immediately returns back along the return flow space of the transfer line without going through the cryostat heat exchanger. When the coupling nut is tight (under normal operation), the LHe flows into the heat exchanger, is vaporized, and returns through the return flow space of the transfer line, out through the barb/polyflo connection, through the polyflo line to the gas flow controller (in through the "to cryostat" port, through the vacuum gauge, needle valve, out through the "to pump" port), through the pump, back through the gas flow controller (in through the "from pump" port), through the flow meter, and finally out (through the "recovery" port) to atmosphere (He is not recovered).

During normal operation, the LHe consumption with our older transfer lines is >1.5 liter/hour at 4 K, ~1.0-1.2 liter/hour at 10 K, ~0.6-0.7 liter/hour at 80 K and ~0.2-0.3 liter/hour at 200 K. Our newer transfer lines consume ~30-50% less. The cryostat temperature becomes somewhat unstable at 💿200 K due to the low LHe flow required, which is difficult to regulate. We do not recommend using the cryostat at temperatures higher than 225 K. (The He flow is measured on the flowmeter at the *lower edge* of the top of the floater).

1.7. Temperature Controller:

The temperature controller can be used in Manual or Auto mode. Since there are several different controllers in use, a detailed description of their use will not be given here - please refer to the respective Temperature Controller Manual. However, the following guide lines are generally applicable:

In Manual mode, the temperature is regulated by the LHe flow only. The cryostats are very stable, and if a regulation of ±1 K over several hours is sufficient, this mode of running is the most economic as no LHe is burned off by the heater. In Manual mode the heater can be set to a desired current, but would serve no purpose but to increase the LHe consumption.

In Auto mode, the controller starts/stops the cryostat heater to maintain the temperature to within ±0.1 K as measured by the Rh/Fe sensor. A temperature Set Point needs to be set, and PID control parameters (proportional, integral, and derivative) can be modified if needed although default values work reasonably well. In this mode the LHe flow is set somewhat too high, and the temperature controller starts/stops the heater to balance the cryostat temperature.

If during the operation of the cryostat it is unclear what to do, and this User Guide does not tell you, please contact an SMB-XAS staff member before taking a potentially erroneous action – thanks!

1.8. Cryostat, Transfer Line, Control Rack Figures:

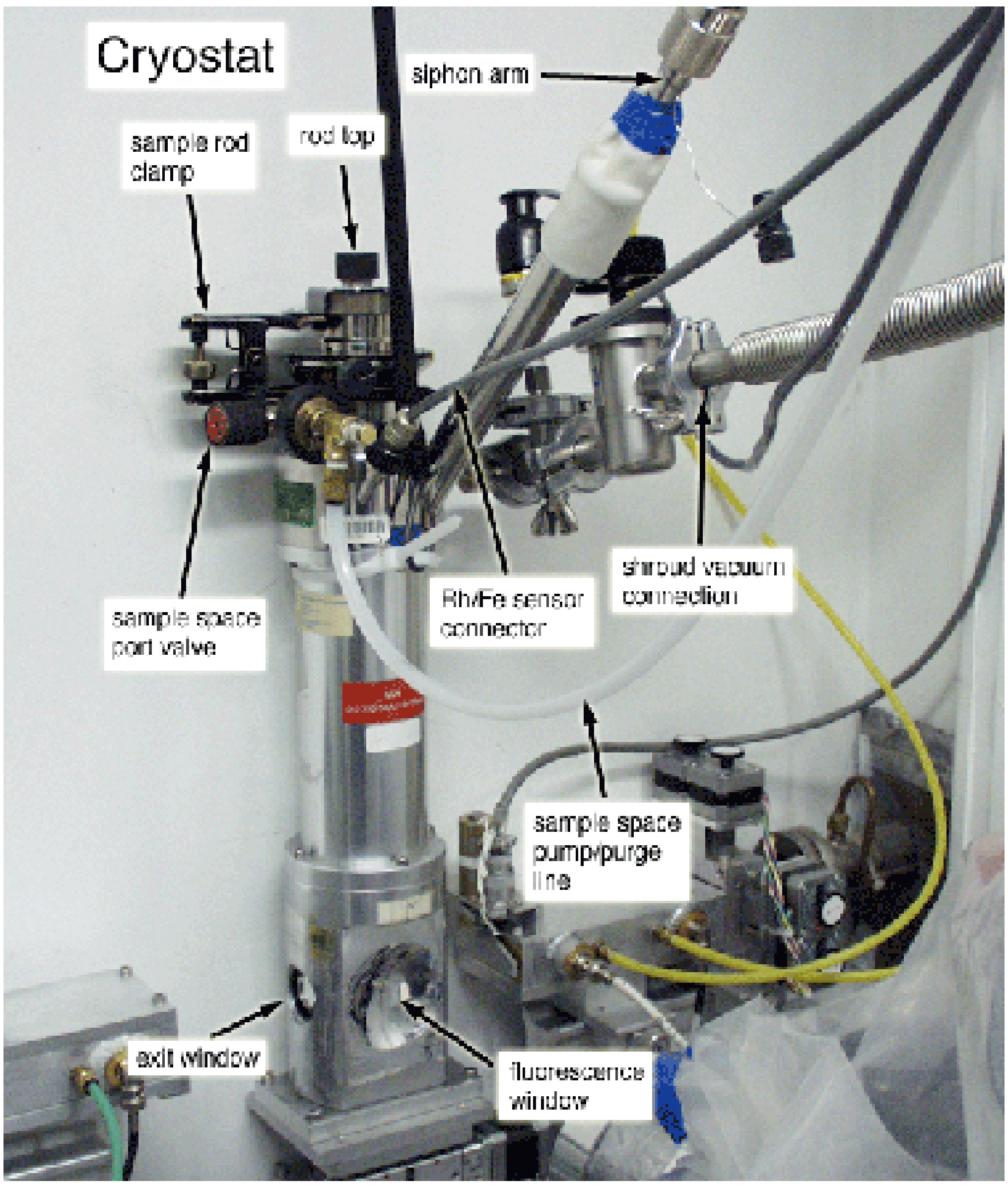

Figure 1: Cryostat

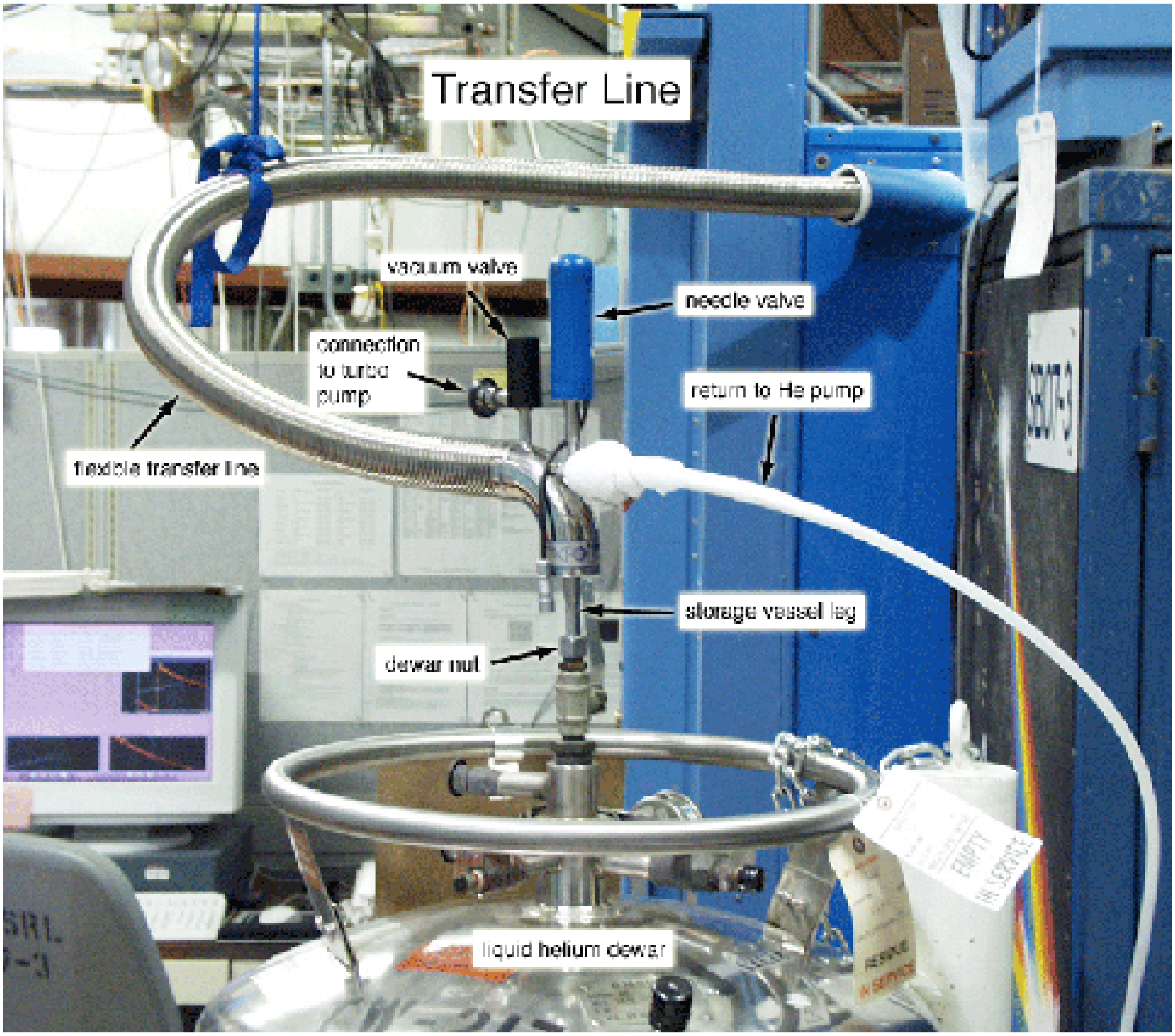

Figure 2: Transfer Line

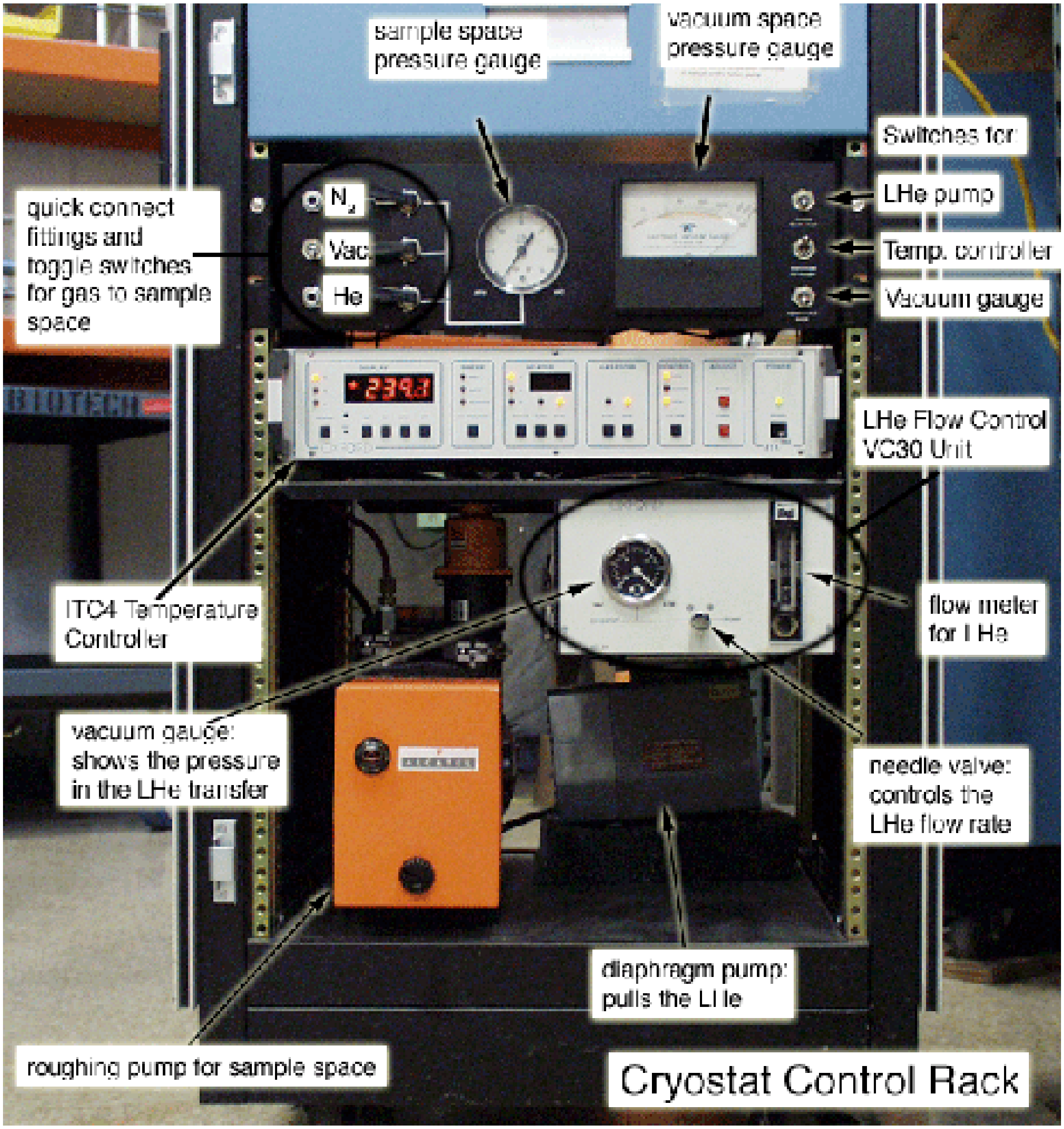

Figure 3: Cryostat Control Rack

Section 2. PUMP DOWN PROCEDURE:

1. Attach the metal flex hose of the turbo pump to the vacuum port on the transfer line. Keep the vacuum valve on the transfer line closed.

2. Open all valves up to the transfer line valve and start the turbo pump system as described in the turbo pump instructions (posted on the turbo pump cart).

3. When the turbo pump gauge shows the desired low pressure ($<10^{-5}$ torr), open the transfer arm vacuum valve very slowly to prevent the creation of overpressure at the turbo pump. The valve should be fully opened gradually.

4. Pump until the pressure of the transfer arm is $< 1 \times 10^{-5}$ torr. Close the transfer arm vacuum valve. Switch off the turbo pump system and vent as described in the posted instructions.

5. Attach the metal flex hose of the turbo pump to the cryostat shroud vacuum port (see Figure 1, Section 1.8).

6. Start the turbo pump system as above. If the cryostat is already under vacuum, open the cryostat valve slowly after the turbo has pumped to $< 1 \times 10^{-5}$ torr. If the cryostat is at atmospheric pressure, open the cryostat valve before starting the turbo pump.

7. Pump until the cryostat pressure is $< 5 \times 10^{-5}$ torr. Close the shroud vacuum port valve. Switch off the turbo pump system and vent as described in the posted instructions.

Section 3. COOL DOWN PROCEDURE:

(NOTE: All internal power cords should already have been connected to the labeled outlets inside the rack cabinet or on the cryostat control box. One or two power cords extending from the top of the cabinet or from the control box are to be connected to a normal electrical outlet.)

1. Make sure both the cryostat and the transfer line are pumped to < 5 x 10^{-5} torr before use. This normally requires 3-6 hours of pumping.

2. Close the cryostat sample space valve. Connect the N_2 and He polyflo lines from the gas cylinders with quick-release connectors to their ports on the cryostat rack panel. Pump out the lines to the He and N_2 cylinders by turning on the sample space vacuum pump (the switch between the He and N_2 ports on the cryostat rack panel), opening the vacuum switch valve, then opening the He and N_2 switch valves. When the vacuum gauge shows full vacuum, close all switch valves and individually flush the gas lines by opening the main cylinder valves and adjusting the pressure and flow rate until the pressure relief valve on the sample space port shows a slight flow.

3. Just before the assembly, flush the transfer line for a minimum of 30 mins with He:

   a. Disconnect the He line quick-connect polyflo tubing from the control panel and connect it to the polyflow/tygon tubing provided. Ensure that He gas is flowing. Unscrew the stainless steel protective sheath which covers the tip of the transfer line, pass the tygon tubing through the sheath and attach it over the tip, pushing it past the teflon ferule. Reattach the protective sheath. Check that there is He flowing at the end of the transfer line storage vessel leg. ***Flush for 30 mins or more***.

   b. The He flow needle valve on the storage vessel leg of the transfer line has been preadjusted to give the proper flow rate. ***PLEASE DO NOT CHANGE.***

4. ***Set up the cryostat*** (while the transfer line is purging):

   a. Position the cryostat in the hutch and temporarily anchor its base with the supplied bolts.

   b. Attach the polyflo tube from the compression fitting on the outside of the cryostat rack or control panel labeled "To Sample Space" to the sample space port on the cryostat (see Figure 1, Section 1.8).

   c. Plug in the Rh/Fe sensor/heater connection (10-pin plug) and the Hastings vacuum gauge sensor connection.

   d. Turn on the temperature controller (the switch on cryostat rack panel, and ON button on controller).

   e. Place a sample rod with an empty sample holder in the cryostat, clamp on, open the sample space port valve, then alternately pump and flush (with He) the sample space at least five times. Leave the sample space under vacuum and the sample space port valve closed. Do a rough cryostat alignment in the beam.

5. ***Initiate LHe transfer***. Two methods are described here- the original method based on Oxford Instruments instructions and a modification of the original method employing a temporary pressurization of the supply dewar. The brief pressurization accelerates the process somewhat and appears to have a lower incidence of blocked lines.

Initiate LHe transfer - Method 1 (old method; note that this procedure requires at least two persons):

a. Connect the (3/8") polyflo from "Cryostat" port on the top of the cabinet (or from the “cryostat” port on the flow controller) to the barbed return port on top of the transfer line.

b. Open the needle valve on the gas flow controller several turns.

c. Make sure that the LHe transfer line has a nut and teflon ferrule of the proper dimension. Wearing gloves and eye protection, insert the storage vessel leg of the transfer line into the storage vessel part way and anchor with the dewar nut.

d. Detach the tygon tubing from the delivery leg of the transfer line. ***Make sure that the white teflon ferule stays on the transfer line.*** If the protective tip is wider than the tip of the transfer line tube, remove the protective tip from the end of the delivery leg of the transfer line.

f. Remove the plug from the siphon arm of the cryostat. Insert the delivery leg of the transfer line into the siphon arm of the cryostat. Note: on some beam lines this will require unbolting the cryostat and holding it up at an appropriate angle. ***Take care not to damage the tip, bend the delivery leg or turn the flexible transfer line through a tight radius bend.*** Tighten the coupling nut all the way, then back it off four complete turns. If the cryostat had to be unbolted, put the cryostat in place and secure the cryostat base to the sample positioner.

g. Turn on the gas diaphragm pump (the switch on the cryostat rack panel) as soon as possible after inserting the transfer line into the cryostat.

h. Loosen the dewar nut on the storage vessel and SLOWLY lower the storage vessel leg of the transfer line all the way into the LHe dewar, then pull it up ~1". Retighten the dewar nut.

i. Support the transfer line by using the provided sling, tying it to some suitable overhead support.

j. Initially, the vacuum gauge on the gas flow controller will indicate *ca*. -22 in. Hg vacuum and the flow will be very slight. Within 10-20 minutes, the flow will increase and the pressure will rise. At this point, LHe transfer has started.

k. As soon as the flow increases (as seen on the gas flow controller flow meter), tighten the coupling nut on the siphon arm of the the cryostat all the way. This directs the LHe flow into the cryostat heat exchanger. The gas flow controller will indicate *ca*. -17 in. Hg vacuum and the flow will be *ca.* 1.0 - 1.5 liter/hour.

l. Within 10 minutes, the temperature should start to drop. It may take as long as a few hours to reach the base temperature (*ca.* 4 K). During this cool-down period, it is useful to check the tightness of the delivery leg coupling nut once in a while to make sure it is sealed.

Initiate LHe transfer - Method 2 (note that this procedure requires at least two persons):

a. Connect the (3/8") polyflo from "Cryostat" port on the top of the cabinet (or from the “cryostat” port on the flow controller) to the barbed return port on top of the transfer line.

b. Open the needle valve on the gas flow controller several turns.

c. Close the Fine pressure relief valve on the He dewar (see Figure 4, Section 10.2).

d. Detach the tygon tubing from the delivery leg of the transfer line. ***Make sure that the white teflon ferule stays on the transfer line.*** If the protective tip is wider than the tip of the transfer line tube, remove the protective tip from the end of the delivery leg of the transfer line.

e. Make sure that the LHe transfer line has a nut and teflon ferrule of the proper dimension. Wearing gloves and eye protection, insert the storage vessel leg of the transfer line into the storage vessel part way and anchor with the dewar nut. Continue to slowly insert the transfer line until the pressure in the dewar begins to rise. The coarse pressure relief will release at ~10 psi, anything above 5 psi should be enough to initiate He transfer.

f. He gas should be noticeably flowing from the tip of the transfer line, increasing to a cold stream of gas within ~10 min. At any point the transfer line can be inserted into the cryostat, but good flow can be visually confirmed by waiting for an increase in flow of cold gas. Once a higher flow of cold gas begins, the transfer line should be quickly inserted into cryostat.

g. Remove the plug from the siphon arm of the cryostat. Insert the delivery leg of the transfer line into the siphon arm of the cryostat. Note: on some beam lines this will require unbolting the cryostat and holding it up at an appropriate angle. ***Take care not to damage the tip, bend the delivery leg or turn the flexible transfer line through a tight radius bend.*** Tighten the coupling nut all the way. If the cryostat had to be unbolted, put the cryostat in place and secure the cryostat base to the sample positioner.

h. Turn on the gas diaphragm pump (the switch on the cryostat rack panel) as soon as possible after inserting the transfer line into the cryostat.

i. Loosen the dewar nut on the storage vessel and VERY SLOWLY lower the storage vessel leg of the transfer line all the way into the LHe dewar, then pull up ~1". Retighten the dewar nut.

j. Support the transfer line by using the provided sling, tying it to some suitable overhead support.

k. Depending on how long one waits before inserting the transfer line into the cryostat, the vacuum gauge on the gas flow controller may indicate *ca*. -22 in. Hg vacuum and the flow will be very slight, but within about 10 minutes, the flow will increase and the pressure will rise. The temperature should start to slowly drop after the flow increases.

l. ***Slowly re-open the Fine pressure relief valve*** on the LHe dewar to release the built up pressure used to initiate transfer - higher pressure is unnecessary for normal running and may result in excess LHe consumption.

m. It may take as long as a few hours to reach the base temperature (*ca.* 4 K). During this cool-down period, it is useful to check the tightness of the delivery leg coupling nut once in a while to make sure it is sealed.

Section 4. CHANGING SAMPLE:

The cryostats are frequently used for very dilute samples that are measured in fluorescence mode. Any contamination inside the cryostat could potentially be disastrous for these experiments. Please ensure that your samples are clean on the outside, and that there is no possibility of sample leakage, in particular for solid powder samples.

1. Prechill the new sample holder in LN_2.

2. If the temperature is below 80 K, He must be used as the sample space gas. If the temperature is above 80 K, N_2 is recommended, since the aluminized mylar windows are permeable to He which can spoil the cryostat vacuum.

3. Open the appropriate gas switch valve on the cryostat rack panel.

4. Check that excess gas is flowing out of the relief valve on the cryostat sample space port.

5. Open the sample space port valve. The relief valve will momentarily cease exhausting excess gas - wait for the exhaust to resume.

6. Remove the clamp from the cryostat, remove the sample rod, immediately insert the next sample rod (or insert the dummy probe), and reattach the clamp.

7. Close the gas switch valve, open the vacuum switch valve, and pump on the sample space for at least one minute.

8. Pump/flush the sample space at least five cycles.

9. Leave gas flowing out of the relief valve, close the sample space port valve, then close the gas toggle valve on the cryostat rack panel.

If the sample rod is stuck in the cryostat and cannot be removed - DO NOT USE FORCE - see Section 9.1 for instructions

Section 5. CHANGING TEMPERATURE:

5.1. Increasing Temperature:

1. Cap the Ge detector window if this detector is in use.

2. Put the sample space under vacuum by opening the vacuum toggle switch for the sample space on the rack, and the sample port valve. This is necessary as the sample space gas will expand when the temperature is increased, and thus might create too high a pressure on the windows.

3. Make sure the turbo pump is pumping on the cryostat vacuum space.

4. Decrease the LHe flow by closing the flow meter (turn clockwise) so that the lead float barely moves. ***DO NOT CLOSE THE FLOW ENTIRELY***.

5. If the rise in temperature is not fast enough, switch the heater to Manual mode, and turn on to <15W heater. Heater wattage is displayed when you hold down the MAN button.

6. As the desired temperature is approached, turn off the heater and gradually increase the flow of LHe to stabilize the temperature.

7. When the temperature is beginning to stabilize, close the vacuum toggle switch and fill the sample compartment with He (if the temperature is <80 K) or N_2 (if the temperature is >80 K).

8. ***Note*** that at low temperatures the cryostat is quite stable in Manual mode - *i.e.* without the heater turned on to finely control the temperature. However, at higher temperatures, >150 K, the required flow is quite low, and thus the temperature stability is less reliable. It might therefore be necessary to run the temperature controller in Auto mode at higher temperatures.

5.2. Decreasing the temperature:

1. Put the sample space under vacuum by opening the vacuum toggle switch for the sample space on the rack, and the sample port valve. ***THIS IS AN ABSOLUTE NECESSITY IF YOU HAVE BEEN RUNNING WITH*** N_2 ***IN THE SAMPLE SPACE AND ARE MOVING TO A TEMPERATURE THAT IS BELOW 80 K.*** Then close the sample port valve to prevent backstreaming from the sample space vacuum pump.

2. If you have been running the temperature controller in Auto mode, change to Manual mode and turn the heater to zero output, or change to the new setpoint. It generally works better and is more economical to manually stabilize at the new temperature, then lock it in with the temperature controller Auto mode.

3. Increase the LHe flow if necessary by opening the needle valve on the flowmeter (turn counter clockwise).

4. As the desired temperature is approached, adjust the flow of LHe to stabilize the temperature.

5. When the temperature is beginning to stabilize, open the sample port valve again, close the vacuum toggle switch and fill the sample compartment with He (if the temperature is <80 K) or N_2 (if the temperature is >80 K).

6. ***Note*** that at low temperatures the cryostat is quite stable in Manual mode - *i.e.* without the heater turned on to finely control the temperature. However, at higher temperatures, >150 K, the required flow is quite low, and thus the temperature stability is less reliable. It might therefore be necessary to run the temperature controller in Auto mode at higher temperatures.

Section 6. CHANGING SAMPLE PROBE ANGLE:

The sample probe can be rotated 360° by turning the red/black knob at the sample space opening. The angle is read off on the dial on top of the cryostat. The probe can be inserted in only one direction, fixed via a pin on the probe located in a slot at the sample space opening.

Due to the fact that the end of the sample rod consists of a threaded part which might become unscrewed while turning, the following procedure should be followed when changing the sample probe angle:

1. Remove the sample and insert the dummy probe (see Section 4 for instructions). Pump/flush with the sample space gas and leave the space with He flowing and the sample port valve open so that there is a continuous flow of He to the sample space.

2. Rotate to the desired angle by turning the red/black knob.

3. Remove the dummy probe and insert the sample rod.

4. If this procedure is not taking place during sample changeover, ***and*** the sample cannot be removed and reinserted, increase the He flow to the sample space with the sample space valve open, lift the sample rod up by ~2" and rotate to the appropriate angle.

5. Pump/flush the sample space gas several times before sealing the space up with the proper gas.

Section 7. EXCHANGING DEWARS:

Please contact SMB/XAS staff for help in changing dewars

7.1. Standard Method:

1. Pump on the sample space (open vacuum toggle switch and then open the sample space port valve).

2. Position the empty and full dewars next to one another, and away from obstructing cable trays, *etc.* overhead.

3. Switch off the gas diaphragm pump (the switch on cryostat rack panel), and wait 15-20 secs.

4. Wearing thick gloves and eye protection, extract the storage vessel leg from the empty dewar, ***make sure that the brass nut and teflon ferule stay on the leg***, and ***QUICKLY*** insert the leg into the full dewar while wiping the storage vessel leg with a paper towel.

5. Tighten the nut and switch on the gas diaphragm pump (the switch on the cryostat rack panel).

6. The flow should restart within a couple of minutes. Let flow at maximum rate for a couple of minutes and then reduce to the desired flow.

7.2. Blocked Transfer Line:

1. Remove the sample rod and replace it with the dummy probe.

2. Pump/flush the sample space a few times with He/N_2 (depending upon the temperature) and leave the sample space under vacuum.

3. Close the sample space port valve and the shroud vacuum valve.

4. Depending on beam line, at this point it might be necessary to entirely disconnect everything from the cryostat, *i.e.* close the vacuum port, turn off the turbo pump, disconnect the metal hose, turn off the temperature controller and vacuum gauge and disconnect cables.

5. Switch off the gas diaphragm pump (the switch on cryostat rack panel), loosen the coupling nut and (wearing gloves and eye protection) extract the delivery leg of the transfer line from the siphon arm, taking care not to damage the delivery leg or overbend the transfer line. Place the black plug in the siphon arm.

6. Remove the polyflo tubing from the barb on top of the leg of the transfer line.

7. Wearing thick gloves and eye protection, extract the storage vessel leg from the empty dewar and ***close the valve on the dewar***.

8. Connect warm He gas flow to the delivery leg of the transfer line by attaching the tygon end of the provided tubing to the delivery leg and connecting to the quick-connect of the polyflo from the He gas cylinder (at the cabinet). Cover the delivery leg with the protective tube sheath.

9. Wait until the transfer leg is at room temperature and has been flushed for a minimum of 30 min. then follow the cool-down procedure (see section 3) from step 5 forward.

Section 8. SHUTDOWN PROCEDURE:

1. Remove the sample rod and replace it with the dummy probe.

2. Bring the temperature up to above 90 K while pumping on the sample space (open the vacuum toggle switch and open the sample space port valve). Also make sure the turbo pump is pumping on the vacuum shroud (check that the shroud vacuum needle valve is open).

3. Close the sample space port valve and the shroud vacuum valve.

4. If the cryostat needs to be unbolted to allow removal of the transfer line, disconnect everything from the cryostat, *i.e*. close the vacuum port, turn off the turbo pump, disconnect the metal hose, turn off the temperature controller and vacuum gauge, and disconnect the cables.

5. Turn off the gas diaphragm pump (switch on the cryostat rack panel).

6. Place the temperature controller on MANUAL and adjust the heater output to zero.

7. Wearing thick gloves and eye protection, disconnect the nut that holds the delivery leg of transfer line to the siphon arm of cryostat. Pull out the delivery leg and insert the black plug into the siphon arm, taking care not to damage the tip, leg or overbend the transfer line. Unbolt the cryostat if necessary for removal of the transfer line.

8. Disconnect the polyflo from the barbed return port at the top of the leg of the transfer line. Extract the storage vessel leg of the transfer line from the storage vessel. (Be sure to wear gloves and safety glasses.)

9. Attach the tygon end of the provided tubing to the end of the delivery leg to flush with He gas (from the He gas cylinder via the quick-connect). Flush until the transfer line has warmed to room temperature.

10. Secure the cryostat in place if it was unbolted earlier.

11. Let the cryostat warm up while pumping on both the sample space and the cryostat vacuum. Occasionally, if possible, fill the sample port with N_2 and then pump again.

12. When the cryostat has warmed up to room temperature, close all valves leaving the sample space and vacuum space under vacuum. Switch off the vacuum pump, the turbo pump, the temperature controller and the Hastings vacuum gauge. Disconnect the polyflo from the sample space port, the vacuum hose from the vacuum port, the 10-pin sensor/heater connection and the Hastings vacuum gauge connection from the top of the cryostat. Cap all ports.

Section 9. TROUBLESHOOTING:

9.1. Sample probe is stuck:

If the sample probe will not come out easily when pulling it up, ***DO NOT USE FORCE***. It most likely means that there is frozen N_2 and O_2 around the sample probe Cu disk. This needs to be removed by heating the cryostat up to >80 K. Use the following procedure.

1. Push the rod back down and secure with the clamp.

2. Put the sample space under vacuum by opening the vacuum toggle valve on the cabinet and leaving the sample port valve open.

3. ***CAP THE GE DETECTOR IF IT IS USED, AND BACK IT AWAY FROM THE CRYOSTAT***.

4. Decrease the LHe flow by closing the flow meter (turn clockwise) so that the lead barely moves. ***DO NOT CLOSE THE FLOW ENTIRELY***.

5. If the rise in temperature is not fast enough, switch the heater to manual mode, and turn on to <15W heater power. Avoid heating too quickly as expansion of trapped gas could pop the cryostat windows. To help accelerate the process periodically (every 1-5 min) backfill with warm He gas, then put the sample space back under vacuum.

6. As the temperature approaches 60-80 K, there will be a small but noticeable increase in the reading on the sample port gauge.

7. If there is significant condensed N_2 in the cryostat sample space, the temperature rise may seem to stall at ~70 K while the LN_2 boils off. Continue heating until the cryostat temperature is >85 K to ensure that all condensed gases have been removed.

8. Fill the sample space with He, make sure there is an overpressure by checking the gas relief valve at the sample space port.

9. Remove the rod and insert the dummy probe.

10. Pump/flush the sample space several times.

11. Turn off the temperature controller heater, and lower the temperature to the desired value (see Section 5), and proceed by inserting the new sample (see Section 4).

9.2. No, or low LHe flow:

This problem would typically appear after a dewar change, and indicates that there is an obstruction in the LHe transfer channel. The obstruction could either be in the transfer line itself, or in the cryostat body. To find out which, do the following:

1. Open the valve on the flow meter several turns. Open the nut on the cryostat siphon arm several

turns. If the flow increases the plug is not in the transfer arm but in the ***cryostat body***. If the flow stays low, the plug is in the ***transfer line***.

2. If the plug is in the ***transfer line***, the cryostat needs to be taken apart and the transfer line heated to room temperature and flushed with He gas for a minimum of 30. Follow the procedure for installation and cooldown to get started again (see Section 3).

3. If the plug is in the cryostat body, the cryostat needs to be brought to room temperature, the LHe channel in the cryostat flushed with dry He gas, and the whole cryostat reassembled. This takes several hours, and at this point the best solution is to switch to a different cryostat, if available.

9.3. High LHe flow and still high temperature:

1. Check that the temperature controller heater is off.

2. Check that the nut on the siphon arm is tightened.

3. Check if the transfer arm outer surface is cold, particularly close to the cryostat. If it is, the vacuum in the outermost jacket could have become degraded. This should be addressed by pumping on the transfer line with a turbo pump.

4. ***Contact a SMB/XAS staff person for this operation!***

5. Attach the flex hose from the turbo pump to the vacuum valve on the transfer line, at the LHe dewar (see Figure 2, Section 1.8). Leave the valve closed.

6. Start the turbo pump according to the instruction sheet on the turbo pump cart - make sure that the pumped space is open up to the transfer line vacuum valve (but that this valve is closed).

7. When the pressure of the turbo pump gauge shows $<10^{-6}$ torr, open the transfer line vacuum valve slowly as not to create a potential overpressure for the turbo pump.

8. Pump until the pressure is in the mid 10^{-6} torr range.

9. Close the transfer line vacuum valve.

10. Close down the turbo pump system as described on the instruction sheet on the turbo pump cart.

9.4. Moisture or frost on the cryostat window:

This will appear if the vacuum of the insulating cryostat vacuum space has been degraded.

This can happen as a result of He slowly diffusing through the Kapton window from the sample space into the vacuum space. It would normally appear if the space has not been dynamically pumped (opened to the turbo pump all the time), and has not been pumped out regularly (about once ever 24 hours). If so:

1. Start the turbo pump (see instruction sheet on the pump cart) and wait until the pressure is below 10^{-5} torr.

2. Slowly open the vacuum shroud valve on the cryostat body (do not create an overpressure for the turbo pump) and pump until the pressure is in the low 10^{-5} or in the 10^{-6} torr range.

3. Carefully wipe off the windows with a tissue.

Moisture/frost on the windows can also appear if there is a leak, either in the innermost shroud window, or in the indium seal. In this case the cryostat needs to be taken to room temperature and the window/seal replaced. ***Please contact a SMB/XAS staff person.***

9.5. Lost sample in the cryostat:

In many cases the sample can be left in the bottom of the cryostat until the end of a user's beamtime and is preferred as the grabber tool can puncture the cryostat window, resulting in significant loss of beamtime to cryostat replacement. A blank scan should be run to assess whether the lost sample is contributing to the fluorescence signal and needs immediate removal. If necessary, to remove the sample do the following:

1. Open the toggle valve on the control cabinet for the sample port for the appropriate gas. Check that excess gas is flowing out of the relief valve on the cryostat sample space port.

2. Open the sample port valve on the cryostat. Increase the flow of gas by adjusting the flow meter on the gas cylinder.

3. Get the sample out by inserting a "grabber tool" to the bottom of the cryostat, expanding its prongs, fishing around and closing the prongs.

4. When the sample is out, reduce the flow of gas by adjusting the flow meter on the gas cylinder to its old value.

5. Insert the dummy rod, put on the clamp and pump/flush the sample space at minimum 5 times.

6. Proceed to the next sample.

Section 10. LHe DEWARS INFORMATION:

10.1. Valve Overview and Precautions:

Currently all LHe dewars at SSRL are delivered from an outside vendor. The size and detailed construction of the dewars vary to some extent, but the principal construction is the same. It is of great importance that the user be familiar with the various valves on the dewar, and which valves should be left open/closed at what time, as incorrect handling potentially can cause a severe hazard. The following section therefore describes schematically the dewars and the necessary procedures.

As an ***absolute*** rule - ***ALWAYS WEAR THICK GLOVES AND SAFETY GLASSES WHEN WORKING WITH THE LHe DEWAR***.

FOR ALL VALVES, THE VALVE IS **OPEN** ***WHEN THE HANDLE IS*** **PARALLEL** ***TO THE RELATED TUBES, AND*** **CLOSED** ***WHEN*** **PERPENDICULAR** ***TO THE RELATED TUBES.***

The dewar has a total of five ports, (see Figure 4, Section 10.2):

- Pressure gauge, which is preceded by a small pressure relief valve.
- Coarse pressure relief valve. This valve will open if the pressure becomes very high, and after the fine pressure relief has already opened.
- Fine pressure relief valve with a manual valve on it. This valve can be briefly closed during cooldown to facilitate He transfer, but ***at all other times should be open*** as it is the initial safety valve in that it has the lowest pressure requirement to open. The Oxford system does not require a pressurized dewar to operate.
- Fill valve for the dewar- ***SHOULD ALWAYS BE CLOSED***. If this valve is left open, air will pull into the dewar and cause an ice plug which could be very dangerous. However, if during installation of the cryostat there is significant overpressure, as judged by the release of He through the fine and/or coarse pressure relief valves the fill valve can temporarily be opened to release overpressure.
- Top port through which the transfer line vessel leg is inserted. It should ***always be closed*** when the dewar is not in use as otherwise an ice plug can be formed.

Before the installation of the cryostat is started, the valve for the top port should be opened to release any overpressure of the dewar. As cold gas will be expelled through this port immediately upon opening the valve, make sure that hands and face are protected.

10.2. L-He Dewar Figure:

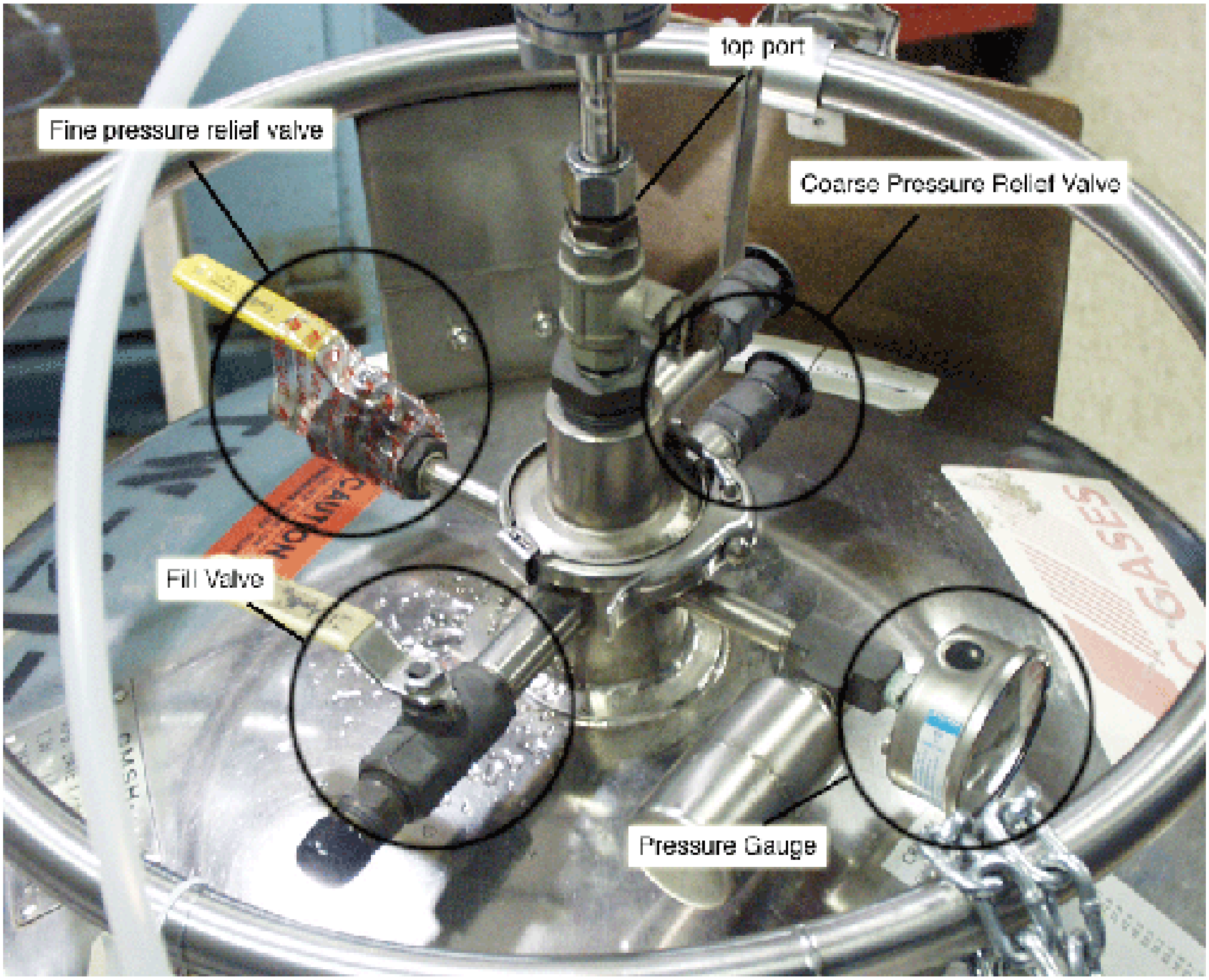

Figure 4: L-He Dewar