

Cell 12 Top Loading Cryostat Operating Procedures

Probe Loading

1. After mounting the sample, pull the load lock completely down and tighten probe clamp securely on probe shaft just above the load lock, see **Photo 4**.

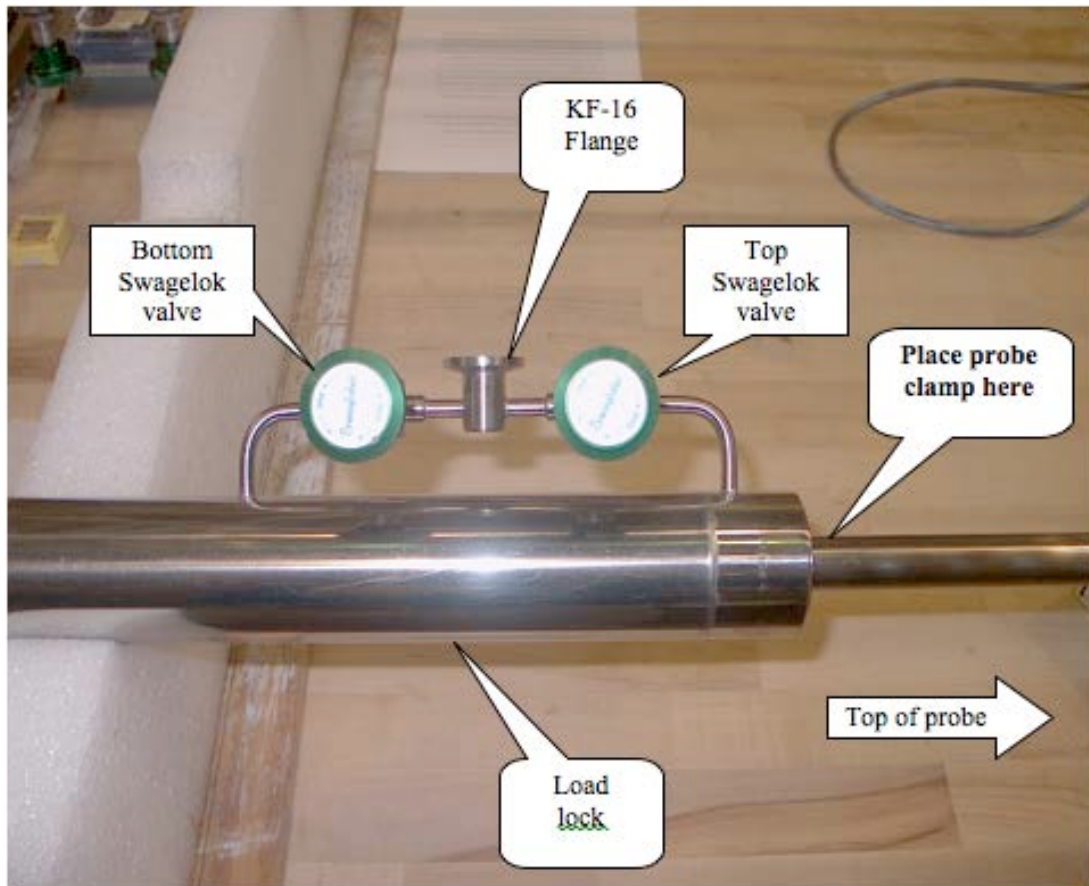


Photo 4

2. Attach the strain relief for the blue instrumentation cables on the head of the probe. This will strain relieve the Fischer connectors and help prevent broken solder joints, see **Photo 5**.

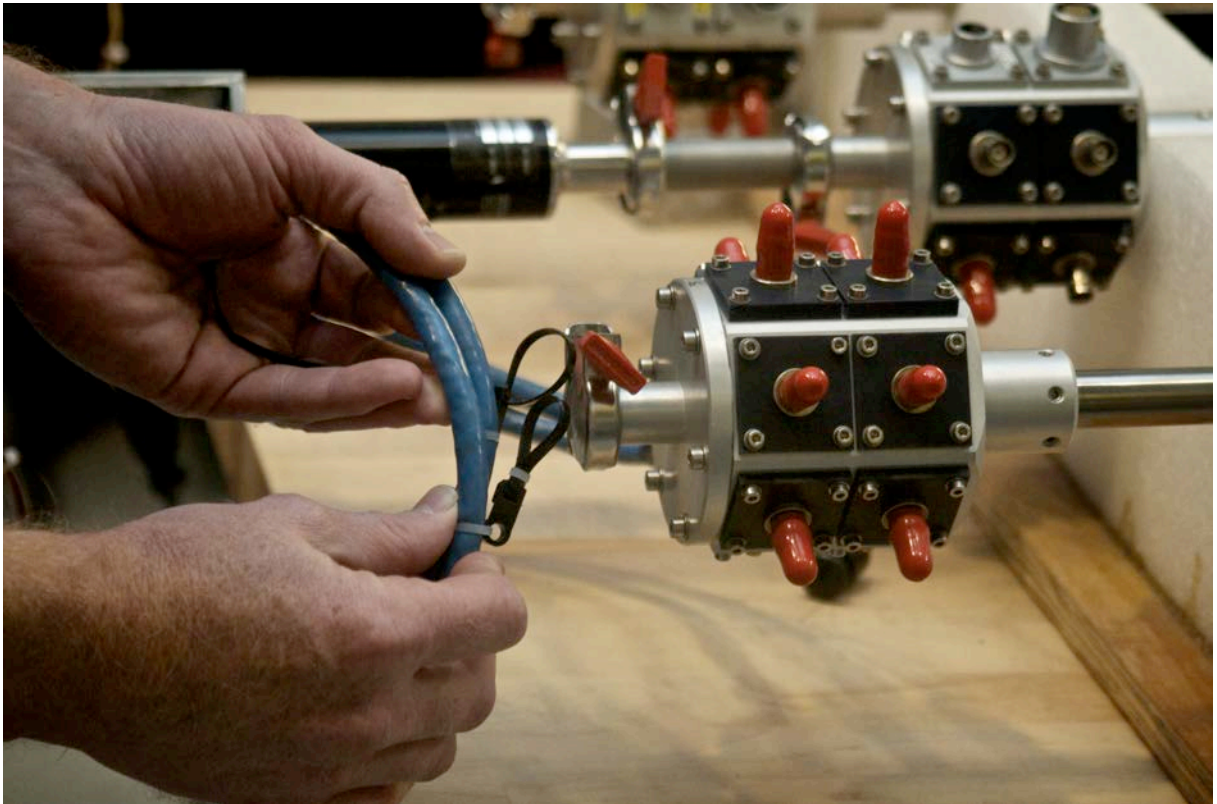


Photo 5

3. Holding the blue cables and the probe shaft in one hand and the probe load lock in the other hand carefully raise the probe up into a vertical position, see **Photo 6**.



Photo 6

4. Walk the probe up the stairs to the cryostat using one person to guide the cables over any obstacles. Once on the platform the person guiding the cables can climb up the scaffolding and hold the probe.

5. Make sure the gate valve on the cryostat is closed then remove the KF-50 clamp and blank. Mount the probe securely to the cryostat flange with a KF-50 o-ring and clamp. Using a Velcro strip, secure the blue cables to the horizontal scaffolding bar, see **Photo 7**.

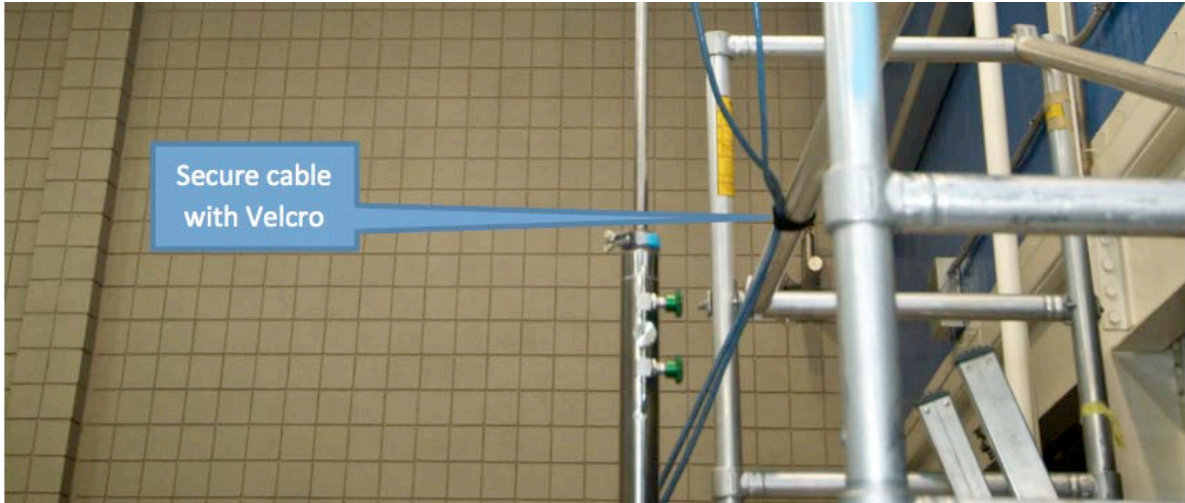


Photo 7

6. Attach the turbo pumping line to the KF-16 port on the probe load lock, open both green Swagelok valves, open the speedi-valve on the turbo pump and turn on the turbo pump.

Lowering Probe

1. Complete steps 1-6 under 'Probe loading procedure'.
2. Wait until the pressure at the turbo pump display reads below 5×10^{-3} mbar. If the pressure does not reach this level in 10 minutes or less there is most likely a leak. It may be necessary to remove the probe to find and fix the leak.
3. **CLOSE** the bottom green Swagelok valve on the probe leaving the top green Swagelok valve open.
4. Double check one more time that you closed the bottom green valve – this is VERY IMPORTANT to avoid losing ^3He gas.
5. Have someone watch the pressure on the turbo pump and then open the cryostat gate valve. If the pressure rises at the turbo pump, immediately close the gate valve. If the pressure at the turbo pump continues to drop it is okay to continue.
6. Before lowering the probe, double check the gate valve to be sure it is completely open.

7. Set the sorb temperature to 21K. This will heat the sorb enough to provide some exchange gas to cool the probe while lowering.
8. Remove the probe clamp above the load lock. Do not let go of the shaft while removing the clamp!
9. Begin lowering the probe. The Lakeshore 336 Temperature Controller channels can be watched on the Data Mac monitor using the LabView VI "Big Temp Display". As the probe is lowered watch the temperature of the 1K pot to ensure it stays below 1.8K. If the probe is lowered too fast, boiling of the helium bath may be heard via the check valve located on the back wall and the probe lowering should be stopped. It can be resumed when the boil off subsides. The probe can be lowered until a hard stop is felt. At this point the bottom of the probe head will be approximately one inch above the load lock.

Note: The probe has two sliding baffles that can occasionally bind up and prevent lowering of the probe. If this occurs, slightly raise and rotate the probe carefully until it becomes free and then to lower the probe.

Condensing ³He

1. Verify that the 1K pot and probe temperatures are below 1.8K
2. Set the sorb temperature to 40K.
3. After approximately 30 minutes the gas will be fully condensed and the sorb heater can be shut off or set to an intermediate temperature.

As the sorb cools the vapor pressure of the ³He will drop, cooling further the ³He liquid. The sorb can be cooled faster by opening the 1K-pot needle valve. The needle valve is controlled using the Oxford ITC 503 Temperature Controller. As the 1K-pot fills, the cooling rate of the sorb will slow and the needle valve will have to be gradually closed to its original position. The expected base temperature is approximately 300 mK.

The sorb is used to obtain sample space temperatures between 300mK and 1.8K. See the graph in the Appendix of this document for reference. For temperatures above 2.2K see the below paragraph. The temperature range of 1.8K to 2.2K is unstable and usually hard to control therefore it is not recommended to stay in this range.

The insert can also be run at temperatures up to 80K using the heater on the sample holder. To do this the probe heater is controlled using 'Output 2' of the Lakeshore 336. The probe breakout box heater plugs should be connected to the back of the Lakeshore 336 Temperature Controller "output 2" connection using banana plug cables, see **Photo 8**. The sorb should be set to 12K to provide some exchange gas for better temperature control and PID numbers listed in the Appendix should be entered into the loop 2 control.

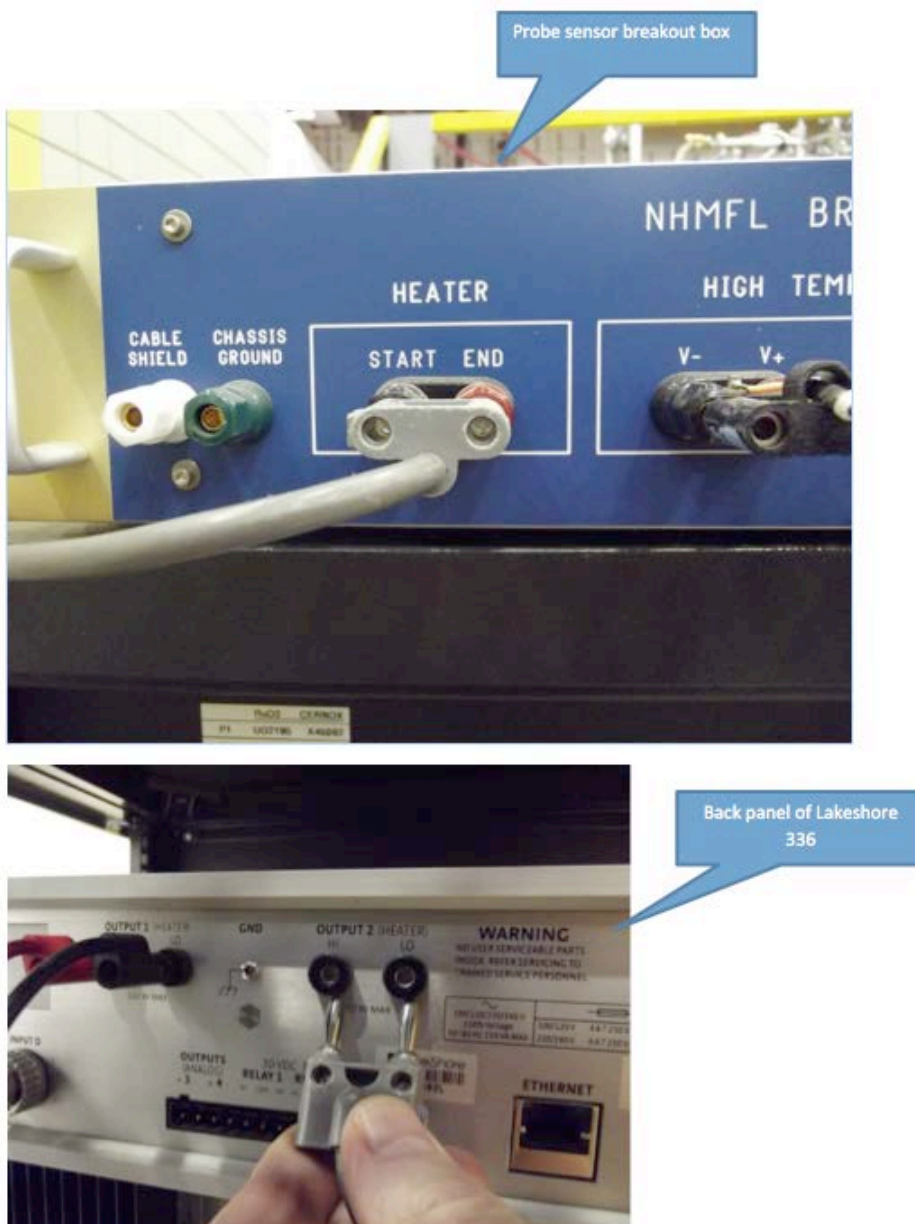


Photo 8

Needle Valve Operation Using The ITC 503 Temperature Controller

1. Using the lower ITC 503 Temperature Controller on the gas handling system push the 'Gas Flow' button and hold the button down while using the up/down arrows to adjust the needle valve. The digital display will show percentage open, see **Photo 9**.

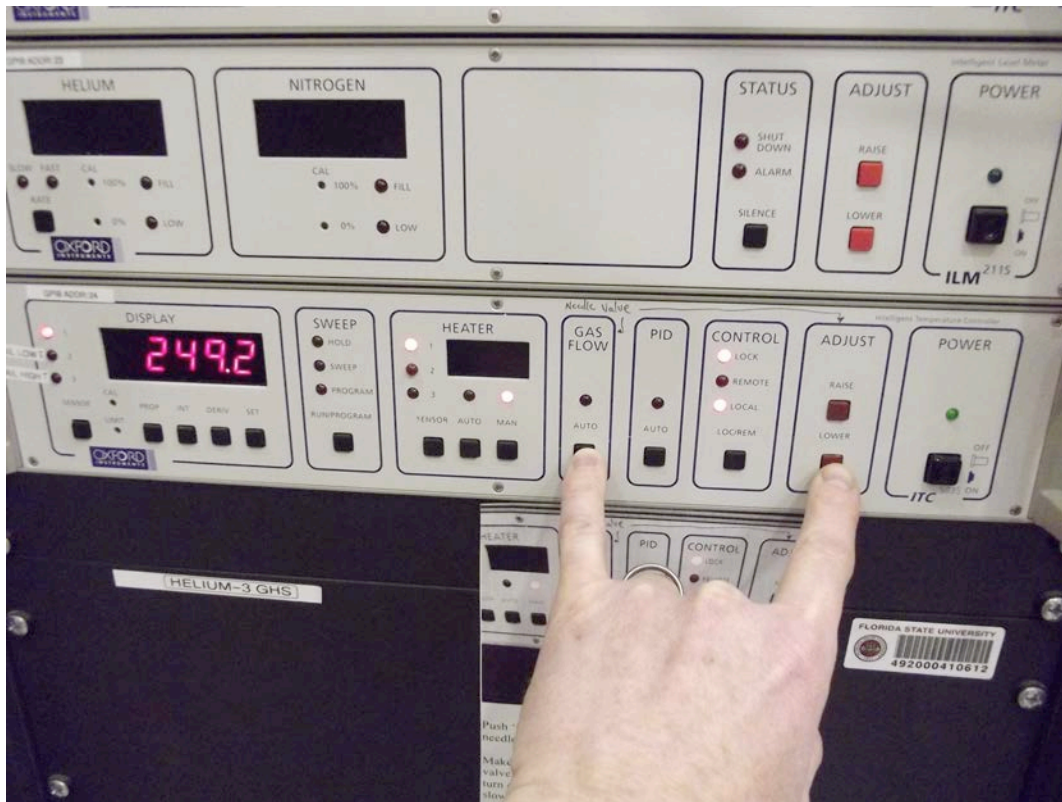


Photo 9

Note: While adjusting the valve, if the 'Auto' LED illuminates, push the 'Gas Flow' button again until the LED turns off. In the 'Auto' mode the valve will slowly open to 100%, which will result in a large consumption of liquid helium from the bath.

Removing the probe from the ^3He insert

- 1) If not already attached, connect the turbo pump line to the KF-16 flange on probe load lock (the flange between the two green handled Swagelok valves).
- 2) Check and **make sure** the lower green handled Swagelok valve on the probe load lock is closed and open the upper green handled load lock Swagelok valve.
- 3) Open the valve on the turbo pump and turn the pump on, the minimum pressure required before raising the probe is 5.0×10^{-3} mbar.
- 4) Set the Sorb temperature to 21K and turn the heater range to 'High'. This will provide a small amount of exchange gas from the sorb which will help warm the probe as it is raised.
- 5) Begin raising the probe once the pressure reaches 5.0×10^{-3} mbar. When the probe shaft feels cool stop raising and put the clamp on the shaft. Wait approximately 5 minutes and begin raising again until the shaft feels cool. The point of the stop and go is to prevent freezing of the sliding oring seals around the shaft. If the orings become frozen it is possible that air will leak into the probe.
- 6) The probe will come to a positive stop once fully raised. There is also a measuring rod on the platform that can be used to double check that the probe is completely raised.

Note: There are sliding baffles on the probe that sometimes catch as the probe is raised. If this happens, carefully lower and twist the probe and pull up. You may have to repeat this up and down/twist motion to free the probe. Just go slow and be careful!

- 7) Put the clamp on the shaft and do nothing further until the probe temperature reaches 285K. The Sorb should still be at 21K which as mentioned previously is helping to warm the probe

- 8) Once the probe reaches 285K turn off the Sorb heater and wait until the sorb cools below 4K. the Sorb can be cooled faster by opening the 1K pot needle valve until the pressure on the 1K pot reaches 20-25mbar. If the pressure rises above this for more than a few minutes reduce the needle valve opening until it starts to drop. Too much liquid in the 1K pot will slow the cooling of the Sorb.

- 9) When the Sorb is below 4K close the gate valve on the insert. (This is the valve that the probe is mounted to). Close the upper green handled Swagelok valve, close the valve on the turbo pump and remove the pumping line from the probe.

- 10) Using TWO people, the probe can now be removed from the cryostat and carefully walked down to the probe table.

- 11) Cap off the gate valve with a KF-50 blank.

VTI Operating Procedure

1. Complete steps 1-6 under 'Probe loading procedure' on page 3 of this manual.
Wait until the pressure at the turbo reads below 5×10^{-3} mbar. If the pressure does not reach this level in 10 minutes or less there is most likely a leak. It may be necessary to remove the probe, leak check, and repeat steps 1 through 6.
2. Once the pressure is 5×10^{-3} mbar on the turbo pump, close the bottom green Swagelok valve on the probe leaving the top green Swagelok valve open, double check that you closed the bottom green valve. The probe can be lowered before setting the temperature of the VTI or it can be set ahead of time. Before lowering, double-check the gate valve to be sure it is completely open.
3. Set the temperature of the VTI. The temperature of the VTI can be set anywhere between 1.5 and 325K. Using the Lakeshore 336 Temperature Controller enter a set point and heater range. It is best to use the lowest heat range that will stabilize the system. See page 8 for Lakeshore 336 operation.

The needle valve may have to be adjusted depending on the temperature set. See 'Needle valve operation using the ITC 503' on page 4 for details.

NOTE: If the VTI is used at temperatures above 80K it can take up to an hour or more to cool back down to 1.5K due to the conductance of the capillaries feeding the evaporator decreasing with higher temperature.

Lakeshore 336 Temperature Controller Operation

The 336-temperature controller is a straightforward user-friendly controller. Operational details can be found in the manual located in the OP-128 Instrument Shop of the NHMFL. Below is an example of setting the Sorb temperature of the ^3He insert:

- 1) Make sure controller is in 'Local' mode

- 2) Push Channel 'A'

- 3) Push 'Set Point' and enter '40' using the numeric pad then 'Enter'

- 4) Push 'Heater Range' and use the up/down arrows on the right side of the controller to select the High range, press 'Enter' The Sorb heater should turn on after pressing 'Enter'.

To shut the Sorb heater off push channel 'A', press 'Heater Range' and using up/down arrows set the range to Off, or press the red 'All Off' button, which will shut off both heater outputs of the Lakeshore controller.

Note: The 'All Off' button can be pressed at any point during operation to shut off all outputs.

Removing the probe from the VTI insert

- 1) If not already attached, connect the turbo pump line to the KF-16 flange on probe load lock (the flange between the two green handled Swagelok valves).
- 2) Check and make sure the lower green handled Swagelok valve on the probe load lock is closed and open the upper green handled load lock Swagelok valve.
- 3) Open the valve on the turbo pump and turn the pump on, the minimum pressure required before raising the probe is 5.0×10^{-3} mbar.
- 4) Begin raising the probe once the pressure reaches 5.0×10^{-3} mbar. When the probe shaft feels cool stop raising and put the clamp on the shaft. Wait approximately 5 minutes and begin raising again until the shaft feels cool. The point of the stop and go is to prevent freezing of the sliding oring seals around the shaft. If the orings become frozen it is possible that air will leak into the probe.
- 5) The probe will come to a positive stop once fully raised. There is also a measuring rod on the platform that can be used to double check that the probe is completely raised.

Note: There are sliding baffles on the probe that can catch as the probe is raised. If this happens, carefully lower and twist the probe and pull up. You may have to repeat this up and down/twist motion to free the probe. There may be a metal on metal clinking sound when the baffle becomes free. Just go slow and be careful!

- 6) Put the clamp on the shaft, close the gate valve that the probe is mounted to and do nothing further until the probe reaches approximately 290K.

- 7) Close the upper green handled Swagelok valve, close the valve to the turbo pump, remove the pump line and using TWO people carefully remove the probe and walk it down to the probe table.

- 8) Blank off the gate valve with a KF-50 blank.

Appendix

PID settings

VTI insert: P(80) I(12) D(0)

³He insert: P(50) I(6) D(99)

When using heater on probe set probe PID's to: P(80) I(20) D(0)

Probe temperature sensor calibration files:

Probe No.	Cernox	Ruthenium Oxide
1	X46092	UO2185
2	X46085	UO2186
3	X46087	UO2983
4	X46139	UO2156

System temperature sensor calibration files:

VTI	X46088
1K Pot	RX-202A-AA
Sorb	Curve 32 User AB###

Other useful information:

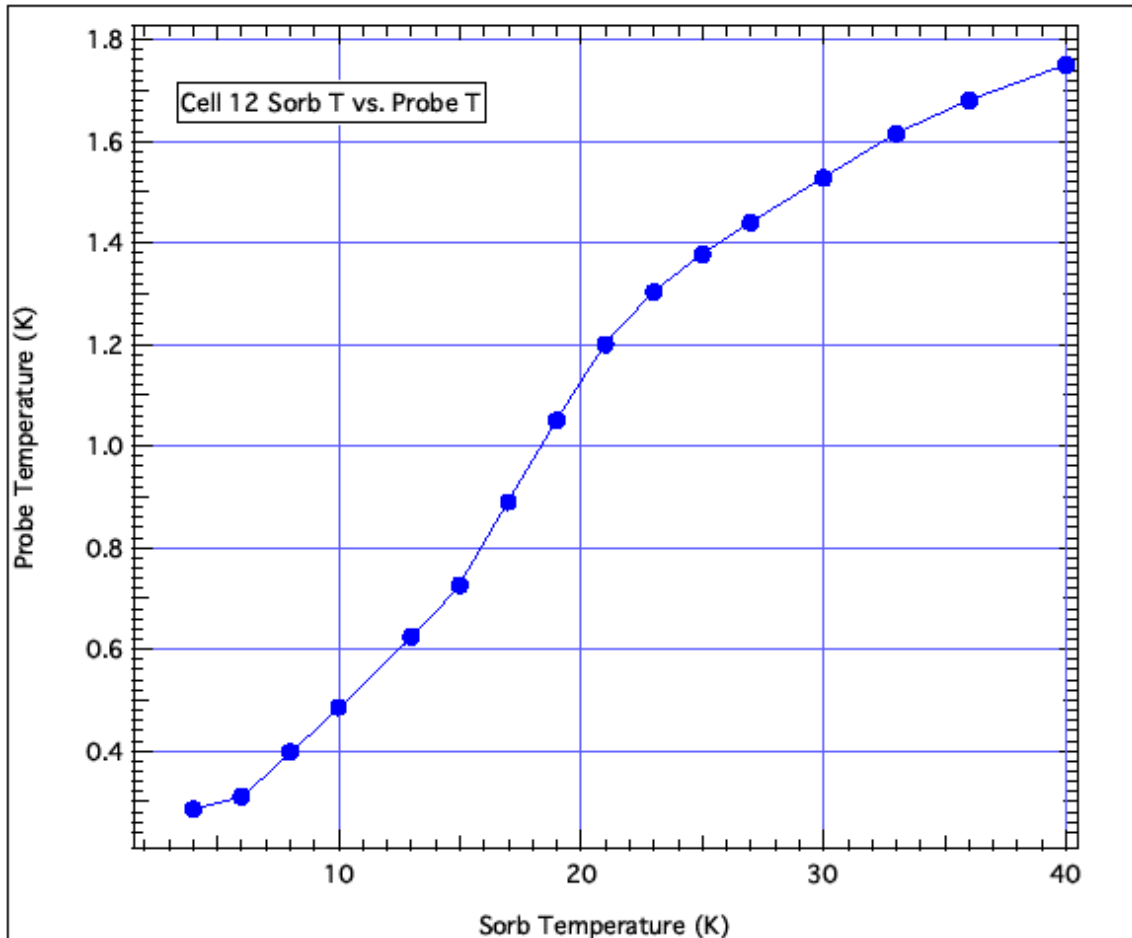
-Rotator probe: sample holder rotates 8.4 degrees per one revolution of the linear actuator knob.

-Measured insert sample spaces: VTI (0.671" / 17.04mm) ³He(0.695" / 17.65mm)

1 cm^3 of liquid ³He = 622 cm^3 ³He gas at STP

1 cm^3 of liquid ⁴He = 866 cm^3 ⁴He gas at STP

Reference graph for setting the Sorb temperature when running probe from base to 1.8K



Point	SorbTemp	ProbeTemp
0	40	1.75
1	36	1.68
2	33	1.615
3	30	1.527
4	27	1.44
5	25	1.377
6	23	1.303
7	21	1.2
8	19	1.05
9	17	0.89
10	15	0.726
11	13	0.625
12	10	0.486
13	8	0.397
14	6	0.31
15	4	0.285