5. Pump out the Omniplex vacuum space to a pressure less than 1.3 Pa (10 mTorr [10 µm]). If you are using a mechanical vacuum pump which can backstream oil, pump the minimum time necessary to achieve this pressure. Dry pumps can be run longer and the vacuum will be cleaner if the system is pumped for a day or overnight.
6. Close the vacuum valve using one of these corresponding procedures:
   - *Plug valve:* push in the Cryolab™ valve operator handle to insert the valve plug. Rotate the handle counterclockwise to unscrew the handle from the plug. Pull the handle back out. Turn off the vacuum pump and vent the vacuum hose. If desired, remove the valve operator from the valve and replace the retainer ring and dust cap.
   - *Diaphragm valve:* turn the large black knob clockwise all the way to fully close the valve. Turn off the vacuum pump and vent the vacuum hose. If desired, remove the vacuum hose.
7. Monitor the pressure on the vacuum gauge. The pressure should rise slowly and level off at a pressure less than about 13 Pa (0.1 Torr [100 µm]). If the pressure rises rapidly above this pressure level, a leak or contamination may exist. Test the system for leaks to isolate the cause of the problem, and repair it. If everything appears normal, proceed to the next step.

NOTE

Dry vacuum pumping can continue until no longer desired, but if using a mechanical pump stop pumping before starting the CCR compressor.

4.4.3.5 Cool Down

To begin the cool down, simply start the compressor. During cooldown, check for condensation on the outside of the Omniplex tail. Condensation indicates that the vacuum is not good enough and the vacuum pumpout must be repeated. The cooldown to 15 K should take about 1 h to 2 h.

4.4.3.6 Sample Insert Removal

Use the procedure below to remove a cold sample insert (FIGURE 4-9) from the Model 75014A CCRSM.

1. Unplug the triaxial BNC cables from the sample insert junction box. If desired, leave 10-pin circular connector in place to monitor the temperature.
2. Twist the sample insert junction box in the rotation stage to release the bayonet lock.
3. Remove the sample insert quickly from the Omniplex sample well.
4. Insert the sample well plug and make sure it seals tightly.
5. Press the flush-fill valve button two or three times to remove the gas (possibly contaminated with air) from the sample well.
6. Wait until the sample insert is dry before removing or mounting samples. If desired, you can blow dry it with a hot air gun, but do not overheat it.

4.5 Model 75016 Oven Sample Module (OSM) Option

The Model 75016 oven sample module (Model 75016 OSM) features and specifications are given in Chapter 1. Stable operation at a range of temperatures is made possible by balancing the cooling power provided by the ambient environment against two heater circuits powered by a temperature controller. An assembly overview is shown in FIGURE 4-13.

WARNING

The outer surface of the oven module around the sample aea can be very hot during opertion. Do not touch the oven outer surface during operation above 400 K.

The Model 75016 OSM consists of the following components:

- Oven body (FIGURE 4-14 and TABLE 4-8)
- Rotation stage
- Flush-fill unit

- Mounting base plate
- Sample insert (FIGURE 4-13)
- Power booster for a Model 340 loop 2 heater output

Also required for operation of the Model 75016 CCRSM, but not included in the option:

- Model 750TC option (only need one Model 750TC for both the CCR and OSM)
- Vacuum pump

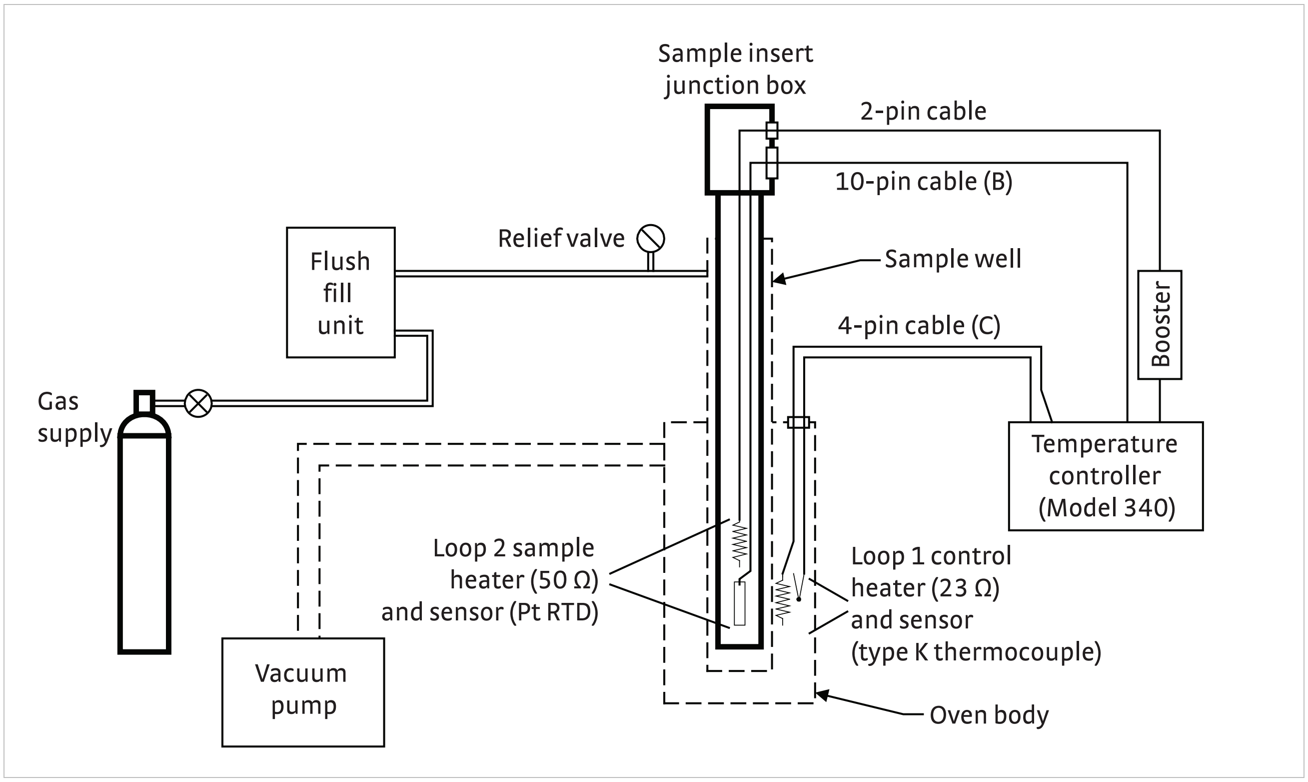


FIGURE 4-12 ***Model 75016 OSM operational schematic (the electromagnet and measurement systems are not shown)***

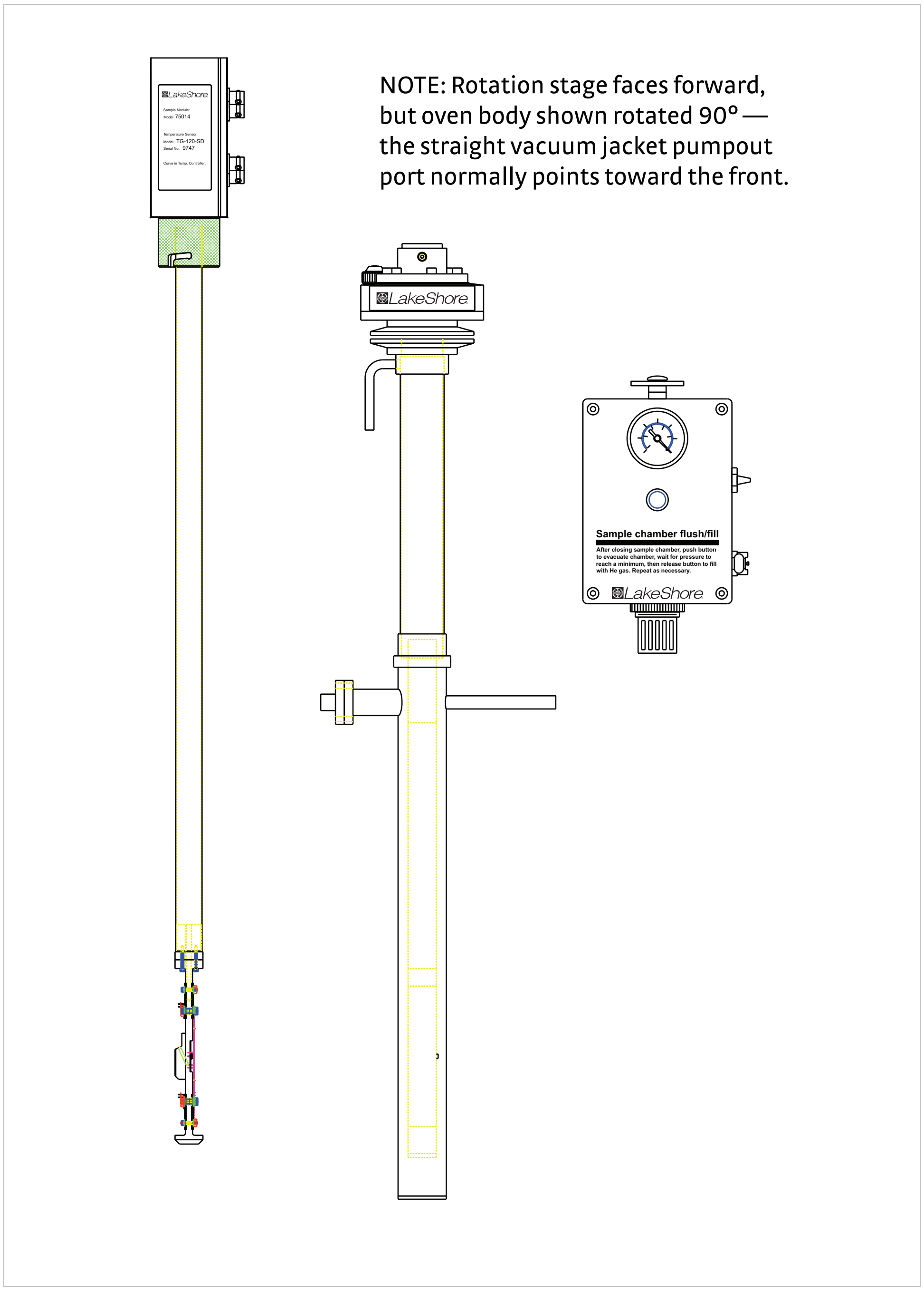


FIGURE 4-13 ***Model 75016 OSM assembly overview (view from front of electrmagnet)***

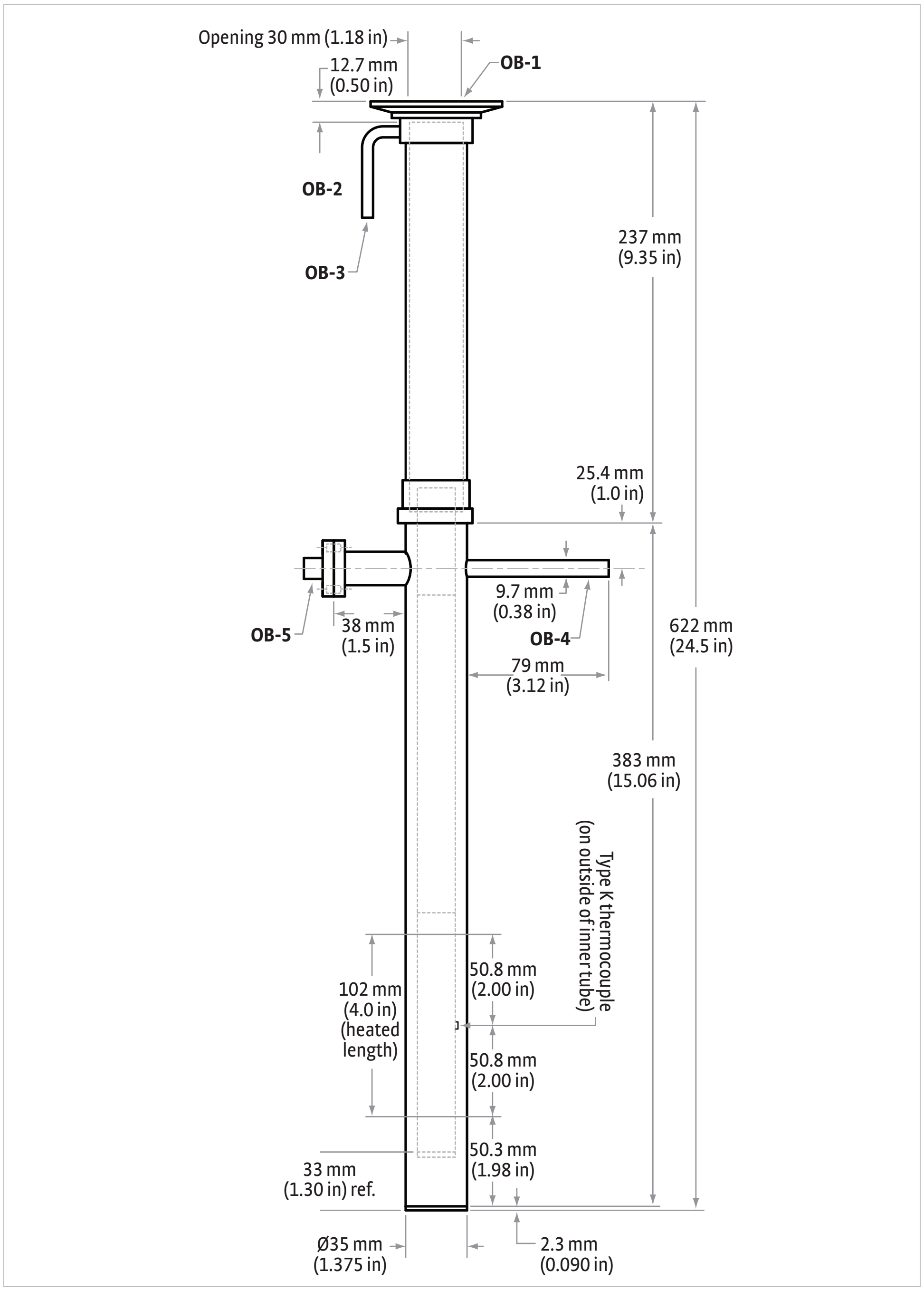


FIGURE 4-14 ***Oven body for the Model 75016 OSM***

The standard sample module has one 10-pin connector and one 4-pin connector for temperature measurement and control.

For the CE-certified system, the sample module has an additional 8-pin connector and along with the connecting cable it provides the necessary CE safety requirements. Also, an interlock switch is installed in the quarter turn bayonet of the sample module so that the unit will not operate until the sample enclosure is properly installed and all the cables are connected.

OB-*	Oven Body
1	Sample well access port, NW 50 flange, 19.1 mm (0.75 in) clear bore
2	Sample well relief valve, 28 kPa (4 psig) cracking pressure
3	Sample well exchange gas inlet, Swagelok® for 6.35 mm (¼ in) OD tube
4	Vacuum pumpout port, 9.52 mm (⅜ in) OD tube
5	Loop 1 temperature sensor and heater (23 Ω), 4-pin circular connector (red)

**The locations of the connections listed here are shown in FIGURE 4-14.*

TABLE 4-8 ***Model 75016 OSM oven body connection point and interface designations***

4.5.1 Connecting the Sample Chamber Flush-Fill Unit

Refer to section 4.5.1 to connect the sample chamber flush-fill unit properly.

4.5.2 Sample Mounting with the Model 75016 OSM

Mount samples by following the Model 75013 SCSM prober sample card sample mounting instructions given in section 4.3.1.3 with the following exceptions:

1. The sample mounting portion of the Model 75016 sample insert is permanently attached, not removable like the prober sample card used in the Model 75013 SCSM.
2. The sample mounts in the center of a sapphire mounting plate that is centered between solder posts. The solder posts around the sapphire mounting plate are not numbered, but are laid out in the same pattern and orientation as the numbered triaxial bulkhead connectors on the sample insert junction box (FIGURE 4-12). The figure on the cover of the junction box shows connections to two common sample geometries (van der Pauw square and 1-3-3-1 Hall bar).

NOTE

There is no sample identification space on the Model 75016 sample insert sample mounting plate. If desired, attach a removable note to the sample insert junction box with the sample identification and other relevant information.

4.5.3 Operation of the Model 75016 OSM

This section provides instructions for using the Model 75016 top loading oven.

WARNING

High voltage can be present at sample terminals. Always install the measurement insert into the oven before making measurements, and stop measurements before removing the module.

For the CE-certified system, an interlock switch is installed in the quarter turn bayonet so that the unit will not operate until the sample enclosure is properly installed and all the cables are connected.

4.5.3.1 Sample Insert Insertion

Use the procedure below in loading the sample insert to avoid contamination or condensation from air.

1. Clean the sample insert to remove any dust or dirt accumulated on its surface. Verify that the sample insert is dry.
2. Ready the sample module, remove the sample well plug, and carefully insert the sample insert.
3. Twist the sample insert junction box in the rotation stage to fully engage the bayonet lock.
4. Press the valve button on the flush-fill unit two or three times. This removes any air from the sample well and backfills it with argon gas.
5. Set the rotation stage to 0 ° so the sample is perpendicular to the applied magnetic field.

4.5.3.2 Sample Insert Cable Connections

1. The four or six triaxial sample cables should already have a protective sleeve plus a short section of spiral cable wrap and a tie down clamp near the sample end. Connect the triaxial sample cable to the sample module.
2. Bolt the tie down clamp into the right rear bolt hole on top of the saddles. This strain relieves the cables, but positions them close to the sample well where they will be attached to the sample insert.
3. Connect the 10-pin cable to the sample insert.
4. For the CE-certified system, connect the 8-pin connector cable to the sample module.

4.5.3.3 Vacuum Pump Out Procedure

The customer must supply a vacuum pump to evacuate the sample module to a pressure of 0.1 Pa (7x10-4 Torr) or lower prior to initial operation. Vacuum pressure gauging must be provided or added to the oven. The oven must be pumped continuously during operation.

1. The oven should be at room temperature for initial vacuum pumpout.
2. Connect the vacuum line to the oven vacuum pumpout port (OB-4).
3. The other end of the vacuum line has an NW 25 flange attached. Connect the flanged end to a vacuum pump.
4. Start the vacuum pump and pump out the oven vacuum space to a pressure less than 1 Pa ($7x10^{-3}$ Torr). If you are using a mechanical vacuum pump which can backstream oil, a trap must be used. Dry pumps can be run longer and the vacuum will be cleaner.

4.5.3.4 Sample Insert Removal

Use this procedure to remove a sample insert from the Model 75016 OSM.

1. Wait until the sample insert temperature is below 350 K before removing it.
2. Unplug the triaxial BNC cables from the sample insert junction box. If desired, leave the 10-pin circular connector in place to monitor temperature.
3. Twist the sample insert junction box in the rotation stage to release the bayonet lock.
4. Remove the sample insert from the oven sample well.
5. Insert the sample well plug and verify that it seals tightly.
6. Press the flush-fill valve button two or three times to remove the gas (possibly contaminated with air) from the sample well.

4.6 The Configuration Utility

The configuration utility shipped with the system allows for you to change hardware configurations that do not normally need changed. This section describes the use of the configurator to set up the temperature control parameters. The configurator can be run from the HMS start menu under the program section of the system start menu. All the temperature measurement option setups are similar.

4.6.1 Temperature Controller Setup

There are many parameters of the system that determine stability of the temperature control. This section allows you to change the default parameters. This section also allows you to change some default settings of the temperature controller.

FIGURE 4-15 shows the first page of the temperature options. The most useful entry on this screen is the curve number of the sample sensor. For most experiments, the sample sensor is connected to the B input of the Model 340. It is the temperature sensor mounted closest to the sample and is the control sensor for the loop 2 heater.

FIGURE 4-16 shows the second page of the controller setup. On this page, the A channel sensor is defined and the control setup and control limits for both heaters are determined. Also on this page is a check box to determine the state of the heaters when the software exits. To turn the heaters off every time the software exits, select the box. If the box is not selected, the heaters will be left on as the software exits.

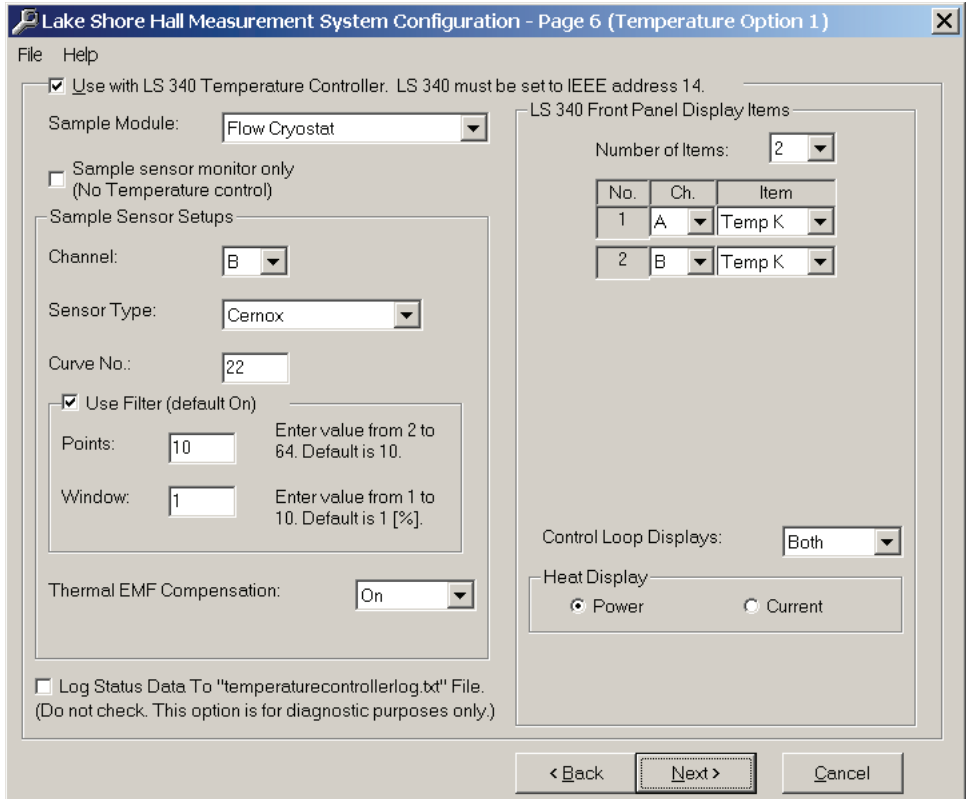


FIGURE 4-15 ***Temperature options***

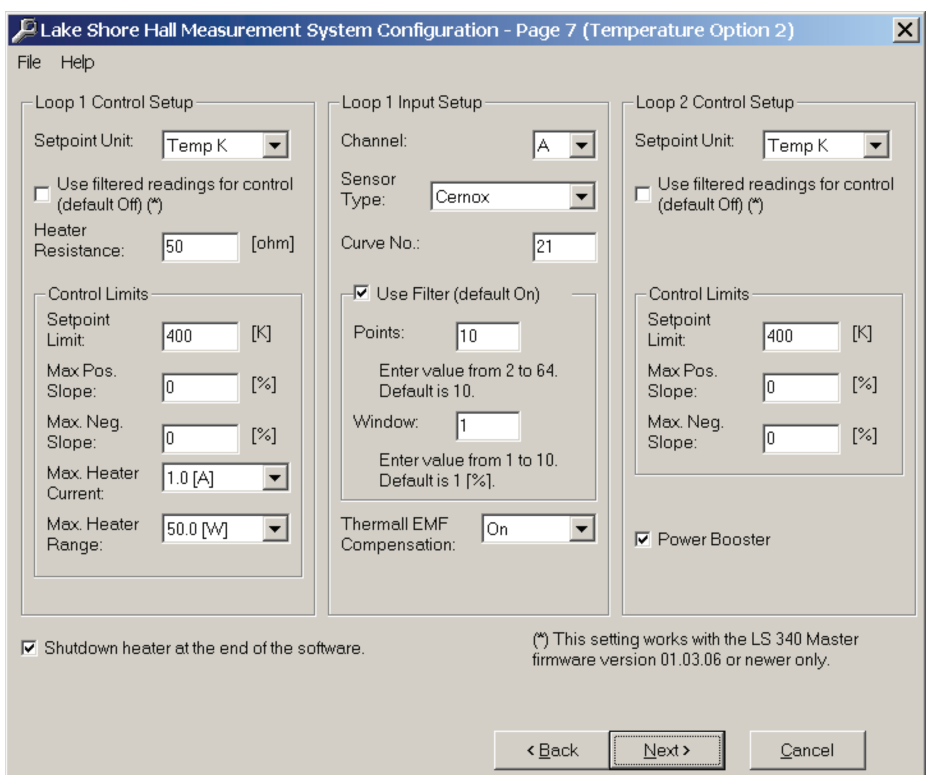


FIGURE 4-16 ***Controller setup***

4.6.2 Understanding How Temperature Settle is Determined

The HMS software is designed to provide a stable temperature environment for the samples using various temperature options and the Lake Shore Model 340 temperature controller. The software has default parameters to determine control and settle criteria. This section describes how the software defines the settle criteria. The next section shows how the users can change the parameters of the control and settle criteria.

Every standard Lake Shore temperature option for the HMS systems has two sensors and two heaters. The first heater, called the loop 1 heater, is always the higher power heater and uses one of the two sensors, called loop 1 control sensor, to control the temperature. The loop 1 control sensor is mounted near the loop 1 heater.

The second sensor, called the sample sensor, and heater, called the loop 2 heater, is mounted near the sample. This smaller heater is used to precisely control the temperature of the sample.

FIGURE 4-17 shows sample sensor temperature during a temperature change. Initially the set point of both loop 1 and loop 2 is changed from the initial temperature to the final set point at a constant rate. This is shown as time t1 to time t2 in the figure. A designated wait time is observed (shown as t2 to t3 in FIGURE 4-17). At this time the temperature drift, time derivative of the sample sensor temperature, is calculated along with the difference between the sample sensor temperature and the final set point. When the drift is less than a specified amount (at time t4 in FIGURE 4-17) and when the difference is less than the temperature band (at time t5 in FIGURE 4-17) the temperature is considered to have settled and a measurement can be obtained.

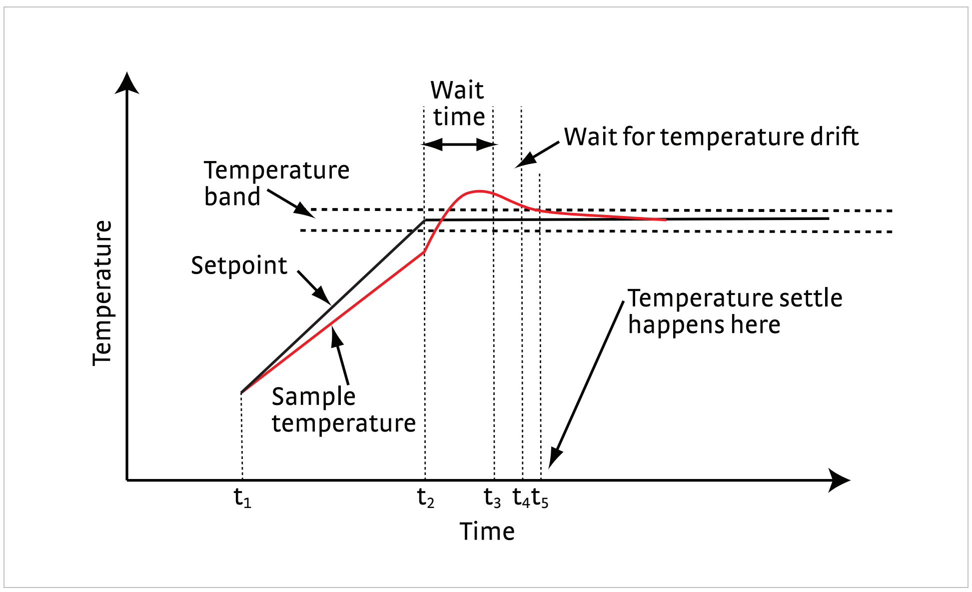


FIGURE 4-17 ***Sample sensor temperature during a temperature change***

4.6.3 How to Change Temperature Control and Settle Parameters

As we have seen from the previous section there are 4 parameters that determine the settle criteria:

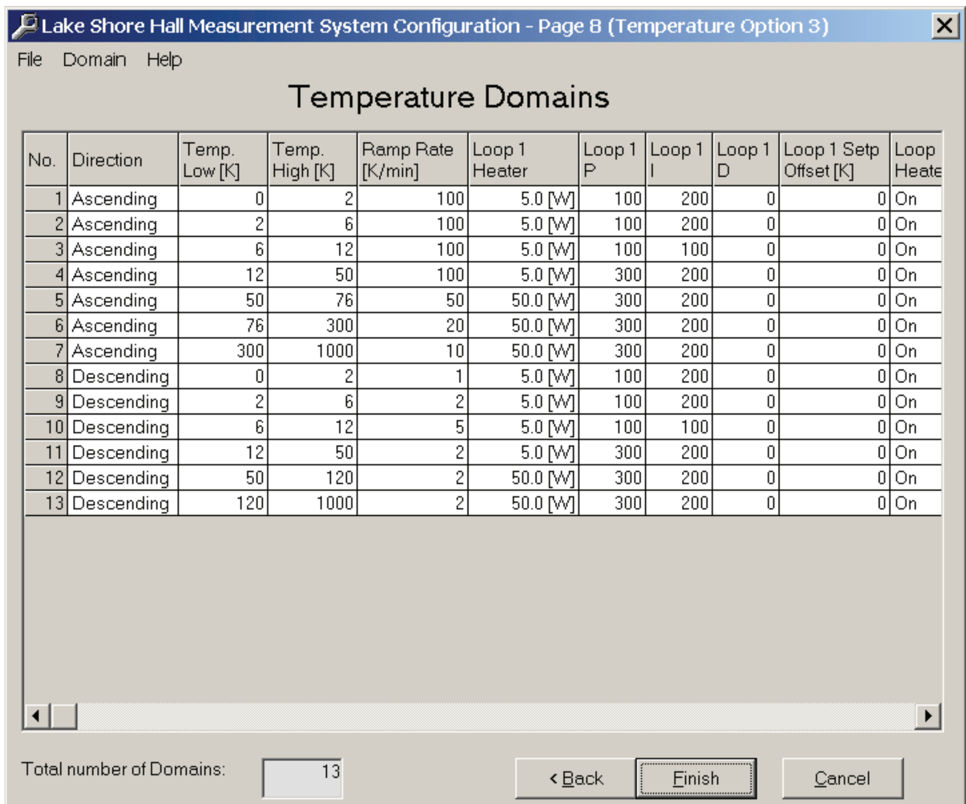
- *Ramp rate:* the rate, in degree/min, that the setpoint changes from the initial setpoint to the final setpoint.
- *Settle wait time:* the time, in minutes, to wait after the final setpoint is reached. This value can be zero.
- *Settle temperature drift:* the drift rate, in degree/min. The drift of the temperature must be less than this value for settle to be met. If this value is large (10 °/min), then this criteria is essentially not used.

- *Settle temperature band:* when settle criteria is met, the temperature will be within the settle band and the drift will be less than the settle temperature drift. If the temperature band is large (10 °) then this criteria is essentially not used.

In addition, there are parameters for the temperature controller. These parameters define how control of the temperature is obtained. These include:

- Loop 1 PID (gain, reset, and rate) values
- Loop 2 PID (gain, reset, and rate) values
- Loop 1 heater range
- The difference between the loop 1 setpoint and loop 2 setpoint.
- The setting of the needle valve to determine the flow through the cryostat.

These parameters depend on temperature. They are maintained by the configuration utility in structures called domains. A domain is defined with a beginning temperature and an end temperature. In addition, different parameters can be defined for ascending (heating) directions and descending (cooling) directions. FIGURE 4-18 shows the domain tables in the configuration utility.



No.	Direction	Temp. Low [K]	Temp. High [K]	Ramp Rate [K/min]	Loop 1 Heater	Loop 1 P	Loop 1 I	Loop 1 D	Loop 1 Setp Offset [K]	Loop Heate
1	Ascending	0	2	100	5.0 [W]	100	200	0	0	On
2	Ascending	2	6	100	5.0 [W]	100	200	0	0	On
3	Ascending	6	12	100	5.0 [W]	100	100	0	0	On
4	Ascending	12	50	100	5.0 [W]	300	200	0	0	On
5	Ascending	50	76	50	50.0 [W]	300	200	0	0	On
6	Ascending	76	300	20	50.0 [W]	300	200	0	0	On
7	Ascending	300	1000	10	50.0 [W]	300	200	0	0	On
8	Descending	0	2	1	5.0 [W]	100	200	0	0	On
9	Descending	2	6	2	5.0 [W]	100	200	0	0	On
10	Descending	6	12	5	5.0 [W]	100	100	0	0	On
11	Descending	12	50	2	5.0 [W]	300	200	0	0	On
12	Descending	50	120	2	50.0 [W]	300	200	0	0	On
13	Descending	120	1000	2	50.0 [W]	300	200	0	0	On

FIGURE 4-18 ***Domain tables in the configuration utility***

To modify a domain click on the domain, the window in FIGURE 4-19 will open.

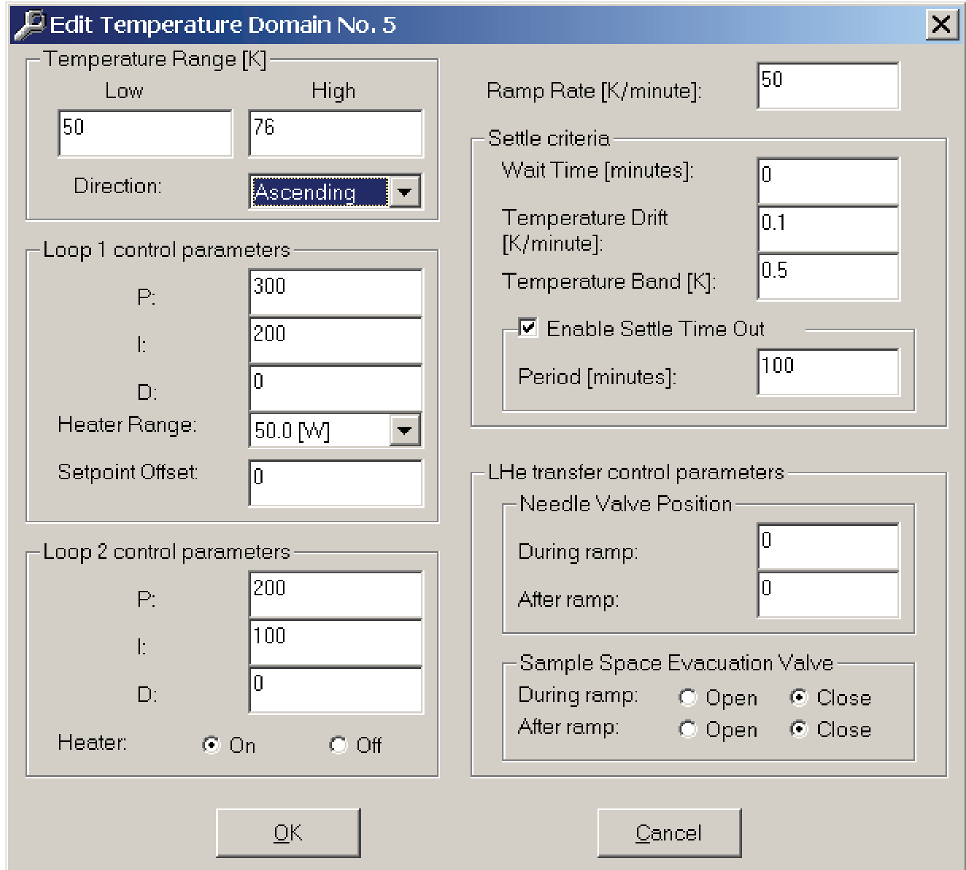


FIGURE 4-19 ***Domain edit window***