Lakeshore CRX-VF Cryogenic Vertical Field Probe Station SOP

Please contact Neil Dilley (ndilley@purdue.edu) or Xiangkai Liu (liu2490@purdue.edu, cell 765-409-7578) if you notice anything abnormal during the operation of the probe station.

The CRX-VF is a versatile cryogen-free micro-manipulated probe station targeted for the electrical testing of the devices from DC to 40 GHz with the vertical field up to 2.5 T, within the temperature range between 10 K and 400 K with chuck biasing capability (if using the grounded chuck instead, the temperature can go up to 500 K).

The operation of the probe station covers a few steps including **the chamber preparation**, **sample loading**, **vacuum operation and system cooling**, **electronic temperature control**, **magnetic field control**, **electrical measurement**, and **finishing measurement**. Please follow the procedures carefully listed below to ensure a successful measurement.

Part I: Chamber preparation for safety check

- Before loading the sample inside the chamber, please make sure the probe station is in the following condition:
  - The probe station is under vacuum to exclude contamination and oxidation.
  - The two Model 336 controllers, both in the upper position and the lower position read the temperature at around 298 K, as shown in Figure 1. This ensures the chamber is ready to be vented for sample loading. You can also refer to the temperate shown on the control panel from the MeasureLink interface to ensure all the temperature have reached room temperature, as shown in Figure 2.

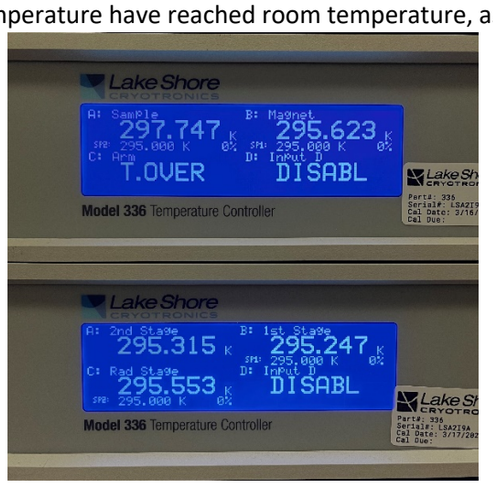


Figure 1. Model 336 controllers

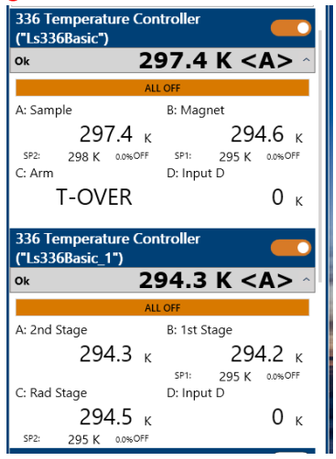


Figure 2. Temperature on the control panel

  - The cooling assembly closed cycle refrigerator (CCR) is OFF, as shown in Figure 3, as no noise could be heard from the CCR.

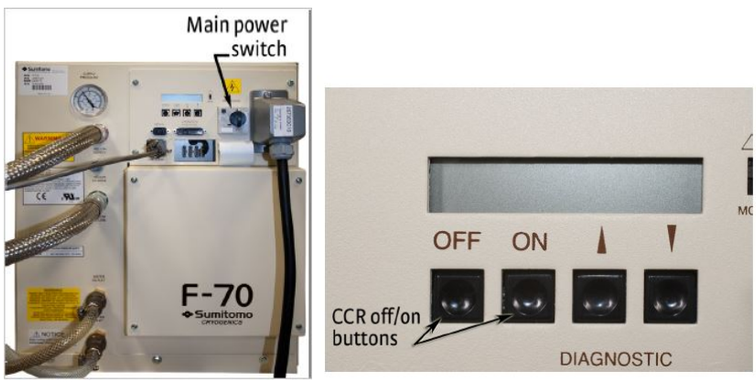


Figure 3. CCR compressor panel and off/on buttons

  - All the probe tips are raised.

- **Please note that magnetic field should NEVER be ramped if the magnet is not fully cooled down by CCR. Failure to comply will lead to the magnet quenches or the misfunctioning of the magnet protection diodes which could result in the severe damage of the magnet.**

- The following are some **common mistakes** that can be made while operating the probe station. **These mistakes can result in costly damage to the probes or sample cooling assembly and should be strictly forbidden.**
  - **Energizing the magnet with the magnet above the operational safe temperature for field.**
  - **Moving the heat switch from the Rad or Open position to Base with the magnet energized.**
  - Opening the vacuum chamber to atmosphere with a cold sample cooling assembly.
  - Heating the sample cooling assembly when it is not under vacuum.
  - Not raising the probe tips before changing temperature or evacuating the chamber.
  - **Placing a ferromagnet with in-plane easy axis in the chamber, and applying out-of-plane magnet field.**

Part II: Sample Loading

Please wear nitrile gloves in the following operation. Hand oils will contaminate the surfaces, resulting in poor vacuum and thermal performance.

- Vent the chamber:
  - Move up and retract all probes from the sample stage to provide maximum room for the sample stage movement during the venting process.
  - Turn off the vacuum isolation valve shown in Figure 4 in the clockwise direction to cut off the nitrogen purge gas flow into the vacuum pump.

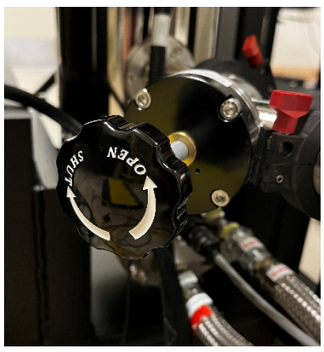


Figure 4. Vacuum isolation valve

  - Turn off the vacuum pump by pressing the button indicated by the arrow, as shown in Figure 5.

Figure 5. Vacuum isolation valve

  - Using the 3 mm hex driver, unlock the four captive screws on the vacuum chamber lid, and turn on the vent valve in the counterclockwise direction to vent the chamber. The vent valve is shown in Figure 6.

Figure 6. Chamber vent valve

  - Once the dry nitrogen purge gas fully backfill the chamber, the vacuum chamber lid shown in Figure 7 could be lifted. Please gently move up the lid, and place it on the designated area outlined on the adjacent table, as shown in Figure 8.

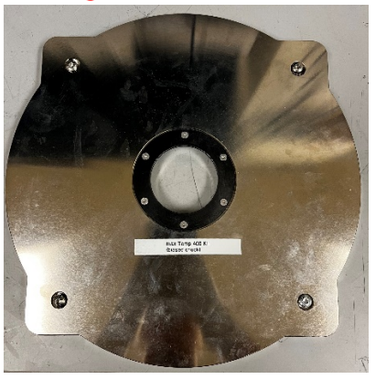


Figure 7. Vacuum chamber lid

Figure 8. Designed area for chamber lid placement

- Sample mounting:
  - Use the 2.5 mm hex driver, loosen the M3 captive screws from the outer edge of the radiation shield lid shown in Figure 9. Rest the radiation shield lid on top of the vacuum chamber lid.

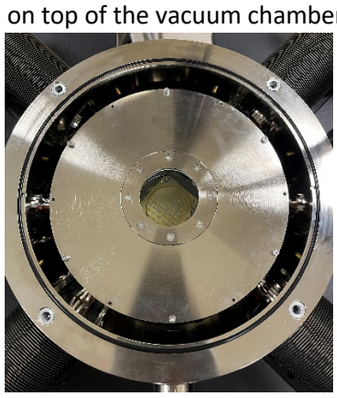


Figure 9. Radiation shield lid

  - Properly mount the sample onto the sample holder to ensure close thermal contact. Align the sample properly in parallel to the grooves of the sample holder.
  - Silver paint could be used in the mounting process to ensure the sample is firmly fixed on the holder. If it is used for the sample mounting, leave the chamber open and wait **10 minutes** before putting back the radiation shield lid, so that the solvent would be fully evaporated and would not enter into the vacuum pump. **Please DO NOT use the acetone to remove the sample if not given the permission from Neil or Xiangkai, as excess acetone might damage the connection of top gold plate with the main body of the sample holder**.
  - Move in the probe arms and land down the probe tips on the sample. Make sure a good physical contact could be achieved between the tips and the sample with the current configuration. For RF probes, make sure each probe should land on all three points on the sample.
  - Keep the purge gas running as it would keep the moisture and contaminants from entering the chamber.
  - Use the 2.5 mm hex driver, attach the radiation shield lid to the chamber and tighten the screws.
  - Put the vacuum chamber lid back on, clean the o-ring groove in the vacuum chamber by the wiper soaked with IPA if any contamination is noticed. Please note that do not tighten the screws of the vacuum chamber lid before turning off the nitrogen vent valve. This would prevent the pressure accumulation inside the chamber with the vacuum chamber lid fixed and nitrogen purge gas fluxing in at the same time.
  - Turn off the vent valve. Push down the screws of the vacuum chamber lid until they lock into place. There is no need to overtighten the screws, as the vacuum inside the chamber already ensures a tight seal of the chamber from the outing atmosphere.

Part III: Vacuum operation and system cooling

- Evacuating the vacuum chamber:
  - Use the z-axis micrometers to raise all probe tips 3 mm to 4 mm above the sample, and retract all the probe arms back. This would prevent the potential collision between the probe tips with the sample holder due to the vertical displacement of the sample holder during the evacuation and cooling process.
  - Open the vacuum isolation valve completely in the counterclockwise direction.
  - Turn on the vacuum pump by pressing the button indicated by the arrow. The vacuum gauge TC1 should begin reading immediately, and the turbo pump should reach full speed in approximately **5 minutes** with the working frequency at 1100 Hz, as shown in Figure 5.
  - Wait **20 minutes** until the CRX-VF chamber pumps below 10^{-2} Torr, and ion gauge IG1 automatically turns on with the reading in the 10^{-5} Torr range. At the same time, make sure the power of turbo pump is around 6 W to ensure it is functioning properly.
- System cooling:
  - Turn on the CCR by pressing the ON button, as shown in Figure 3. Please wait about **5 minutes** after the CCR is turned on to ensure it is running properly without shutting down by itself.
  - The recommended operation for the temperature measurements is to first let the entire CRX-VF station be cooled down to base temperature at 10 K, and then warm the sample stage to the set measurement temperature. To cool the sample to the base temperature, the manually operated heat switch control knob shown in Figure 8 should be rotated all the way down in the Base direction, in the clockwise rotation until it reaches the full stop.

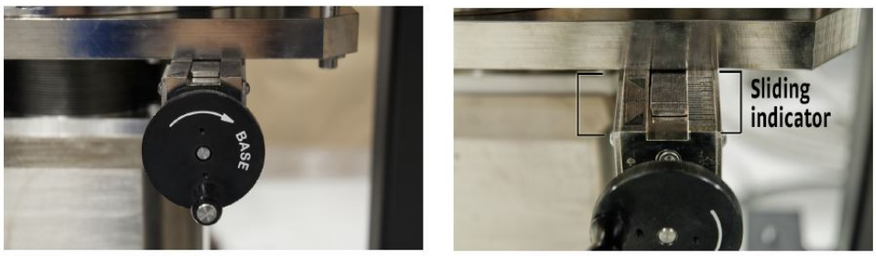


Figure 8. Heat switch control knob

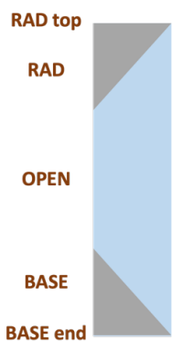
- It would take approximately **6 hours** for the system to be fully cooled down.
- If your measurement would start from a different temperature other than 10 K, you could also directly set up the sample stage temperature by changing the position of the heat switch and the setpoint on Model 336 controller. Please refer to the following section for more details.

Part IV: Electronic temperature control

The two Model 336 temperature controllers provide the electronic control for the CRX-VF stage temperatures. The top Model 336 provides control of the sample stage, the magnet stage, and monitors the probe arm sensor. The bottom Model 336 monitors the CCR second stage and provides control of the CCR first stage and radiation shield.

During normal operation, **the sample stage is the only stage actively controlled to a temperature setpoint. The other control loop setpoints should be set to zero with the heaters turned off.**

In the temperature control, the controller balances its heater power against the cooling power of the CCR to the temperature setpoint. The cooling power is controlled by the position of the manually operated heat switch. The following Table 1 summarizes the sample stage temperature ranges for different heat switch positions.



Heat Switch Position	Sample Stage Temperature
BASE end	10 K
BASE range	10 K to 35 K
RAD top	36 K
RAD range	36 K to 100 K
OPEN	100 K to 400 K

Table 1. Heat switch position temperature ranges

- Sample stage temperature control:
  - To operate the sample stage at **base temperature 10 K**, the heat switch should be in the full stop (**Base**) position, and no electronic control is required.
  - To operate the sample stage **between 10 K to 35 K**, the heat switch should be in the **Base** range, where the sample stage is thermally connected to the magnet stage, as is shown in Figure 9 (a). Turn the heat switch knob towards the Rad position to increase the thermal resistance between the sample stage and magnet stage; while on the top Model 336 controller, set the sample stage setpoint to the desired temperature, and select the heating range to **Low**. The magnet and radiation shield stage heaters should remain off at this stage.
  - ❖ **Please ensure that the applied magnet field must follow the temperature limits in Table 2 when charging the superconducting magnet. Failure to comply will result in a magnet quench. Numerous or severe quenches can damage the magnet.**

Maximum magnetic field capability	Magnet temperature for safe operation	Sample stage temperature
±2.5 T	<5 K	Base
±2 T	<5.5 K	10 K to 400 K

Table 2. Maximum magnetic field capability at different sample stage temperatures

  - To operate the sample stage **between 36 K to 100 K**, the heat switch is moved to the **Rad** position where the sample stage is thermally connected to the radiation shield stage, as is shown in Figure 9 (b). Adjust the heat switch from full stop in the Rad direction (for temperatures close to 36 K) to 2.5 turns in the Base direction (for temperatures close to 100 K). On the top Model 336 controller, change the sample stage setpoint to the desired temperature, and select the heating range to **Med**.

- To operate the sample stage **between 100 K to 400 K**, the heat switch is moved to the **Open** position where the sample stage is thermally isolated from both the magnet stage and the radiation shield stage, as is shown in Figure 9 (c). The heat switch is set at the midpoint of its travel (five turns from either full stop position). Over this entire operating temperature range no fine adjustment of the heat switch is required. On the top Model 336 controller, change the Input A sample stage setpoint to the desired temperature, and select the heating range to **High**.

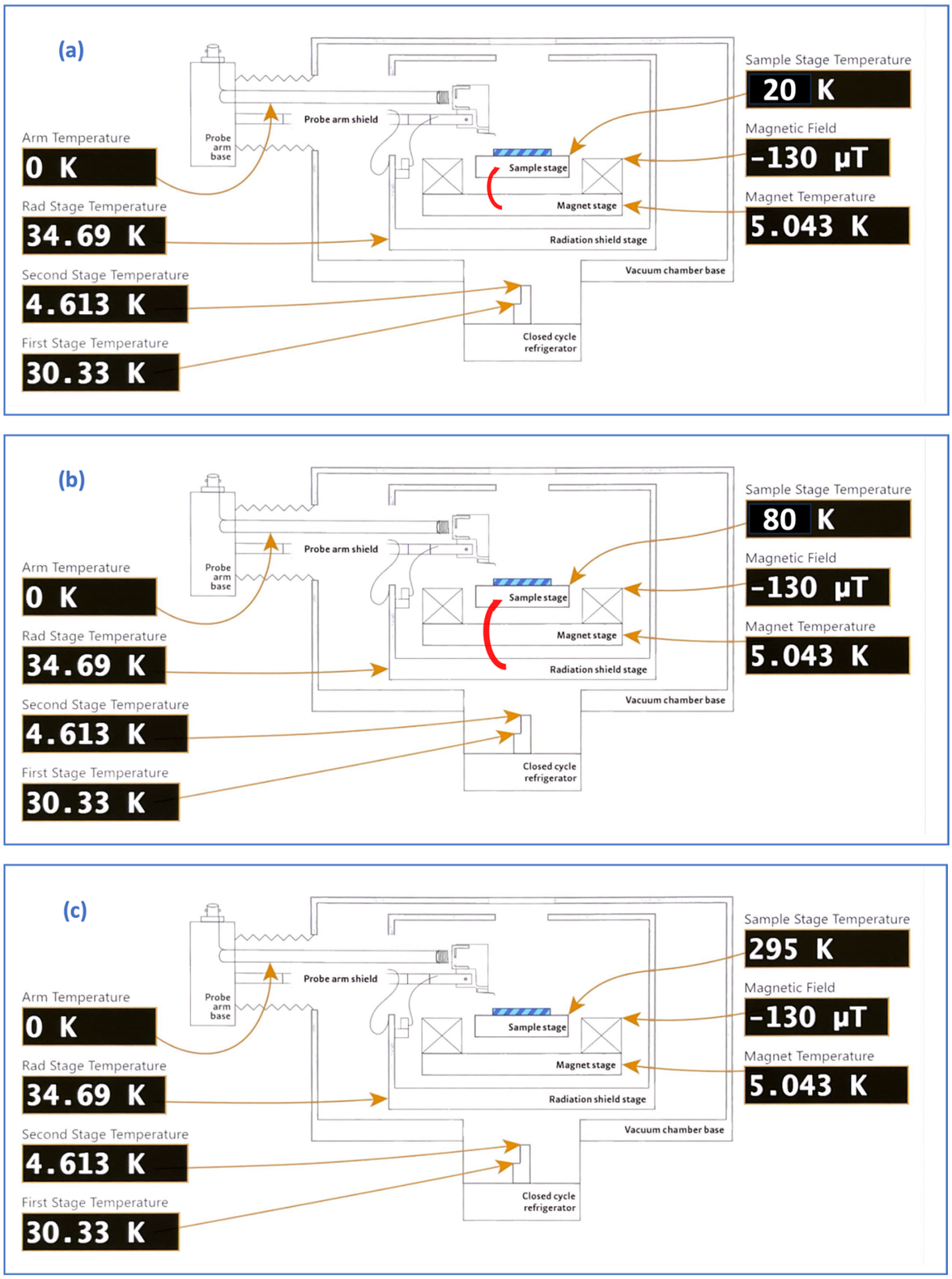


Figure 9. Sample stage thermal connection diagram

- A summary of the temperature control with the heat switch and Model 336 controller settings is listed in the following Table 3.

		Probe station cool down	Maintaining sample stage at base temperature	Operating sample stage 10 K to 35 K	Operating sample stage 36 K to 100 K	Operating sample stage 100 K to 400 K	Returning sample stage to room temperature
CCR control settings	Main power	On	On	On	On	On	On
	CCR on/off	On	On	On	On	On	Off
Mechanical control settings-1	Heat switch range	Depending on the targed temperature	Base (full stop)	Base (variable tension, see below)	Rad (variable tension, see below)	Open	Open
	Sample stage Heater range	Depending on the targed temperature	Off	Low	Med	High	High
Eletronic control settings	**Magnet stage** Heater range	Off	Off	Off	Off	Off	High
	Radiation shield stage Heater range	Off	Off	Off	Off	Off	High
	CCR first stage Heater range	Off	Off	Off	Off	Off	High
Mechanical control settings-2	Heat switch rough position	Depending on the targed temperature	Base (full stop)	0 ~ 1 turn --- 6 ~ 7 K 1 ~ 2 turn --- 7 ~ 10 K 2 turn ~ middle --- 10 ~ 14 K middle~ full --- 14 ~ 35 K	0 ~ mid range --- ~ 50 K mid ~ full range --- ~ 100 K	Open	Open

Table 3. Heat switch and Model 336 controller settings for temperature control

Part V: Magnetic field control

Once the magnet stage is cooled and stabilized in temperature, the magnet can be charged using the Model 625 superconducting magnet power supply. The maximum magnetic field capability in the CRX-VF is dependent on the operating temperature of the sample stage due to magnet heating, as shown in Table 2.

❖ **Please note that magnet should NEVER be charged if the magnet is not fully cooled down by CCR. Failure to comply will lead to the severe damage of the magnet.**

Maximum magnetic field capability	Magnet temperature for safe operation	Sample stage temperature
±2.5 T	<5 K	Base
±2 T	<5.5 K	10 K to 400 K

Table 2. Maximum magnetic field capability at different sample stage temperatures

- Applying the magnetic field:
  - Turn on the Model 625 superconducting magnet power supply. The power supply performs self-diagnostic tests on power up and will show any error on the display.
  - Check that the following power supply parameters in Table 4 are properly displayed. These settings are necessary to protect the magnet. **The maximum ramping rate of the magnet is 840 G/s and please do not exceed it during the operation of the magnet.**

Field constant	Max ramp rate	Max voltage	Max current	Quench detection	Quench limit	Ramp segments	Current (A)	Ramp rate (A/s)
Each magnet has a unique value between 0.071 to 0.075 Tesla/A	0.12 A/s	1.2 V	35 A	Enabled	1.0 A/s	Enabled	5	0.12
							10	0.10
							15	0.08
							20	0.06
							25	0.05

Table 4. Power supply parameters

  - Press **Output Setting**, enter the desired field using the sign to set polarity (+/-), and press **Enter**. The power supply will not accept a field setting that exceeds the max current limit entered in max settings.
  - The ramping LED will turn on as the supply ramps field to the desired value. When the ramping LED turns off, the field change is complete. The magnet field should be reset to zero when measurements are not being taken. The easiest way to do so is to press **Zero Output**.
  - ❖ Please **DO NOT** move the heat switch from the Rad or Open positions to the Base position with the magnet energized. Failure to comply will result in a magnet quench. Numerous or severe quenches can damage the magnet.

Part VI: Electrical measurement

Before doing the measurement, please make sure the following conditions are met:

- ✓ The vacuum pump is running with the full speed at 1100 Hz at around 6 W, and the ion gauge IG1 shows the reading of below 10^{-5} Torr.
- ✓ The CCR is turned on for the cooling, and the temperature of all stages are stable.
- ✓ **The temperature of the magnet is below 5 K if the magnetic field will be applied and ramped.**

Once all the above conditions are satisfied, the electrical measurement could be performed. Different types of electrical measurements could be carried out, including but not limited to: electrical characterization (e.g. I-V) of electrical devices (e.g. FETs) with semiconductor analyzer; Hall-type of measurement with M81 module or external electronics equipment (please refer to the M81 manual); and RF measurements etc.

One example LabView program for the simple FET measurement could be found in the folder with the pathname "C:\Users\PS_Administrator\Desktop\Example programs".

- **If your measurement needs to be carried out below 77 K, please pay special attention and follow the procedures below**:
  - **Close the vacuum isolation valve** shown in Figure 4, **while keeping both the CCR and vacuum pump running**.
  - Perform the measurement at any temperature below 77 K.
  - After the measurement below 77 K, **before moving to the next temperature above 77 K**, **set the sample stage temperature at 77 K** and **open the vacuum isolation valve**, **stay at 77 K for 5 minutes**, to ensure that the condensed contamination be fully pumped away.
  - Continue the measurement at any other temperatures above 77 K as normal.

- When the sample stage is kept below 77 K, the cooled radiation shield acts as a cryopump, creating a better vacuum than the turbo pump. In this case, the cryo-pumping reduces the vacuum pressure in the chamber even below that of the turbo pump, which makes it possible to draw outside contamination in through the vacuum system. Turning the isolation valve off would minimize the outside contamination and the accompanied condensation onto the chamber and the sample.

Part VII: Finishing measurement

After all the measurements are finished, please see the following procedures to prepare sample unloading.

- **Turn-off the magnetic field:**
  - **The magnet field should be reset to zero after the measurements. Make sure the magnetic field is ramped back to 0 T.**

- Move up all the probe tips, and retract all the probe arms to prevent the potential collision with the sample holder during the chamber vent process.

- Temperature ramp up:
  - Move the heat switch to the **Open** position.
  - Turn off the CCR cooling assembly.
  - On both the top and bottom Model 336 controllers, change the setpoint of the sample stage, magnetic stage, radiation shield stage, and CCR first stage to 295K, and select the heating range to **High** to ramp up all the temperatures to room temperature. Please refer to Table 3 for more details.

- Unload the sample:
  - Once the temperature of all stages has risen up to around 295 K, follow the steps mentioned in **Part II** to vent the chamber and unload the sample.
  - After the sample is taken out from the sample holder, follow the procedures mentioned in **Part III** to evacuate the chamber. Make sure the probe station is kept in good vacuum after all the measurement process.