Setting up the TSC Cryostat System

Walkthrough Manual

ISU ECpE Senior Design Group May14-03
Joshua Straquadine*, Mouhamadou Diallo, Nicholas Rodriguez, and Martin Andersen
(*Team Leader)

Contents

Sample Prep

CAUTION: In order to protect the organic samples from the oils in your skin, be sure to wear a pair of rubber gloves while handling the sample!

1. Before placing the sample into the chamber, make sure that the bottom glass surface is free of any cryogenic grease or other substance. If any such material is present, use a lint-free wiper and some isopropyl alcohol to gently clean the surface. Do NOT apply the alcohol directly to the sample, but rather apply it to the wiper first, and then rub the wiper on the surface.

Chamber Prep

CAUTION: In order to protect the organic samples from the oils in your skin, be sure to wear a pair of rubber gloves while handling the sample!

1. If the lower chamber insulation is not already in place, carefully insert aluminized Mylar sheets into the chamber beneath the cryostat one at a time, taking care to pack them all the way to the bottom, but avoiding the wiring within the chamber. The thin cryogenic wires are fragile and will not withstand any considerable strain. The packing does not need to be too dense: five or so Mylar sheets (roughly one square foot each) should suffice. Finally, take care not to allow the insulation to make contact with the suspended heater wire, as contact can cause the insulation to melt.

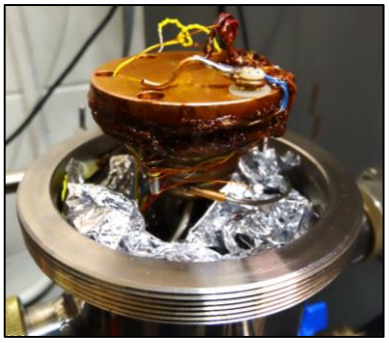

Figure 1: Aluminized Mylar packed into the bottom half of the chamber

2. Mylar sheets should similarly be added to the chamber cover as well. Loosely pack aluminized Mylar along the inside walls of the cover, making sure to leave enough space to accommodate the shroud when fully assembled. About three sheets of Mylar should be enough. The blue cap for the liquid nitrogen Dewar can be used to compact the Mylar enough to make room for the shroud.

3. Make sure that no screw is placed in the marked screw hold in figure 3. The tie-downs for the RTD wire and the electrical contact wire should be threaded through this hole.

Figure 2: Aluminized mylar packed into the top cover of the vacuum chamber, leaving enouh space for the shroud

4. Before placing the sample in the chamber, make sure that the cryostat surface is clear of any cryogenic grease or other substances. If any such material is present, clean the surface by applying isopropyl alcohol to a lint-free wiper and rubbing the surface, taking care not to apply any alcohol to the brown varnish. Isopropyl alcohol slowly dissolves the varnish and will degrade it over time. With the same alcohol and wiper, gently clean the tip of the yellow, electrical contact wire, to ensure that it will make good contact to the sample.

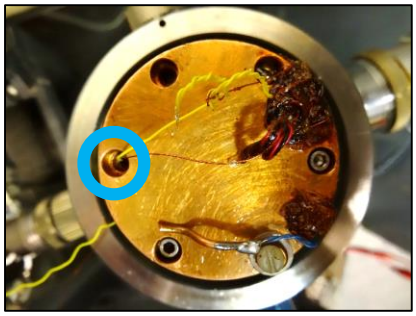

Figure 3: The marked screw hole should be left open, and the tie-down wires should be threaded through this hole.

5. Using a sheet of fine grit (>400) sandpaper, gently sand the underside of the copper ITO clamp. Copper oxide slowly forms on the clamp at ambient temperature and humidity, and if not removed it can lower the quality of the measurement.

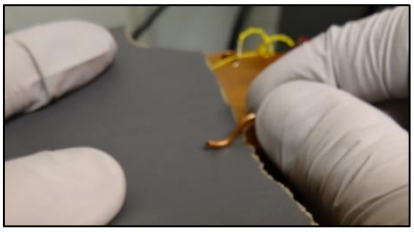

Figure 4: Sanding the underside of the clamp

Loading a Sample

CAUTION: In order to protect the organic samples from the oils in your skin, be sure to wear a pair of rubber gloves while handling the sample!

1. Pull up on the contact and RTD wires to create a large enough space to accommodate a sample on the surface of the cryostat head. Loosen the nylon screw on the copper ITO clamp and rotate the clamp away from the center of the cryostat.

2. Carefully, and without scratching the top surface, slide the sample onto the copper stage. Center it as well as possible, and orient it such that the ITO clamp can make solid contact.

3. Rotate the ITO clamp back over the ITO region, carefully lower it to the surface, and press down on the clamp. While holding it down with one hand, use your other hand to tighten the screw and lock the clamp in place.

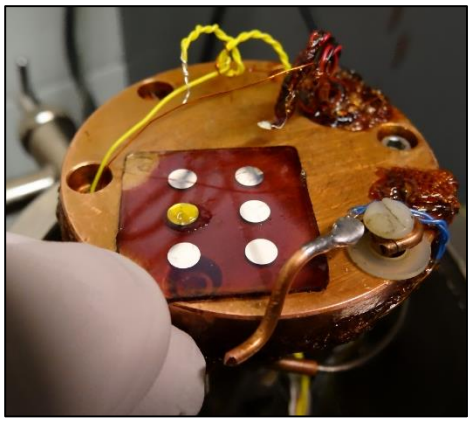

Figure 5: Carefully sliding the OPV sample onto the cryostat. This sample had already been used for a few tests, so the yellow grease had already been deposited on one of the contacts.

4. Designate one contact on the surface to act as the thermal reference. Using the tip of a needle, wire, or other thin metallic object, carefully apply a small dot of cryogenic vacuum grease to that contact.

5. Using the tie-down wire connected to the RTD, lower the RTD onto the greased contact. For best results, bend the RTD wire such that the RTD naturally comes into contact with the closer edge of the contact, and then pulling the tie-down tight causes the head to slide into the center of the contact. The head should be flat on the sample surface. Wrap the tie-down wire around the screws on the underside, as close to the copper as possible.

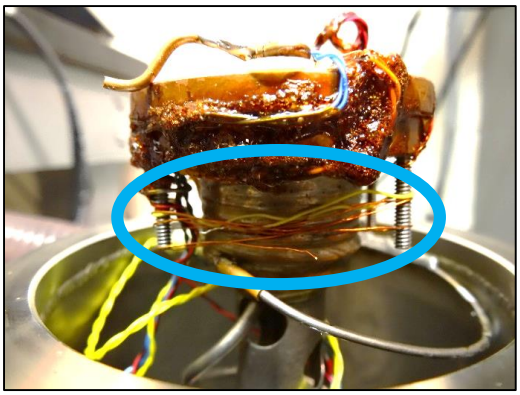

Figure 6: Stabilizing the tie-down wires by wrapping them around the screws on the underside

6. Repeat the same procedure with the electrical contact wire. Because this wire is smaller, care must be taken to ensure that the wire makes perpendicular contact to the surface, or else it will buckle and slide under the tie-down force. Wrap the tie-down wire on the same screws, taking care not to disturb the RTD tie down.

7. Carefully lower the shroud onto the cryohead, and gently push down until the shroud is snug and secure. Make sure the shroud remains upright, and does not become canted off to one side, as this can disrupt the contacts to the sample.

Figure 7: The shroud on the cryostat

8. Carefully lower the vacuum cover onto the chamber, making sure that no Mylar or tie-down wires get caught between the vacuum chamber halves. Tighten the brass collar to complete this process.

Connecting the Instruments

1. First, ensure that all four instruments (Keithley 2400, Keithley 617, Keithley 6485, and Lakeshore 331) are interconnected by stacking GPIB cables. The order of these cables does not matter as long as all devices are connected to the same bus.

2. In order to protect the sensitive amplifiers in the Keithley 6485 Picoammeter, make sure that the “Zero Check” function is activated. When it is enabled, either “ZZ” or “ZC” will appear after the current measurement. If this is not the case, turn this function on by pressing the “ZCHK” button on the front panel. Do not attempt to connect or disconnect any cables until this is done.

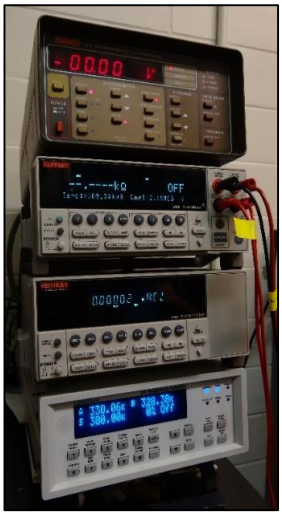

Figure 8: The stack of measurement instruments

3. Two of the female connectors on the chamber are labeled with ITO. These two should be connected together using the 2-1 BNC adapter, and then connected directly to the positive terminal (marked with a plus sign) on the shielded box.

Figure 9: The 6485 Picoammeter front panel, with the position of the ZCHK button marked.

4. Connect a BNC cable to the negative terminal on the box (marked with a minus sign) and then, using a BNC-Banana adapter, plug the center terminal (the red connector) into the black binding post on the rear panel of the Keithley 617. Connect the black connector to the "Common" terminal on the back of the Keithley 6485.

5. With a second BNC Banana adapter, connect the red terminal to the red binding post on the back of the Keithley 617, and the black connector also to the "Common" terminal on the back of the Keithley 6485. Then connect the other end of the BNC cable to the two remaining connectors on the chamber by using a 2-1 BNC adapter.

Figure 10: The shielded junction box with the connectors and adapters

6. Finally, use the Keithley BNC-terminated-triax cable to connect the isolated jack on the shielded box (the center connector with the white plastic collar) to the rear panel of the Keithley 6485.

7. Ensure that the KUSB-488 GPIB to USB adapter is connected to a GPIB cable on the rear panel on any one of the instruments. Connect the USB terminal to the computer which will be running the automation software.

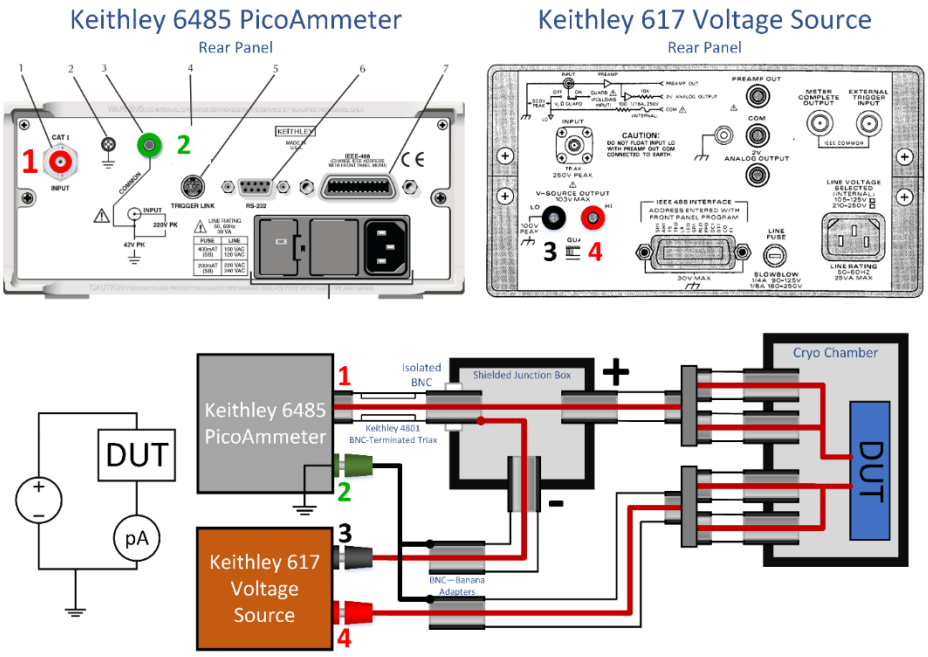

Figure 11: Schematic diagram of the necessary connections for running TSC experiments

Running the Software

1. To run the software, open Labview 2012 or later. Use the file browser to navigate to the VI titled "TSC Control.vi". If the computer in the lab is being used, a desktop shortcut exists with this name.

2. Open program, and Labview runs the program automatically. Input your setpoints, and click on the "Start" button when ready. For more details, see the TSC Software Manual, located on our website.

Starting the Vacuum Pump

1. Once you press the "Start" button on the software, the software will prompt you to turn on the vacuum pump. First, check to make sure each feedthrough into the chamber is snug, then close the chamber valve. Flip the small switch near the back of the pump to turn it on, and allow it to equilibrate to a low pressure. Turn the vacuum off, and observe the pressure for five seconds. If the pressure gauge changes noticeably with the vacuum off, there is a leak somewhere between the pump and the chamber valve. Check each joint and try again.

2. Once the hose and pump are leak free, turn the vacuum back on, and open the valve to the chamber. The pressure gauge will rise momentarily as the chamber evacuates, but will eventually settle down. Turn the pump off one more time and check for leaks. If the pressure changes noticeably, there is a leak in the chamber, which most likely is caused by getting some material stuck between the top and bottom halves of the vacuum chamber.

3. If such a chamber leak is suspected, vent the chamber by turning off the vacuum pump and unscrewing the valve connector from the chamber. Once the pressure is equilibrated back to ambient, open the chamber, readjust, and try again.

Figure 12: The vacuum pump. Ensure that the oil levels (visible on the bottom left) is within the recommended limits.

Starting the Cryogen Pump

1. The cryogen pump is a variable-strength pump controlled by a variac. Tests have shown that the optimal voltage setting is 40 V. To minimize sample vibrations, turn the dial down to zero while the variac is off, turn it on, and then turn the voltage up smoothly to 40 V.

Unloading the Sample

CAUTION: In order to protect the organic samples from the oils in your skin, be sure to wear a pair of rubber gloves while handling the sample!

1. The software will automatically activate the "Zero-Check" function on the picoammeter. This will be denoted by the letters "ZC" after the current reading. Take care NOT to disturb this setting, as turning the zero-check off leaves the sensitive components vulnerable to ESD events from your body or your tools.

2. After a TSC experiment is completed, the temperature will be 300 K. If an experiment has been aborted for any reason, the software will set the setpoint to 300 K, but the system may take a few minutes to reach that temperature. For safety reasons, and to minimize condensation within the cold chamber, wait until both outputs on the sample show at least 280 K before proceeding to vent or open the chamber.

3. Begin by unscrewing the collar at the chamber valve to vent the chamber to atmospheric pressure. Then carefully unscrew the brass collar around the cover and gently lift the cover straight upwards off of the system.

4. Remove the shroud by pulling up on it, taking care not to squeeze to hard to disrupt or scratch the sample inside.

5. Unwrap the tie down wires and pull up on the electrical contact wire, then the RTD. The thermal grease tends to form whiskers when pulled, so the tip of a needle or wire, or the corner of a lint-free wiper may be used to catch the excess from landing on the sample.

6. Carefully loosen the nylon screw holding down the ITO clamp and rotate the clamp away from the sample.

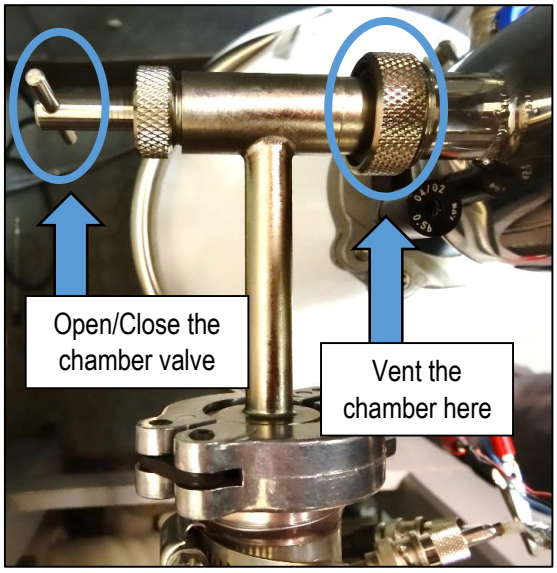

Figure 13: Chamber vacuum connector, showing the valve between the chamber and the pump, as well as the collar which can be used to vent the chamber.

Filling the Liquid Nitrogen Dewar

1. If the liquid nitrogen runs out, begin by removing any samples and disconnecting all of the connectors from the bottom half of the vacuum chamber.

2. Open the collar above the Dewar, but below the black safety-valve assembly, by loosening the wingnut, swinging the bolt out, and separating the two halves.

3. Carefully pull the vacuum chamber directly upwards until the entire tube is exposed. The tube is roughly two feet long. The tube may still be at a very low temperature, so care must be taken in handling it.

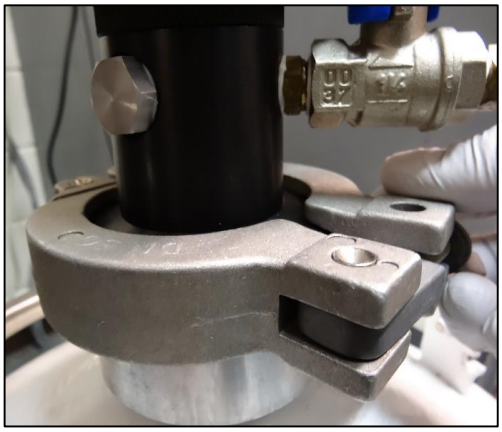

Figure 14: Opening the collar to remove the flask

4. Place the blue insulating cap on the liquid nitrogen flask and take it out to be filled. Due to safety concerns, instructions for operating the liquid nitrogen source at the MRC are not included here. See your supervisor or other user of that system for training if you have not yet been trained.

5. After filling, remove the blue cap. Using several lint-free wipers to protect your hands from the potential cold, wipe any extra condensation from the tube before carefully reinserting it into the Dewar.

6. Reattach the chamber to the flask using the large collar, and then reconnect the vacuum tube, instrument connectors, etc. to prepare for another experiment.

Figure 15: Extracting the tube from the flask. The tube is still dangerously cold.

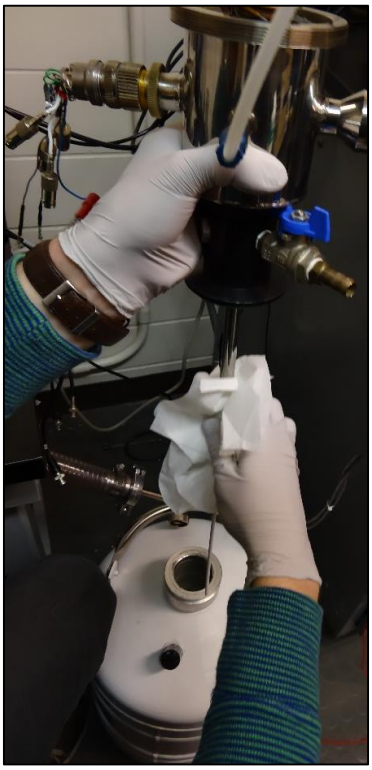

Figure 16: Wiping the condensation from the tube before reinserting it.