PicoLE Temperature Control User's Manual

v1.1

Molecular Imaging

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Temperature Control Overview

The Molecular Imaging temperature controlled sample stages add another aspect to SPM studies. Such studies can now be done while maintaining physiological temperatures, melting two-dimensional crystals, or while imaging any temperature-sensitive surface. The instruction assumes some experience with the standard sample plate and electrochemistry cell. Users with no prior experience can simply refer back to the **PicoLE System module**.

Choosing Cantilevers

Temperature changes during AFM experiments present a special problem when using gold-coated cantilevers. Cantilevers coated on one side will bend due to differential thermal expansion. These cantilevers are still useful, but the stepper motor and set point (or detector position for extreme values) may have to be adjusted each time there are temperature changes. Because the temperature influences the cantilever, even when it is far from the surface, the force is not easily calculated without observing a force-distance curve. Silicon cantilevers are not coated and therefore will not bend. This makes silicon cantilevers essential when imaging while the temperature is being changed (as discussed in the following sections).

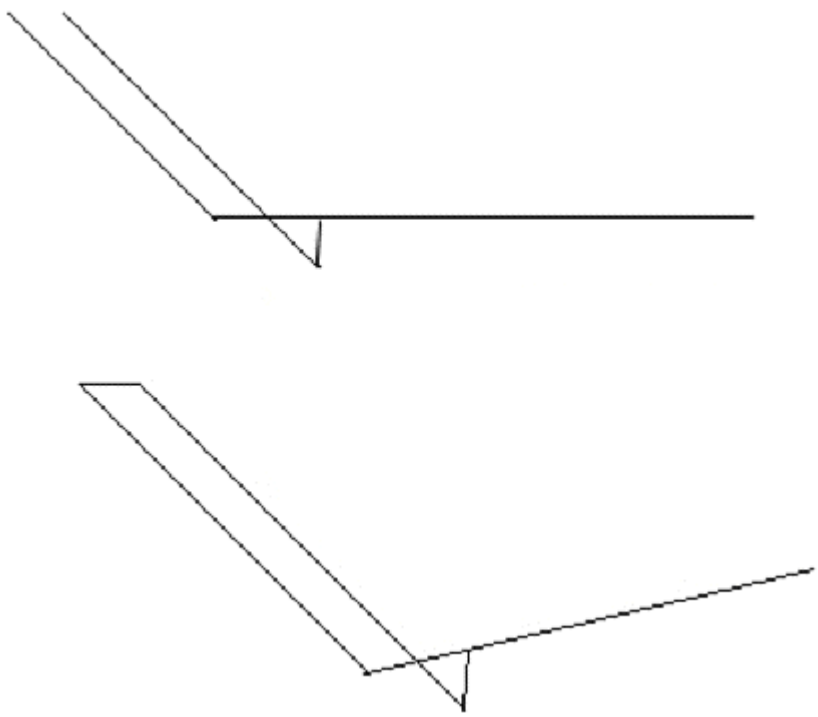

Figure 1 (Force-distance curves at and above room temperature. Bending due to thermal expansion causes deflection of the cantilever before it contacts the surface. The direction and amount of slope depends upon the temperature and cantilever.

High Temperature Stage

The high temperature stage has a temperature range from ambient (23°C) up to 200 °C. Specifications for the temperature controller are given in the Lakeshore Temperature Controller User's Manual. Please limit the ramping rate of the temperature to less than 10 degrees per minute to avoid permanent damage to the stage.

Connection

The following three cables are supplied with the heating stage package as shown:

- One long cable (round Din 7 plug connector to Din 6 and Din4 round connectors)
- One short flexible cable (round Din 7 socket to flat 6-pin female connector)
- One extension cable (round Din4 to Banana plug)

Figure 2 (Connector cables for the High Temperature Stage)

Procedure

1. Connect the extension cable with Din4 (female) to the Din4 (male) on the long cable.
2. Plug the round Din 6 connector from the long cable into the sensor input on the controller back panel as shown in Figure 3 below.

3. Insert the red and black banana jacks into the Hi and Lo Heater outputs respectively on the controller back panel.
4. Connect the round Din 7 plug from the opposite end of the long cable into the Din 7 socket of the shorter cable.
5. Plug the flat 6-pin female connector from the short cable into the 6-pin male connector on the bottom of the hot stage. In order to use a heated stage with MAC mode operation, see Figure 4 below for hot MAC sample stage connections.
6. Connect the other cables the same as operating the microscope without the temperature control stages.

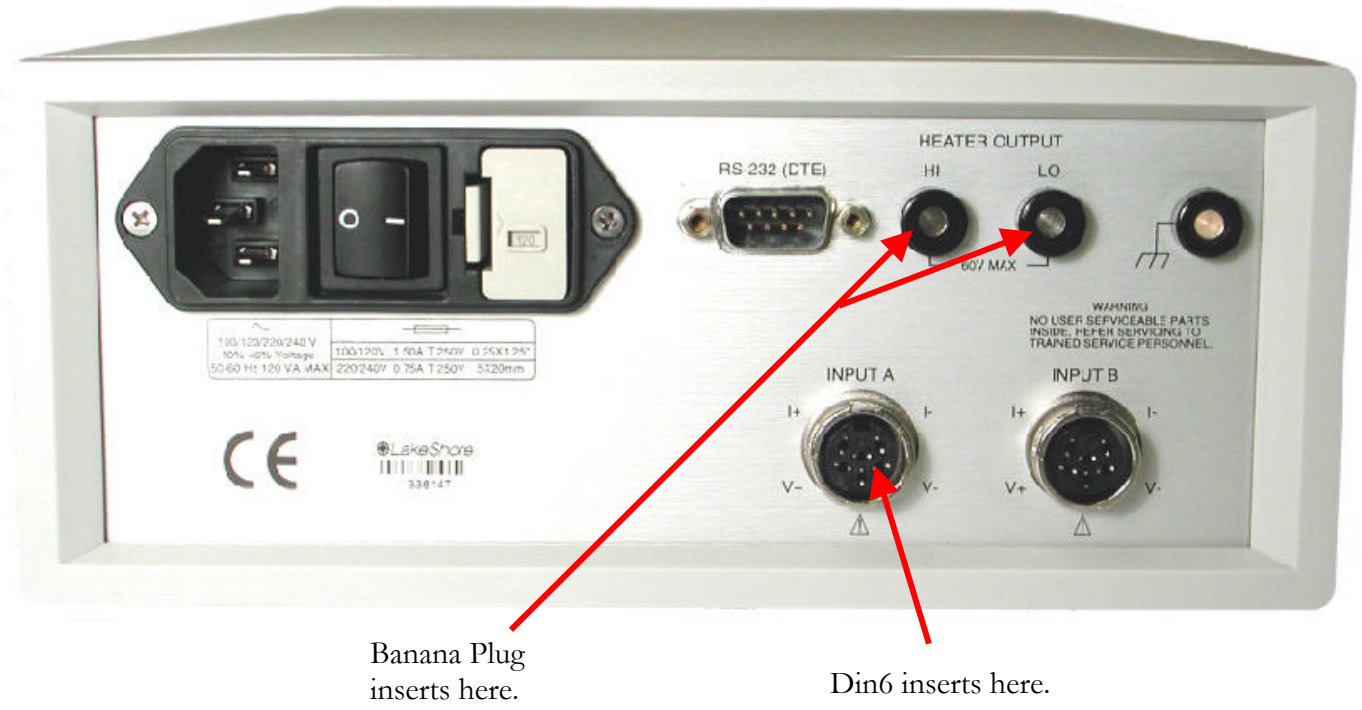

Figure 3 (Lakeshore Temperature Controller - back)

Notes

- Set up the microscope and align the tip-sample distance as previously described. Mount the sample using the air cell or liquid cell. Try not to use double-sided tape to fix the sample on the heating stage if possible because during the heating the glue may soften or melt, causing large sample drift.
- Every sample is different. In general keep the sample thin and avoid solvents that evaporate quickly when the heating stage is used.
- Use uncoated cantilevers when it is undesirable to have the cantilevers bend during heating experiments. Silicon cantilevers, which do not have a metallic coating, are available through Molecular Imaging.

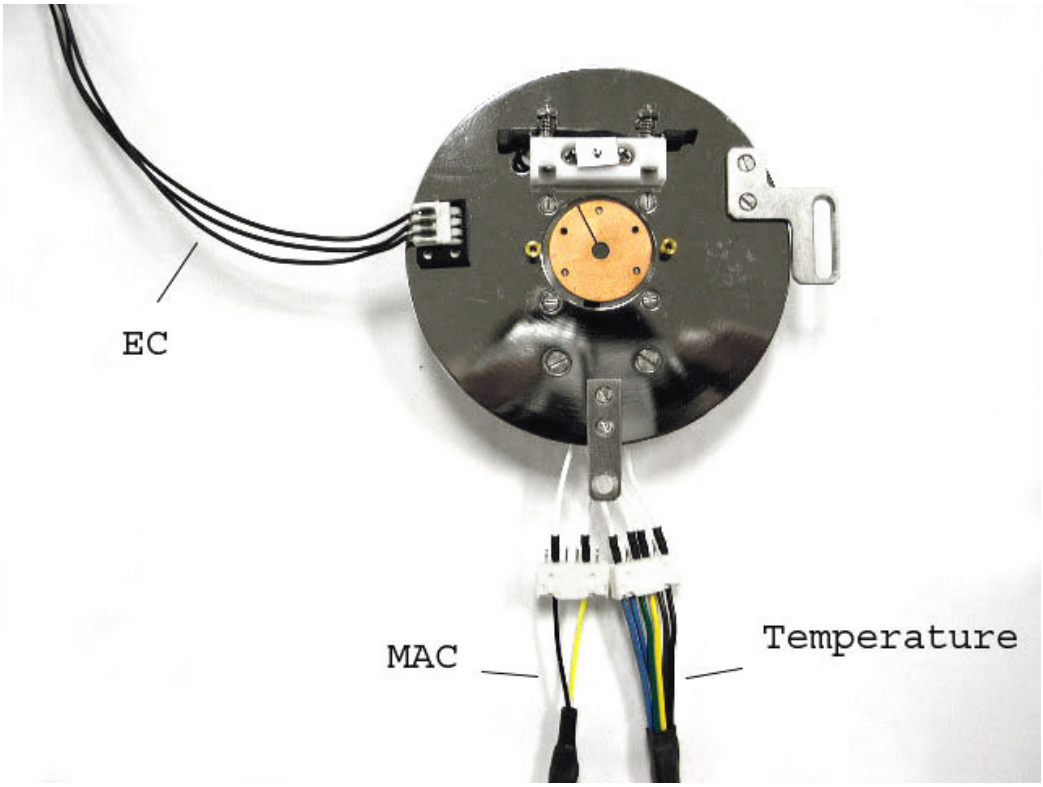

Figure 4 (Top view of hot MAC sample plate connections)

Using the Lakeshore Controller with the High Temperature Stage

1. Turn the Lakeshore controller On, the Heater should be initially Off.
2. Press Auto-Tune and then the up or down arrow keys until the display reads Tune: Manual.
3. Enter the proportional, integral, and differential gains. For example to set the proportional gain press P then entering a number followed by Enter. The numbers 20, 20 and 100 for P, I and D respectively work well in factory tests.
4. Press Set Point, and enter a temperature slightly lower than room temperature.
5. Set the ramp rate to no more than 10 degrees per minute (typically 5).
6. Press Heater Low or Heater High (Lakeshore controller model 330 also has Heater Medium setting). Use Low for desired temperatures lower than 20 °C above room temperature, and Medium or High for other desired temperatures; however, these settings are sample-dependent.
7. Enter the desired final temperature and press Enter.
8. Once the temperature has stabilized, engage the microscope as previously described and start scanning. If the temperature has not stabilized, the signal will continue to drift due to thermal expansion. The graph below in Figure 5 shows a set of data for several temperature jumps from room temperature 23 °C, zero on the time axis corresponds to the time the sample reached the setpoint temperature.

Warning: Setting the rate too high will permanently damage the heater.

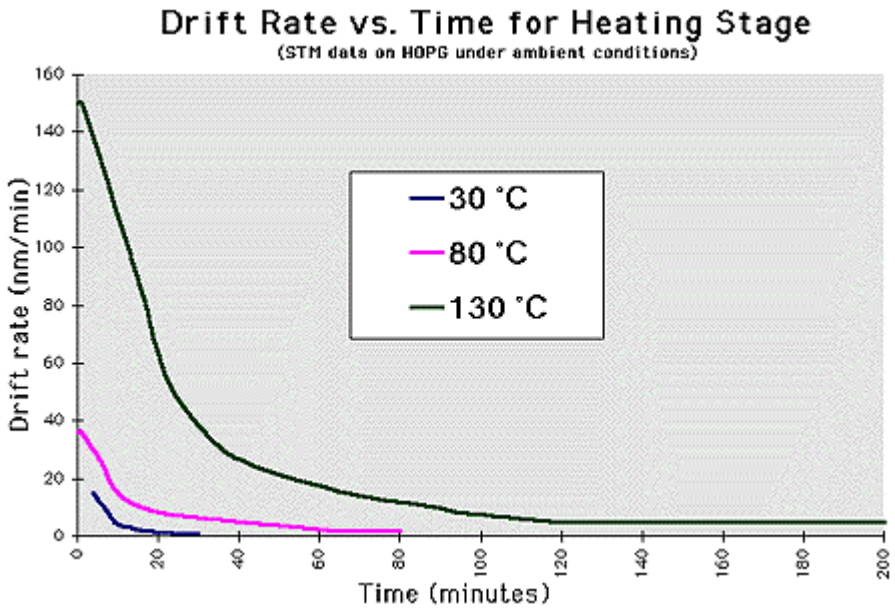

Figure 5

Peltier Stage

The 1x Peltier Stage has a temperature range of –5 °C to 40 °C (assuming room temperature is 23 °C), and 3x Peltier Stage has a temperature range of –30 °C to room temperature. The temperature range for a specific experiment will depend upon the thermal load of the sample. The current booster should be used with the Peltier stage and the water-cooling must be used when lowering the temperature. The current booster includes a safety device that shuts off the power to the Peltier cooler if the reverse side of the Peltier becomes excessively hot, for example, if the water should stop flowing. The supplied current booster cannot exceed the maximum power rating of the Peltier stage.

Water Cooling for the Peltier Stage

When a sample is cooled using the Peltier stage the opposite side of the device becomes hot. The hot side of the device is water cooled to decrease the minimum sample temperature, reduce power requirements and to prevent overheating of the device. Vibrations from the water-cooling system are minimized by using gravity feeding rather than mechanical pumping. Two reservoirs are provided with the Peltier stage. One is used as a source and the other is a receptacle to store the water for recycling. A height difference of three feet between the source and receptacle gives a minimum temperature of –25 °C or approximately 50 °C below room temperature. Some ice cubes can be added into the reservoir to increase the water-cooling efficiency and give a minimum temperature of –30 °C. A diagram showing how to connect the tubing to the reservoirs is given in Figure 6 below.

Warning: The water-cooling connections should be made and tested for leaks before the electronics are connected.

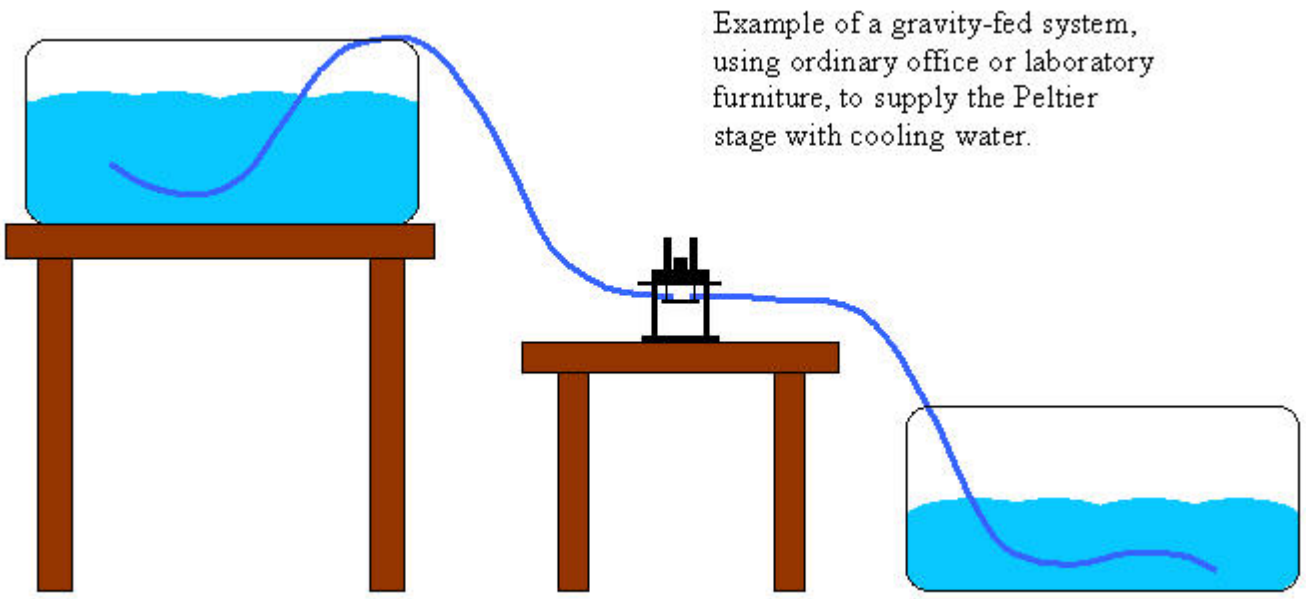

Figure 6 (Gravity-fed water cooling schematic)

Current Booster

The front and back panels of the current booster are shown in Figure 7 below. The LED indicator will light up after the power is on. The power range controls the amount of power output into the Peltier stage. The more power, the colder (or warmer in the case of 1X Peltier for heating) the stage will get. The overheat indicator will be on when the back of the Peltier stage is overheated (it often happens when the cooling water is off) and the current booster will turn off the current supply to the Peltier stage. It automatically recovers when the backside of the stage cools off.

Figure 7 (Above: front view, Below: rear view of the current booster)

There are three cables supplied with the Peltier stage:

- One long cable (round Din 7 plug connector to Din 6 and Din 4 round connectors, same as the one for heating stage)
- One short flexible cable (round Din 7 socket to flat 6-pin female connector, same as the one for heating stage)
- One BNC to double banana plug cable as shown in Figure 8 below.

Figure 8 (BNC to double banana plug cable)

Connection

1. Connect the round Din 7 plug from the opposite end of the long cable into the Din 7 socket of the shorter cable.
2. Take the long cable and plug the male Din 4 to the female Din4 on the back panel of the power booster, and insert the round 6-pin male connector into the sensor input Din 6 female connector on the Lakeshore controller back panel.
3. Take the BNC to the double banana plug cable (see Figure 8 above) and plug the BNC end into the power booster. Plug the double banana plug into the Lakeshore controller with the tabbed side in the LO. (see Figure 9 below)
4. Insert the flat 6-pin female connector of the short flexible cable into the 6 pin male connector on the back of the sample stage. In order to use a cooled stage with MAC mode operation, see Figure 10 below for cold MAC sample stage connections.

5. Other connections are the same as operating the microscope without the temperature control stages.

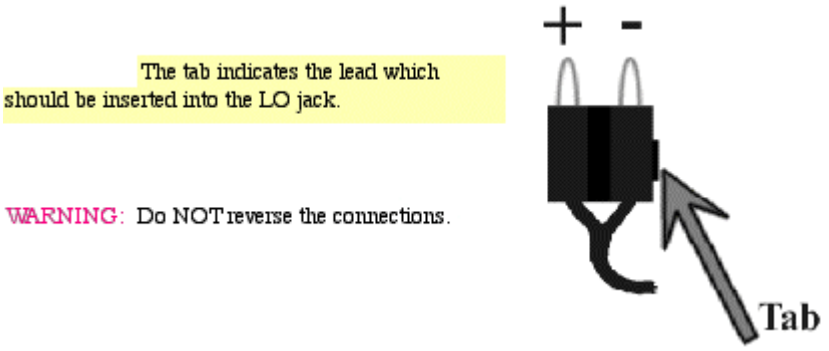

Figure 9

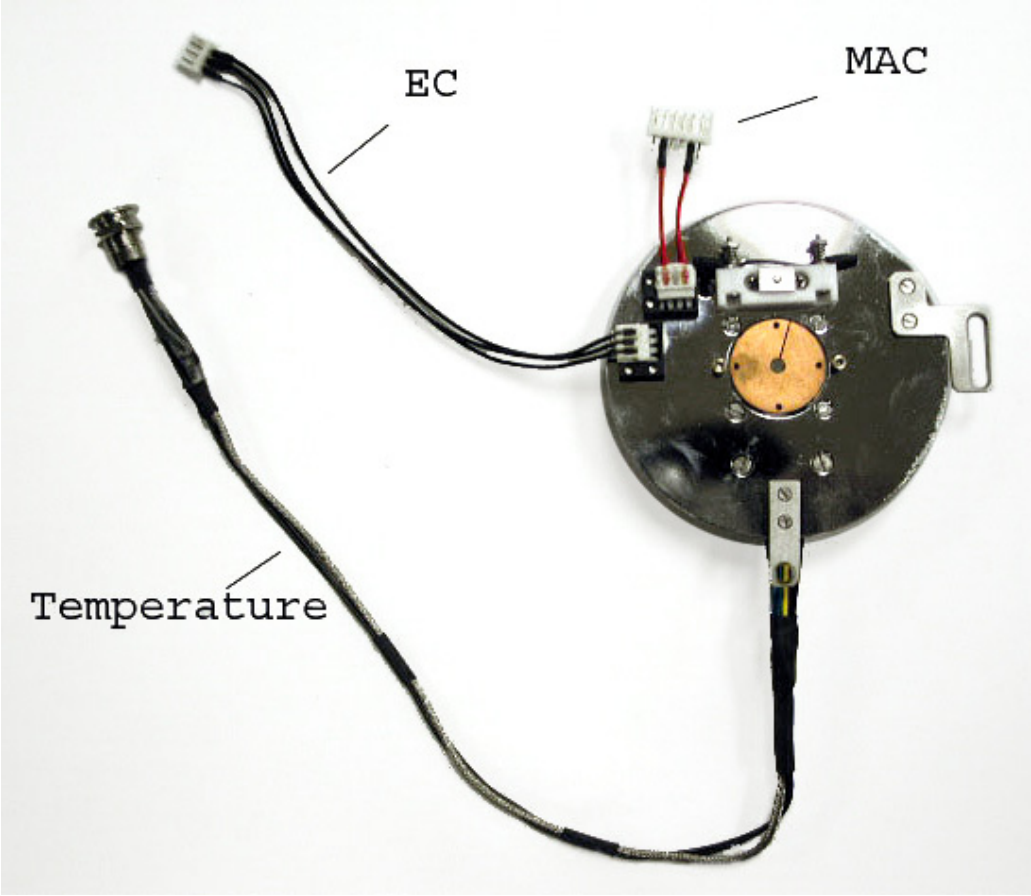

Figure 10 (Top view of cold MAC sample plate connections)

Using Lakeshore Controller and Current Booster with Peltier Temperature Stage

1. Make sure the Range adjust knob is turned to the minimum setting (counterclockwise all the way).
2. Turn on the Lakeshore controller and current booster.
3. Press Auto-Tune then press the Up or Down arrow keys until the display reads Tune: Manual.
4. Enter the proportional, integral, and differential gain. For example, to set the proportional gain press P and then enter a number followed by pressing Enter. The numbers 12, 12, and 5 respectively have worked well in previous tests.

5. Press Set Point, enter the desired final temperature and press Enter.
6. Turn the range adjust to maximum, so the current booster gets full power.
7. Press Heater Low for Model 321 or Medium for model 330 Lakeshore controller.
8. Once the temperature has stabilized, engage and scan the sample as usual. If the temperature has not stabilized, the microscope will continue to drift due to thermal expansion.

Warning: Never use Heater High with the Peltier stage.

Tips and Tricks

- Make sure there is good thermal contact between the sample and the sample plate. Mount the sample using the air cell or liquid cell even for ambient imaging. Adding an extra layer such as the double-sided tape for sample mount will reduce the thermal conductivity between the sample and the temperature stage.
- Do not forget to set the Z range to the full scale in the software before beginning scanning and changing the temperature.
- It is possible to ramp the temperature while imaging. This depends upon the difference between the initial and final temperature, the thermal expansion coefficient of the sample, the ramp rate, and the type of cantilever (for AFM). Using slow ramps, typically less than 1 °C per minute, may prevent the user from having to withdraw due to limited Z range.
- Remember: every sample will react differently under temperature control. A thin piece of graphite makes a good test sample.
- Temperature fluctuations due to excessive gains will cause the surface to appear wavy.
- Make connections to the sample and electrochemistry cell as needed (electrode connections) when combining the electrochemical and temperature controls.
- Using the environmental chamber is recommended because, generally, it can help to keep the temperature more stable. The chamber can be used to control the sample environment when, for example, the water condensation during the cooling for ambient imaging or liquid evaporation for liquid imaging during heating become a problem.