**Setting Up Degradation Experiments on Single-Composition Perovskite Samples**

Revised 29 June 2021 by Wiley Dunlap-Shohl

1. Configuring the Box and Other Equipment.

This section is only necessary if the box is not already set up to run single samples; if the correct objective, XY stage, and humidifier are all in place, then skip to the next section.

1. Install the objective
   1. **BE CAREFUL:** some of the objectives are worth >$10,000, and most are at least $2000. Do not allow the lenses to contact anything whatsoever, and never let the objective fall or drop. Always use two hands to support the objective when installing and removing it.
   2. It is generally best to set up the objective before making the box too crowded with other equipment, as the risk of dropping or otherwise damaging the lens increases with loss of dexterity. If the box is set up to run gradients (i.e. with the stress light and 100X objective), first remove the stage, then the stress light.
   3. When removing the stage, be sure to lower the photodiode first so the stage does not catch on it when you are removing it.
   4. If the sample plate is present, drive the stage all the way to the left using the X control on the joystick, and remove it.
   5. Drive the stage to the center position in X (so the movable portion is not extending past the fixed portion of the stage to the right or to the left).
   6. Drive the stage all the way to the position furthest away from you using the Y control.
   7. Insert the 5/32” Allen wrench (Thorlabs, with the yellow handle) into the hole in the front of the stage to engage the set screw, but do not begin turning it yet.
   8. Drive the stage back toward you using the Y control until it is almost touching the handle of the Allen wrench.
   9. Switch off the Tango controller to the right of the microscope.
   10. Remove the X and Y control cables to the right side of the stage. The connections to the stage are loose; avoid putting any torque on them if you can avoid it so that you don’t stress the internal wiring. **(Do not attach or remove the leads if the Tango controller is on – the wires will arc and it may damage them.)**
   11. Double-check that the photodiode has been lowered and is out of the way of the stage, and that the stage is empty of the sample plate.
   12. Unscrew the set screw holding the stage onto the microscope ring platform, and ***very carefully*** lift the stage up and remove it from the box. **Be very careful not to hit the objective.**
   13. Set the stage to the side in an unobtrusive location.
   14. If the square stress light is present, first unscrew the black external power connection, but leave it slightly undone – it is possible to partially engage the threads so that the leads are mechanically but not electrically connected.
   15. Unplug the yellow cable from the right side of the square stress light.
   16. Raise the microscope platform as high as it will go, using the black knob on the right side of the microscope base.
   17. Loosen the two screws attached to the box ceiling suspending the light from opposite corners, and carefully lower the light onto the microscope platform.
   18. Place the light and screws to the side.
   19. Lower the microscope platform as high as it will go.
   20. Unscrew the objective (***carefully!***) and return it to its container in the black bin to the right of the microscope.
   21. Attach the new objective (***carefully!***) to the microscope.
2. Reinstall the XY stage
   1. Make sure the Tango controller is still off.
   2. Place the stage on the circular microscope platform, and secure the set screw using a 5/32” Allen wrench. Make sure the stage is properly secure, but *do not use more force than necessary on the set screw or you will damage the platform*.
   3. Attach the X and Y control cables to the right side of the stage. The connections to the stage are loose; avoid putting any torque on them if you can avoid it so that you don’t stress the internal wiring. **(Do not attach or remove the leads if the Tango controller is on – the wires will arc and it may damage them.)**
   4. Turn the Tango controller back on.
   5. Confirm that the stage is operating properly by driving it with the Tango controller joystick.
3. Set up the humidifier
   1. Make sure that:
      * The muffin fan in the near right-hand corner of the box is plugged in, blowing air, and is pointed at the humid air inlet. The fan is unshielded, so watch your fingers if you need to move it.
      * The water level in the humidifier is visible in the sight glass on the right side of the tank. If it isn’t,
        + Fill the humidifier tank with water and assemble the humidifier.
        + Attach the humidifier tube to the box.
        + Seal the humidifier tube connection at the top of the tank with Parafilm.
4. Verify the electrical connections
   1. Confirm that the positive lead from the bottom Keithley is connected to one of the input terminals of the lock-in amplifier, that the other input lead from the lock-in amplifier is connected to one of the sample probes, and that the other sample probe is connected to the negative lead from the bottom Keithley.
   2. If necessary, place the 470 kΩ resistor typically used for single samples across the lock-in amplifier leads; return the resistor you remove in a safe place.
   3. Confirm that the photodiode is connected to the top Keithley.
   4. Verify that the waveform generator is connected to the reference input of the lock-in amplifier and the “Green” TTL input of the Lumencor light source.
5. Configure the software and illumination intensity
6. Fill out and run the Python scripts that generate the “Sample Info” and “Experiment Info” metadata files.
7. Make any necessary changes to the main Python data acquisition program to achieve the desired intensity, data acquisition period, and destination folder for the data. Verify that the electrical parameters (shunt resistance, lock-in sensitivity) are correct, and that the n\_gradpoints variable is equal to 1.
8. Verify that the neutral density filters are adjusted commensurately with the desired illumination intensity.
9. If making dark field or bright field measurements, set the respective variables in the Python script to True.
10. Load the sample on the stage
11. If using temperature control, make sure the heaters and thermocouples are in place on the sample plate and connected to the controller box, and that the box itself is connected to a power source. ***Make sure that the only heaters plugged into the controller box are the ones in the sample plate!***
12. Use electrical tape to secure the substrate to the aluminum heating plate and block off any stray light paths through the plate.
13. Attach the electrical probes to the Au pads, as close to the left edge of the substrate as possible. Confirm that good electrical contact has been made by putting the Keithley in ohmmeter mode and measuring the resistance of the Au pads on the sample. ***Do not forget to return the leads and probes to the standard measurement configuration afterwards!***
14. Drive the XY stage to the right, and lift the sample plate onto it.
15. Carefully drive the XY stage to the left, and place the sample plate in the pocket just to the right of the leaf springs at the near left edge.
16. Slide the sample plate to the left so that it engages with the leaf spring and does not slide around within the XY stage.
17. Set up temperature control.
18. Switch on the controller box (left side near the top)
19. Use the up and down arrow keys on the front of the controller panel to adjust the temperature set point; wait for the temperature to equilibrate.
    1. Due to the relatively narrow conductive path between the heaters and the thermocouples, the heaters will usually overshoot the set point by a substantial margin. Therefore, it is wise to bump the set point up in increments rather than setting the desired temperature all at once. For example, if you wish to set the plate at 65 °C, choose a set point of 45 °C first, wait for the temperature to stabilize, and then adjust to the final values of 65 °C. Judging from recent experience, the plate will hit 65 °C not long after being set to 45 °C. If there is any doubt, be conservative and adjust the temperature slowly!
20. **Be very careful to avoid setting the temperature too high. Never operate the system without the thermocouples in place, or the heaters could set something on fire!**
21. Focus the sample
22. Drive the stage to a position on the channel that is as free of defects as possible. Some degree of PL quenching near the edges is unavoidable, but if the quenched regions extend almost all the way into the channel across the entire substrate, it may be wise to get a new sample entirely.
23. Use the “Live” camera image in MicroManager to focus the microscope on the sample.
24. With the light on, confirm that the I-V response is linear by scanning the voltage from 0-3 V using the Keithley front panel controls. If the response is highly non-ohmic, check the contact connections.
25. Position the photodiode
26. Place the transmittance measurement photodiode underneath the sample stage, as close as possible to the bottom without contacting it. Make sure that readings are able to be taken by comparing photodiode readings with the filter cube on and off of position 3.
27. Turn the light off to avoid premature stress (particularly important if the sample is at elevated temperature).
28. Set up humidity control.
29. Change the day and night set points on the humidity controller to the desired RH percentage.
30. Close the front door to the box, and seal any remaining major gaps.
31. Turn on the power knob to the humidifier to the “MIN” setting. (Note: this knob controls the amount of power sent to the ultrasonic transducer in the humidifier; the humidity controller is a simple ON/OFF mechanism that switches the controller on when the reading is 5% below the set point and off when it is 5% above. Different power settings on the humidifier knob are appropriate for reducing excursions about different set points – for instance, you would want a higher power setting if the set point is 85%, and a lower one if it is 40%. Generally the “MIN” setting works well for most purposes.)
32. Ensure that the system is working properly, and make adjustments to the power knob as necessary to reduce humidity fluctuations.
33. Starting the run

1. Shut down MicroManager (the script will not execute properly if you don’t)
2. Run through the checklist below to make sure all systems are “go”
3. Start the run either by running the Python script cellwise, or else by clicking the green play button in Spyder.
4. Confirm that the right sequence of operations is occurring, and that the datafiles are being saved to the right folder in the computer.

**Checklist Before Starting the Experiment**

* “Sample Info” metadata file created
* “Experiment Info” metadata file created
* Sample is at the correct temperature
* Correct shunt resistor is in place across the lock-in amplifier terminals, and the right resistance in Python code matches
* Correct probe and soak light intensity settings are set for the desired illumination intensity, and the right neutral density filters are in place
* MicroManager is off
* Filter cube set to position 3
* Box air line is connected and purging
* Humidifier is operational and set to the desired humidity level
* Gas purge and humidifier are working to keep RH at the correct level
* There is enough storage space on the PC (generally 10 GB is sufficient) for the run to complete without filling up the hard drive