Standard Operation Procedure

Degradation Chamber

<Additional details: date, author?, picture>

Table of Contents

System Overview

The environmental chamber is an in-house tool for studying the degradation of perovskite films under different levels of temperature, humidity and illumination. The chamber can house up to 28 samples at a certain time – making it a vital tool in high-throughput experimental studies. Images of the samples are recorded at regular intervals of time and are uploaded into a cloud-based platform automatically. Temperature and humidity control is automatic as well. There exists also a version of the control program that can control illumination intensity between 0 and 0.15 Sun visible illumination.

The main hardware components of the apparatus include:

- Temperature and Humidity Sensor for humidity controller: Si7021
- Sample Holder
- Lamp: Advanced Illumination DL097
- Camera: ThorLabs DCC1645C (with infrared filter removed and replaced with plain glass)
- Camera Lens: ThorLabs MVL6WA
- Desktop computer fans (3)
- Temperature and humidity Tracker: Lascar Electronics EL – USB - 2
- Sample Holder Temperature Controller
- Arduino for humidity controller: Arduino Uno Rev3,
- Laptop

The degradation chamber can be used for many different experiments. Some of its capabilities are:

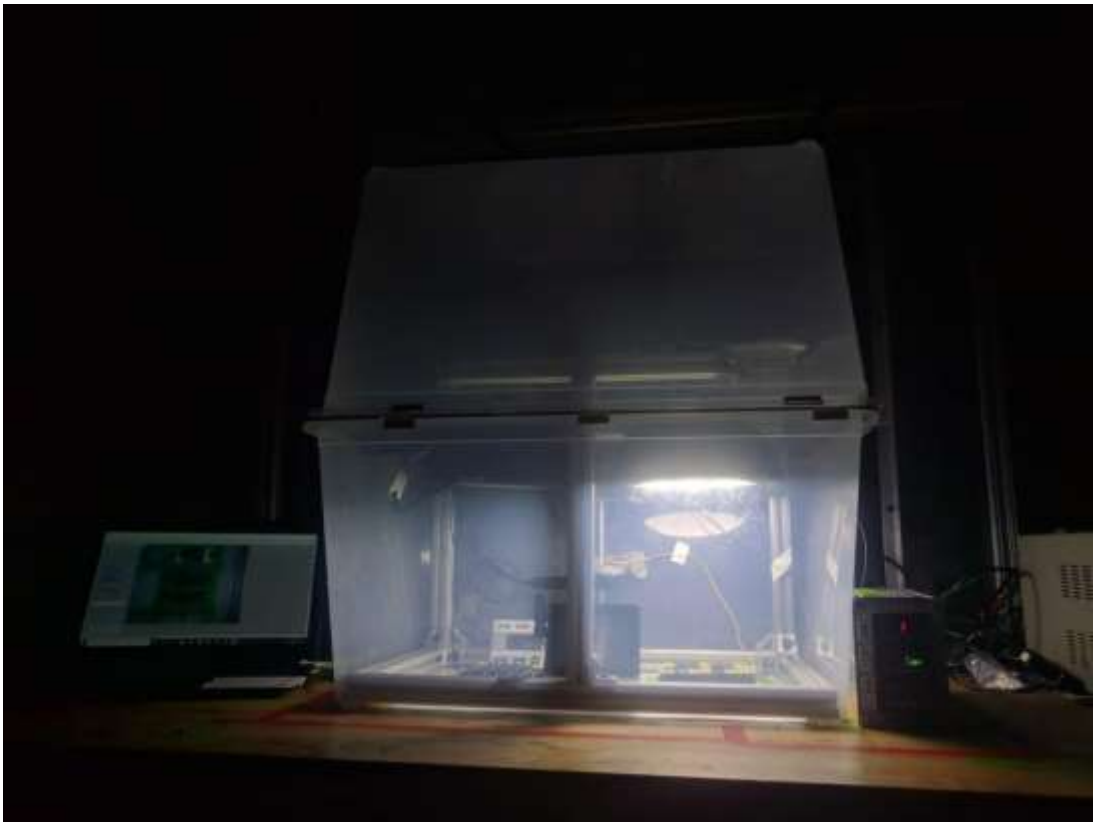
- Humidity Range: Room Humidity (~25% RH) to 85% RH
- Temperature Range: Room Temperature to approx. 100 C
- Illumination Range: 0.15 sun (0-0.15 Sun with control program version that has intensity control)
- Samples: Up to 28 (Will be increased to 56)
- Measurement Frequency: Limited by storage only

<INSERT: CHAMBER Diagram>

Fig 1: Schematic Diagram of Environmental Chamber and its Hardware Components

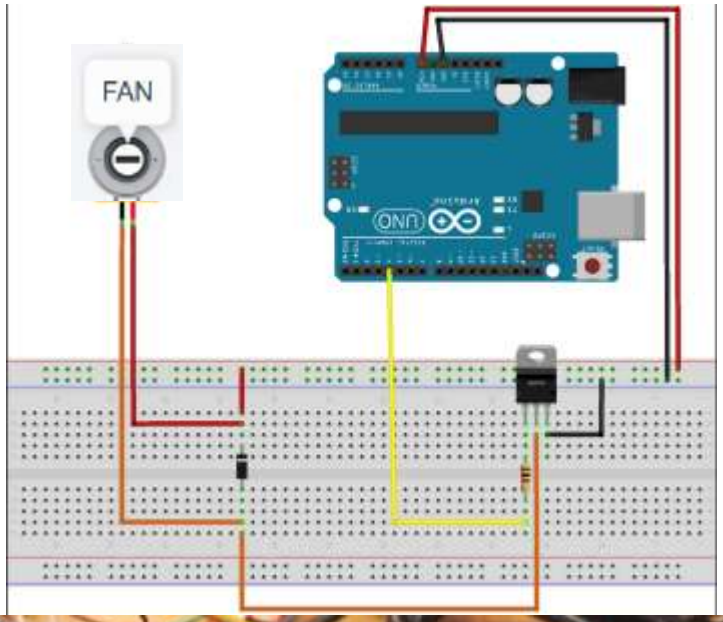
Hardware Components

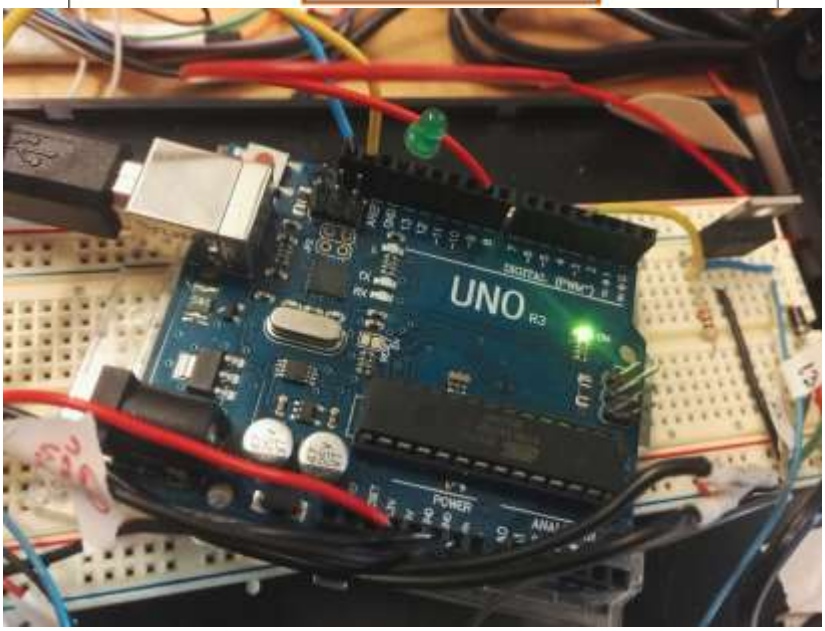
Fig: Image of Environmental Chamber (August 15, 2019)



Arduino for humidity control

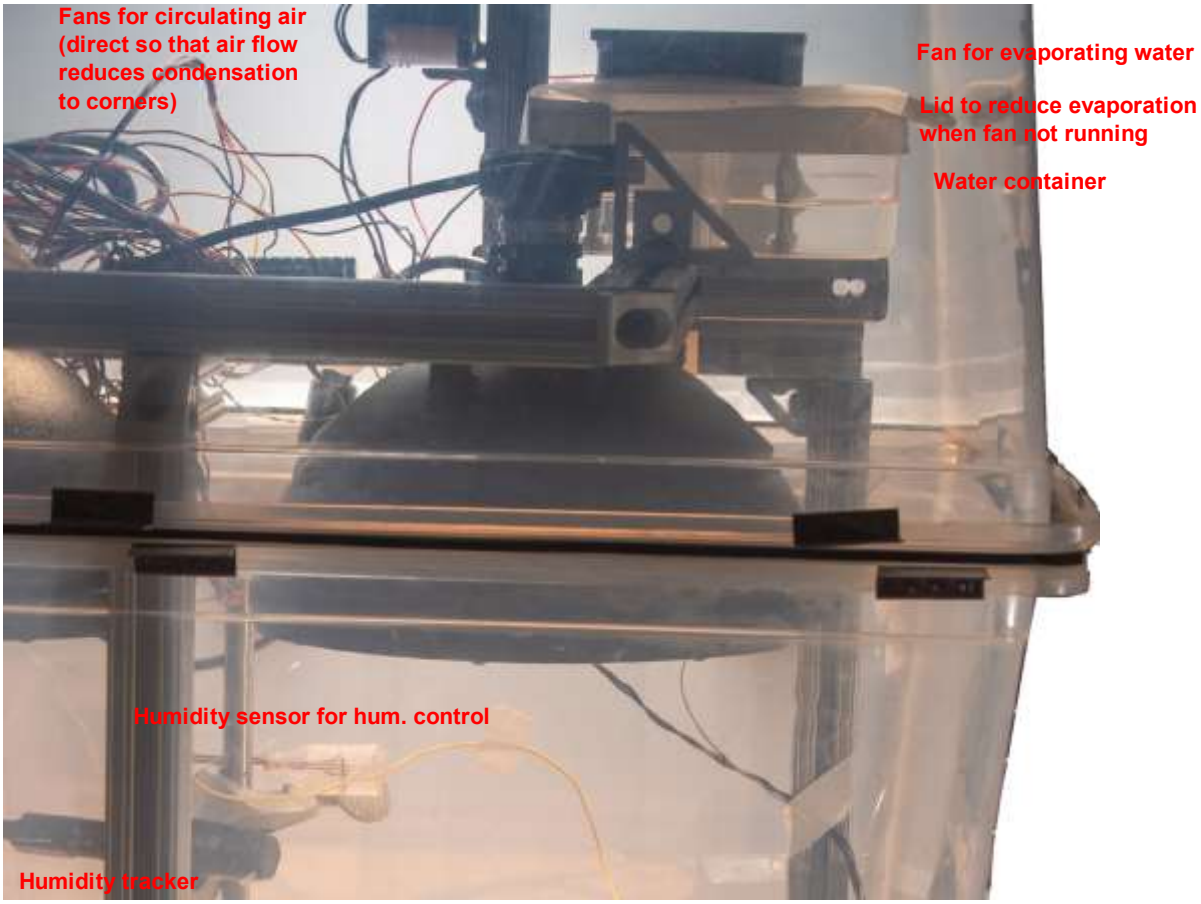
- Function: Communication with sensors and regulators
- Parts/modification:
    - Arduino Uno Rev3 SMD
- Maintenance:
    - Must be wary of
        - Freeze: recoverable by reloading boot
    - Stock up on an extra Arduino and sensor at all times
    - Can buy Arduino with a replaceable microprocessor





Temperature and Humidity Sensor for humidity control

- Function: Continuous monitoring and recording of the environmental variables in the chamber
- Parts/modification:
    - Sensor breakout board: Adafruit Si7021
- Maintenance:
    - Degrades in 1-2 months of full use. Partial recovery with rest
    - Do not place very close to the walls (condensation could result in too high humidity readouts) – the closer it is to the samples, the more it represents humidity the samples experience
    - Tested against
        - Household temperature sensor
        - Temperature humidity tracker



Camera

- Function: Fixed interval image capture of the films
- Parts/modification:
    - Customized thorlabs camera DCC1645C with infrared filter removed and replaced with plain glass
    - Inserted through the hole at the top of the lamp dome
    - MVL6WA lens for focusing
- Maintenance:
    - Clean frequently to avoid any dust or other accumulation in outer lens
    - Ensure tight sealing to avoid any moisture or other accumulation within the camera

Fig: May, 2019 of the parts in the first setup

Circulation Fans

- Function: Provide circulation/flow in the chamber, and ensure humidity and temperature homogeneity
- Parts:
    - 3 fans (small cpu fans to distribute humidity)
    - 2 for general air circulation – run all the time – position optimized by Jim
    - 1 for humidity control – controlled by Arduino
- Maintenance:
    - Please set a fixed speed after calibration
    - Ensure proper and steady power supply and operation

Housing setup

- Function: Houses the whole setup with an isolated environment
- Parts:
    - Two sterilite 66 quart transparent containers (example)
    - Metal prongs and clips to hold two containers together
    - O-ring type material (water-proof and spongy) as a sealant between two containers
    - Thick curtains to block external light and atmosphere (example)
- Maintenance:
    - Replace sealant material as they wear off
    - Line corners with paper towels to avoid condensation in corners
    - Do not shake or jolt the setup or table vigorously

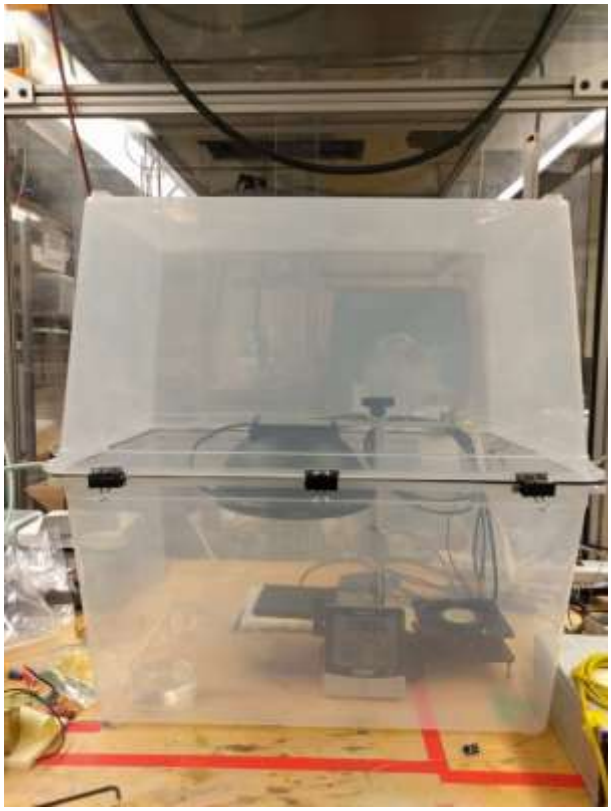
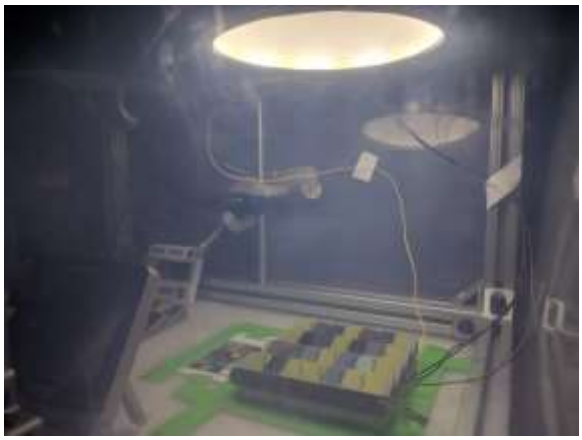


Fig of the housing setup from Jan, 2019

Lamp

- Function: Provides light exposure for degradation and illumination for image capture
- Parts/modification:
    - Advanced illumination DL097
- Maintenance:
    - Ensure all LED lamps are operational
    - Measure intensity frequently as it the lamp deteriorates over time
    - Replace as necessary
- Illumination intensity during aging tests is followed by a color calibration chart printed on photo paper and placed next to the sample holder – if RGB samples of the chart change during the aging test, there is something wrong with the lamp.



Sample Holder Plate

- Function: Holds the samples, allowing exposure to environment variables and continuous monitoring.
- Parts/modification:
    - Made of graphite, built in-house by Jim
    - Modifications below are for our samples/needs and can be changed as necessary
    - Four rows, each cut at a fixed angle to avoid direct reflection back to the camera
    - Seven sample sections per row separated by metal pins, which also help with grounding
    - A hole at the bottom for thermocouple provides temperature reading of the plate
    - Supported atop a heating mesh, separated from plastic with an insulator material
- Maintenance:
    - Ensure thermocouple is properly inserted
    - Ensure proper grounding

Temperature and Humidity Tracker

- Function: Physical sensor for tracking environment in the chamber
- Parts/modification:
    - EL – USB – 2 by lascar electronics
- Maintenance:
    - Replaceable ½ AA battery – every 6-12 months
    - Only switch on USB when reading data
    - Software: EasyLogUSB
    - The sensor needs to be close to the sample holder without casting a shadow on it

Miscellaneous

Another humidity tracker for validation and visual confirmation

- Breadboards
- Jumper wires (rolls of small wires work too)
- Small plastic bowls for holding water in the chamber
- Nitrogen/dry air supply (for purging the chamber of humidity)
- Stopwatch
- Diffuser, if there is reflection from the sample holder
- Aluminum frames and scaffolds for holding setups
- Grounding cables
    - Ensure grounding of all the parts that may become in contact to each other during the aging test (e.g. if table is shaking) to the same point without creating ground loops. In this implementation these are: aluminum frame, camera + laptop, and lamp. Check also fans and sample holder heating. If the components are not grounded properly, voltage peaks could result in the lamp flickering occasionally (i.e., poor data quality).

Safety Precautions

- Perovskite materials, especially those consisting of lead, are toxic materials. Please wear gloves at all times, not only when handling samples but also when touching other surfaces on the chamber, these may have some lead on them.
- The electrical grounding is this part of the laboratory is not well done. When handling the plugs and switches of the various hardware components, be wary of electric shocks.
- The sample holder can heat up to high temperatures thus exercise precaution when touching the holder or any surfaces around it.
- As required by laboratory rules, wear goggles and lab coat at all times.

Operating Procedure

Notes are to be made in the “start and end values” text file. Paste a copy of this file into the Dropbox folder in which the images from your test are going to be saved.

Initial preparations

- Open the EasyLog Humidity Tracker App on the computer. Stop the humidity meter, collect the data and save it on the laptop, and restart the humidity meter. Disconnect the USB cable of the humidity meter, and check that the green LED of the humidity meter blinks in every 1 min.
- Open the Arduino program for humidity control (through fan speed control). Navigate to Arduino/Tools/System monitor and check that the humidity control system reads a valid number (humidity <100%). Adjust the desired humidity level here, accounting for error in the reading of the humidity tracker (Ex: If the Humidity tracker reads 88%, the humidity sensor reads 85%, then the error in the humidity tracker is approximately +3%. If you want to set the humidity to 50%, you must enter – 53%).
- Open the LabView camera control program. Settings for the camera will vary with experiment. You will need to perform a calibration procedure to determine these values (reference:24/15/7.35). Enter the folder path to your desired Dropbox folder. Enter frequency of capturing images. Check that the data is saved into the same folder.
- Turn the lamp on. Remove the sample holder from the picture area, but leave the insulating pad there. Place the Xrite color chart to the picture area so that its lower right corner (i.e., the corner with white color patch) is aligned with the green tapes marking the sample area and it is aligned straight in the live stream of the camera program.
- Open one of the color chart pictures with GNU Image Manipulation Program (GIMP). Navigate to Colors -> Map -> Color Exchange option and check that the white color patch is not oversaturated. This is just to check the camera settings; you need not save the changes to picture.
- Turn on the large fan. Null the timer and fill the water container until the level marked in the container (for experiments with higher than room humidity, otherwise clean to complete dryness). Check that there are no wires that could cause shadows or reflections on the samples.

Putting the samples in

- Insert all the samples in and log the sample order at the end of the “start and end values” text file. Ensure that you are wearing gloves and place the samples on the holder very carefully using tweezers. Avoid dropping the samples or getting scratches on them as this could impact the degradation process.
- Check that the sample holder is aligned straight in the picture and it is positioned into the sample area (green tapes) in the LabView Live Camera View
- Turned on the sample holder heating and set to the desired temperature. Start the timer, and turn on the humidity control system (check).
- Close the lid and put the curtain on the apparatus.

Winding Up

- As soon as the curtain has been put up, note the temperature reading, humidity reading amd sample holder temperature into the test file. Do the same after 10 minutes and after an hour (optional)
- Check that images are being loaded into the Dropbox folder and that everything is running fine.
- When you are ready to end the test, note the duration of the test, temperature reading, humidity reading and sample holder T into the text file.

Shutting Down

- Turn off the parts of the chamber in reversed order compared to Initial preparations.
- Remove the lid carefully and without shaking in case that there is condensation on the lid that could damage the samples if drops are falling on top of the samples. Don’t breathe in the fumes if you smell anything.
- Remove the samples.

Data Analysis

Python scripts are available to convert the images to time-series of RGB values or LAB values, with or without color calibration.

Training Roles and Responsibilities

Training

- New users should start by reading this SOP. They should also accompany a current user and watch an entire degradation test run to learn practical tips.
- The tool owner will train new users by guiding them through a measurement, and then being present the second time they run the tool on their own.
- Many of the steps take a while to master—practice a few times on non-critical samples first.

Responsibilities of Tool Owner

- The tool owner is responsible for training new users for routine experiments, maintaining and changing the lamp, and keeping things clean (e.g., dust builds up on the optics).
- Individual users may manage their own Labview code and data on the local computer, as well as having their own specific sample mounts. They are in charge of keeping these stored properly.
- For non-routine activities like calibration of the camera, changing batteries on the humidity tracker, clearing up space on the laptop, the tool owner should be present.
- Lab members familiar with the tool: Armi Tiihonen, Titan Hartono, Janak Thapa, Shijing Sun, Richa Naik

Tool Monitoring and Records: Tips

Temperature and Humidity Sensor

- Adafruit Si7021
- Degrades in 1-2 months of full use. Recovers with rest (not verified)
- Tested against
    - Household temperature sensor
    - Temperature humidity tracker
- Change when % change in humidity read is over 15%
- Temp Hum tracker does not have a good software interface, more stable though

Arduino

- Arduino Uno Rev3 SMD
- Must be wary of
    - Temporary freeze: recoverable by reloading boot
    - Full freeze after a voltage spike (e.g., when replacing humidity sensor and connecting wrong): irrecoverable, microprocessor gets spoilt
- Stock up on an extra Arduino and sensor at all times
- Can buy Arduino with a replaceable microprocessor
- Arduino gets its power from the USB plug, temperature controller from the other plug point
- Both of these circuits must have the same ground
- Circuit diagram: Shown in the preceding documentation
- More wires, numbered
- Arduino program in Github
- Future: connect Arduino + LabView

Humidity Tracker

- EL – USB – 2 by lasear electronics
- Replaceable ½ AA battery – every 6-12 months
- Only switch on USB when reading data
- Software: EasyLogUSB
- Voltage peak: remove battery for a while
- The sensor needs to be close to the sample holder without casting a shadow on it

Fans

- 3 fans
- 2 for general air circulation – run all the time – position optimized by jim
- 1 for humidity control – controlled by Arduino

Electrical Connections and Grounding

- No circuit diagram : wires numbered
- <INSERT: Diagram >: obtain from Alex

References