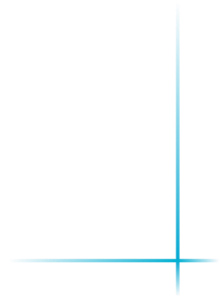


Crystal Chamber

Documentation



Version 1.1.G

2022 - 2024

Crystal Chamber Project, a thermodynamic control chamber controlled by Arduino for nucleation and crystal growth from saturated saline solutions by evaporation method.

Designed and built by Vinícius Coelho (Voelho)

Main sketch version: Crystal Chamber G – 04/12/2024

Thermostat sketch version: CC_E – 13/10/2024

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1. COMPONENTS

Item	Qty
Arduino UNO	1
400-point breadboard	1
12V 10A Power supply	1
XL4015 step-down module	2
Fan + coolers	2
Peltier element 12705	1
SG90 microservo 0 - 180°	2

Item	Qty
DHT22 sensor	3
Mini fan 4 x 4 cm 5v 0,14A	1
Relay module	3
MicroSD module	1
128 x 64 OLED display	1
Buzzer	1
Buttons, cables and resistors	-

2. LIBRARIES

Default operation

Arduino.h		Basic library
DHT.h	1.4.6 Adafruit	DHT reading
PinButton.h	Multibutton 1.2.0 Poelstra	Button input control
SD.h		SD card logging
SPI.h		Serial communication (for debug)
U8x8lib.h	U8g2 - 2.34.22 Oliver	Display with low memory demand
VarSpeedServo.h	Version 2 – 2013 Philip van Allen	Servo control

Alternative

HX711.h	HX711 0.7.5 Bodgan	Load cell interaction for scale usage
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3. PINS

Default operation

Pin	Connection	pinMode
2	DHT 2	-
3	RPM for ext. fan	-
4	TEC	output
5	DHT 1	-
6	DHT 3	-
7	Buzzer	-
8	SD - CS	-
9	Servo 1	-

Pin	Connection	pinMode
10	TEC fans	output
11	SD – MOSI	-
12	SD – MISO	-
13	SD – SCK	-
A0	Inner fan	output
A1	BT Up	input
A2	BT Mid	input
A3	Servo 2 - Thermostat	-

4. ALGORITHM

This section describes the operation of the control algorithm in the execution sequence of the programs in a comprehensive manner. More details are documented in the Sketch itself.

4.1. Operation states

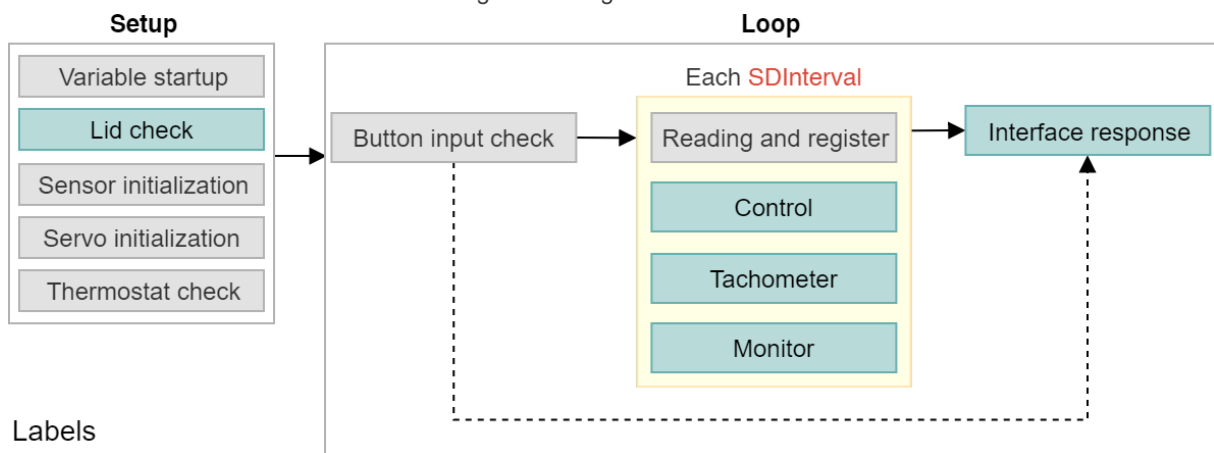
There are 5 operation modes, for different exposure conditions or adjustments

Mode	OPE	Indicator	Description
Maintenance	0	MAN	Mode where the peripherals are turned off for the physical operation of the chamber in case of maintenance, observation, or adjustments. Useful for monitoring crystallization conditions, as access to the humidity chamber is required.
T control	1	T:	First stage of the operating mode, adjusting the temperature range for later humidity adjustment.
U control	2	U:	Second stage of the operating mode, allows humidity control through drying, but only within the established temperature range.
Temperature only	3	T_	Controls only the temperature within the determined range. Useful for sensor calibration
Ventilation	4	—	Ventilation mode does not activate the thermal or drying control. It is useful for sensor calibration or exposing the solution to a temperature close to ambient.
Humidity only	5	_U	Controls only the humidity within a specified range. It varies depending on the behavior of the DHT sensors at different temperature ranges. Useful for sensor calibration.

4.2. Default exposition

The system operates by checking the operating conditions at each *SDInterval*, adjusting the controls and performing safety checks. The initialization assumes operational autonomy in the event of interruptions or power outages, so several verification processes are given execution priority.

Diagram 1 – Algorithm overview



Labels

- Process with response
- Process without response
- Parameter

4.2.1. Setup

The thermal control is initialized as off.

The lid verification also prioritizes operational safety, ensuring that the fan on the hot sink of the TEC (the Peltier element) is working properly to prevent overheating. If there is a reading on the tachometer, then the box is closed, and the external lid is connected to the chamber. The operating modes with thermal control require the lid to be connected, while the calibration mode requires the lid to be open, to prevent overheating from the internal fan of the humidity chamber.

The servo controlling the humidity moves for a visual check of its operation, as it is crucial for humidity control.

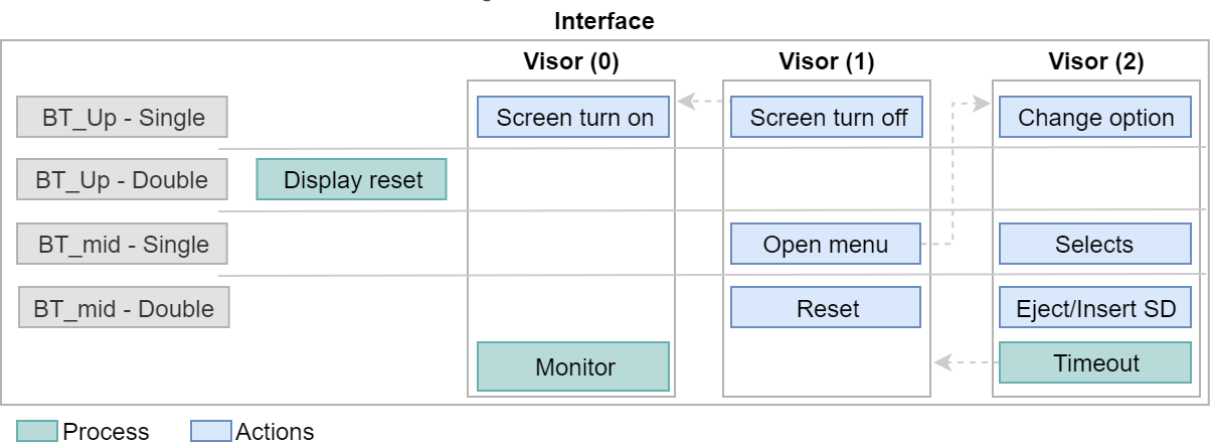
4.2.2. Button operation

The interaction of the buttons is checked every Loop cycle. If there is activity, the response will depend on the display status (variable *Visor*). A double-click on the *BT_Mid* button during *Visor 1* calls the function *retomaOp*, to force a reset on the readings and the *Monitor* state.

A double-click on the *BT_Up* button invokes a display reset, as sometimes the connection to the Arduino can be corrupted by loose wires or some movement in the LCD display terminals.

The variable *intInterval* controls the timeout period of the menu, returning to the operation screen.

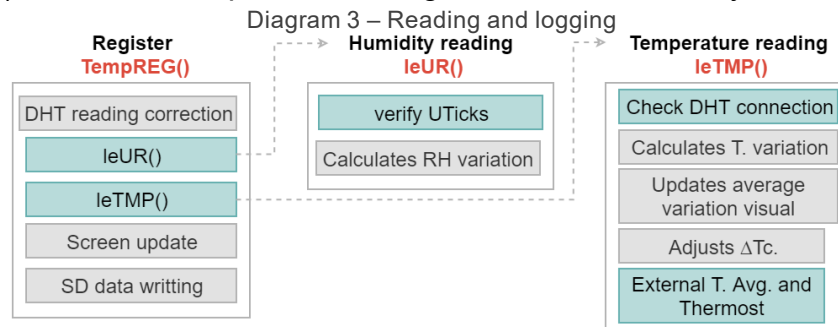
Diagram 2 – Interface actions.



4.2.3. Reading and Logging

Temperature and humidity readings are performed by the function `TempReg()`. At this stage, the calibration equations for the DHT sensors are applied, and the data is displayed if `Visor = 1`. No reading or logging is performed in maintenance state (Topic 4.1).

If the SD card is connected (`vSD = true`), it is checked if it is time to register the information (`SDticks = 0`); if not, the skipping of the interval is recorded. The delay prevents the writing of multiple data entries within a relatively short interval (every `SDInterval`), which would produce a long file with unnecessary information.



Humidity reading (`leUR` function)

The humidity reading is also delayed (`UTicks`) to reduce the influence of momentary fluctuations caused by small temperature variations or operational conditions.

Temperature reading (`leTMP` function)

Faulty wire connections or loss of connection with the DHT sensor in the exposure environment (indicated to the monitor with `MoSt = 7`) are verified. Instant readings are also performed, along with the updating of moving averages and the visual indicator of variation (item 6.1).

This function also calls the adjusts of thermostat correction factor. Sometimes, the calibration curve is not yet aligned with the operating conditions, and deviations from the desired temperature (`Talvo`) may require adjustment. In this case, `DT` (temperature difference between external and internal environment) is compensated with a value `DTc`, equal to the last adjustment value plus the difference between the stabilized temperature and `Talvo`. This adjustment is made every `varInterval` according to the actual condition (if `DTc < 0` and the temperature is rising; if `DTc > 0` and the temperature is falling; or when the temperature is stable), while observing the angular limits of the thermostat servo.

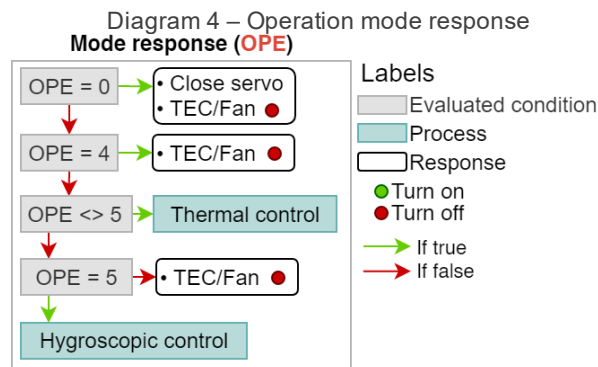
That is, `DTc` is not adjusted to higher values if the thermostat is already at maximum power (180°), nor to lower values when the thermostat is already at minimum power (0°).

4.2.4. Control

Default operation

The response of the control system depends on the current operating mode, variable *OPE* (Item **Erro! Fonte de referência não encontrada.**), with priority given to maintenance mode, which is also used to interrupt operation in case of any safety condition violation.

Safety conditions are checked by the *Monitor()* function, aiming to prevent scenarios that could be unfavorable to evaporation or crystal growth stability (e.g. very low relative humidity).



Thermal control

Given the dependence of relative humidity on temperature, the aim is to stabilize the exposure within the target range before starting the humidity control. A moving average (*T.méd*) is used as a reference for monitoring to minimize the influence of sensor fluctuations.

The events marked in the diagram are illustrated in the temperature scenario charts with the possible behaviors of the average temperature. Given the assumption of constant temperature adjustment, only a critical error signaled by the *Monitor()* will deactivate the humidity control mode once it has been activated.

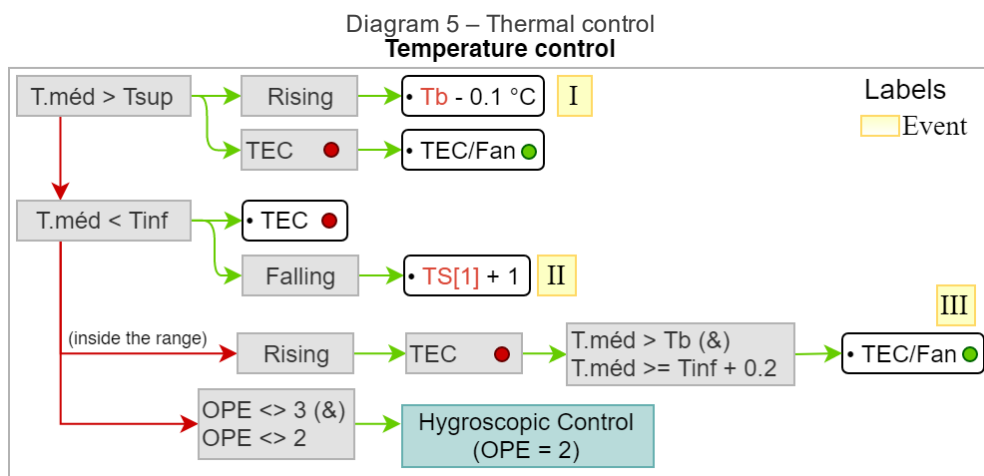
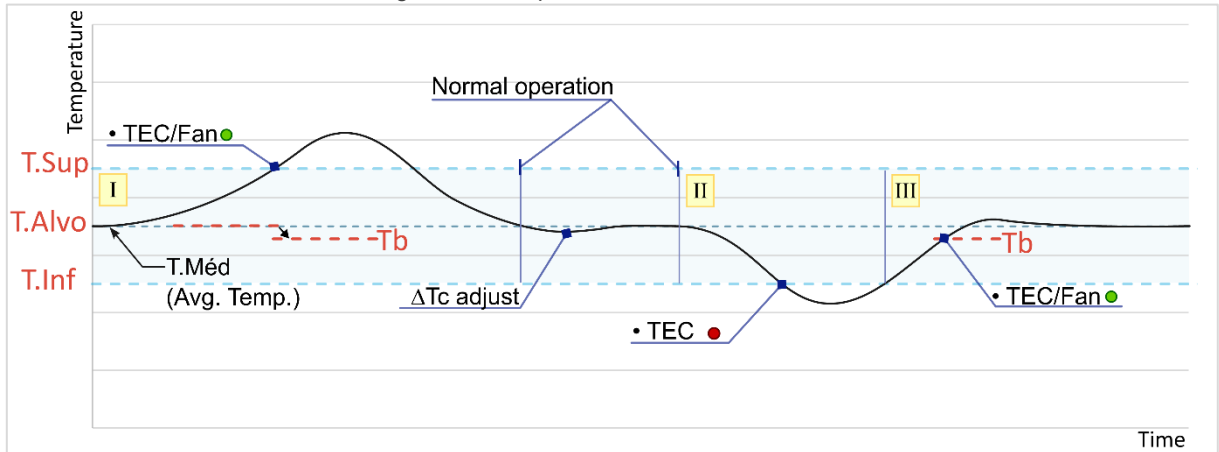


Figure 1 – Temperature variation scenarios



Hygroscopic control

The hygroscopic control regime seeks to adjust the servo's opening to bring the chamber's relative humidity closer to the target value (Ualvo). The equilibrium value of the exposure depends on the evaporation of the saline solutions and the adsorption of silica gel. The first is a function of the exposure area, the amount of solution and its concentration, and the relative humidity itself, while the second also depends on the silica gel's saturation level - interrelated and complex parameters, also dependent on temperature. Therefore, equilibrium is dynamically achieved by observing how a certain trapdoor opening alters the chamber's humidity and adjusting it to maintain the expected levels.

In summary, the control is based on the adjustment of drying intensity. Increases in relative humidity are due to the evaporation of the solution, mainly when the *trapdoor* is at the minimum position.

Diagram 6 – Hygroscopic control

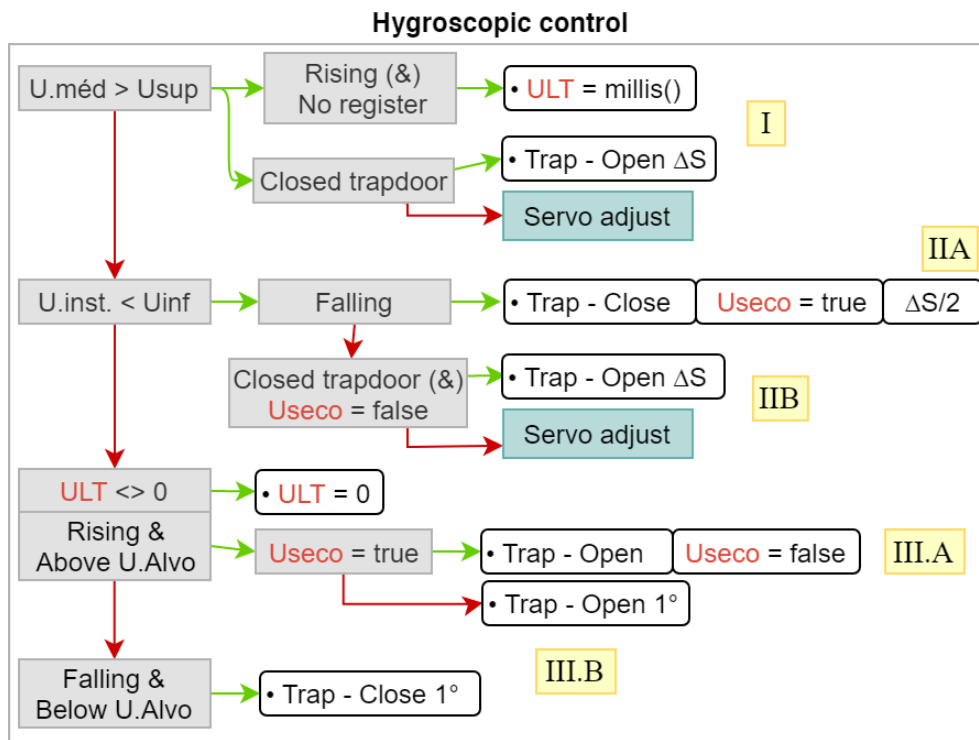
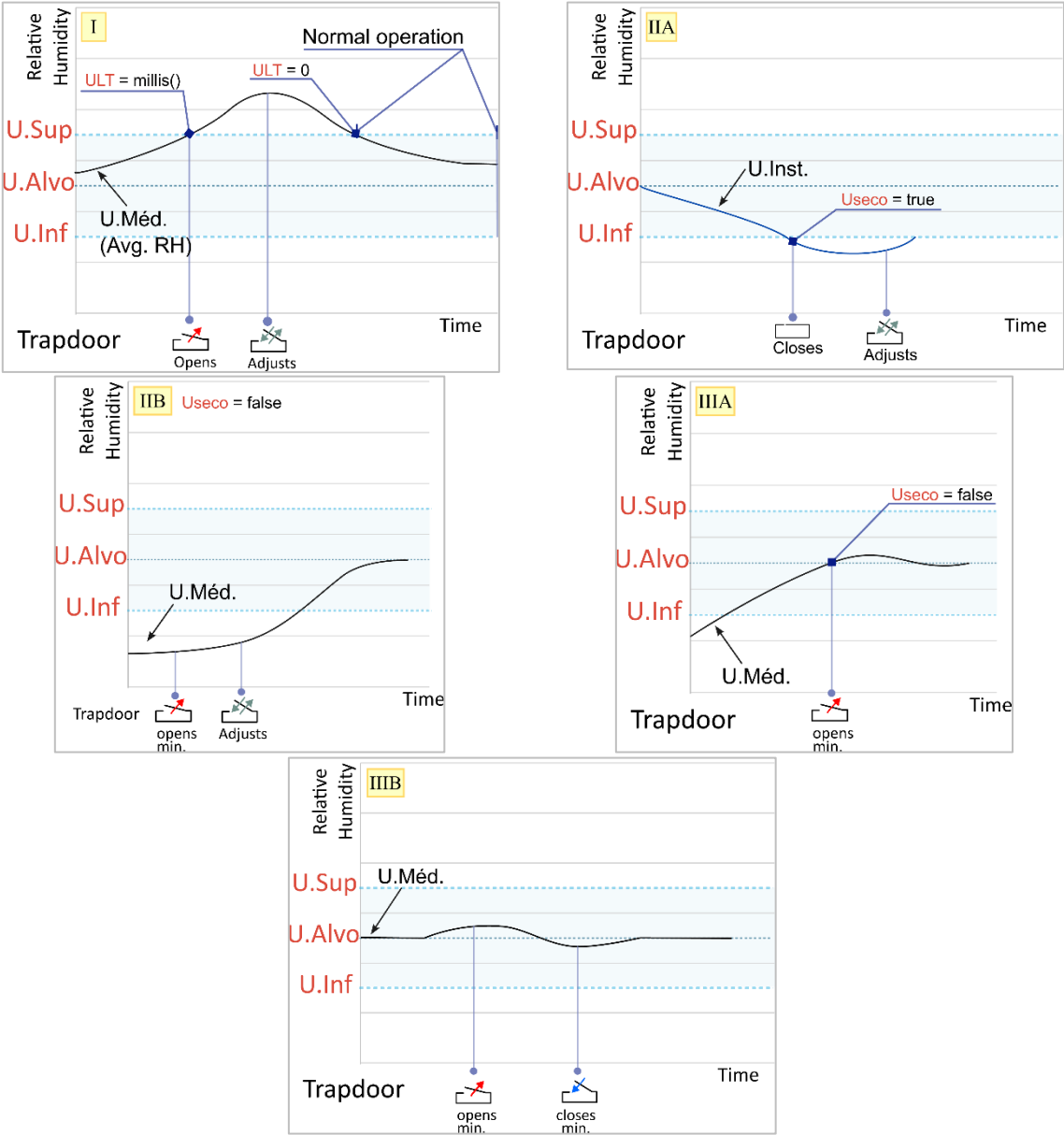
 ΔS = servo variation in degrees

Figura 2 – Humidity variation scenarios



4.2.5. Monitoring

Safety and stability checks are carried out through the Monitor() function every SDInterval if the screen is on, and every cycle of the Loop function if the screen is off. Any violations of the verified situations are reported as errors via the MoSt variable (acronym for Monitor Status).

If an alert is triggered, the display is turned on and an alert tone is emitted by the buzzer.

MoSt	Situation
0	System in maintenance, readings are interrupted, and components are deactivated, allowing for the removal or modification of components without corrupting the readings or operation
1	System returning from maintenance mode, tolerance is allowed for some temperature-related checks, such as the one that triggers <u>MoSt = 6</u>
2	<ul style="list-style-type: none"> ❖ The temperature has been above the operational range for more than 4 hours. Possible low cooling power situation. ❖ If the instantaneous temperature is 0.3 °C above <u>Talvo</u>, 30 minutes after a thermostat average reset was performed when this limit was first detected.
3	Humidity is above the upper limit for more than two hours. Possible saturation of the silica gel to a level where it no longer adsorbs sufficiently, signaling the need for replacement.
4	More than 4 occurrences of average temperature below <u>Tinf</u> . Possible situation of excessive cooling power.
5	External fan RPM lower than the threshold for 1 minute. Possible failure in the connections or the fan, which could cause the system to overheat or lose efficiency. This is a critical situation that triggers maintenance.
6	Internal average temperature 2 °C above <u>Tsup</u> and rising. Possible component failure, critical situation that triggers maintenance.
7	The temperature reading is equal to zero. Possible loss of connection with the sensor or its failure. May trigger maintenance if associated with the condition <u>MoSt = 4</u> .

4.2.6. Operational notes

retomaOp

This is the function that resets the operation, clearing most control parameters.

It's called after a maintenance state or by manual reset through double-click on BT_Mid

In modes 4 and 5, the inner fan is only turned on if the lid is absent

The SD card performs a buffer flush every SDticks cycles to ensure that data is written and not lost in case of a power failure or other interruption.

The interface with the card is only completed with the **operaSD** function, which is responsible for finalizing and reinitializing the card with the system, allowing for temporary removal during operation.

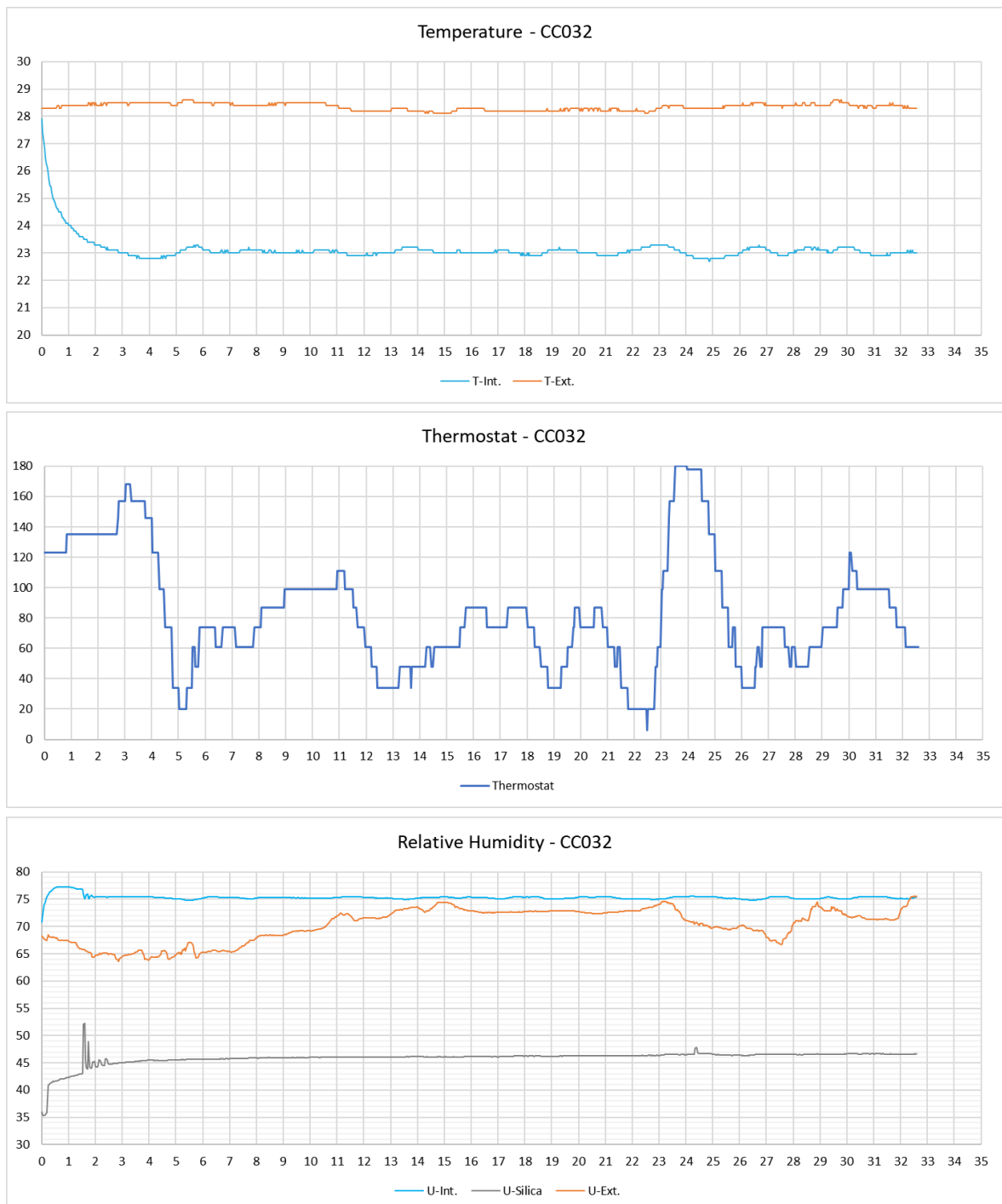
The servo adjustment function (**ajServo**) includes a damping system to prevent abrupt humidity variations that could compromise crystal formation, also ensuring that the variation is not too small when far from the target range, otherwise the exposure would take too long.

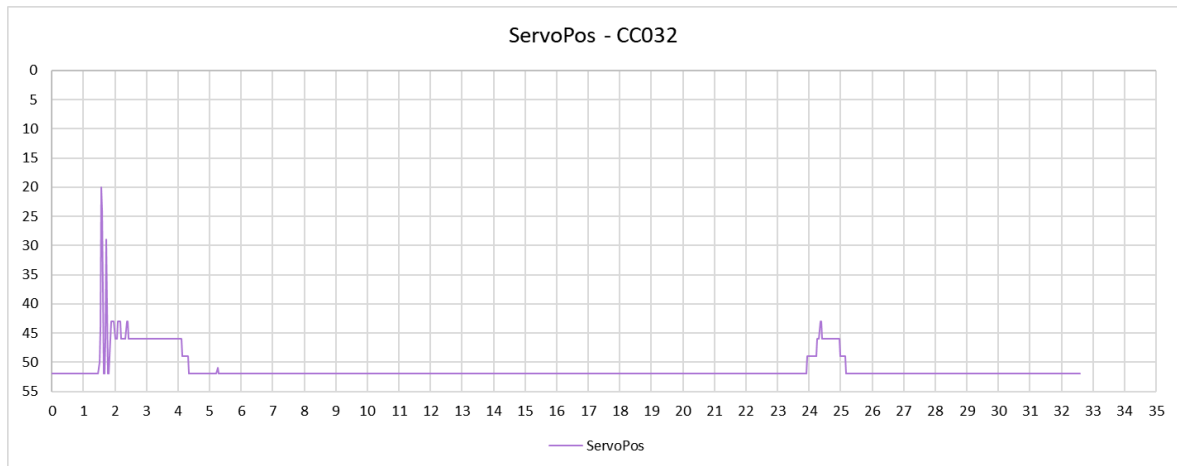
The limit values are relative to the difference between the average humidity and the target range during the last recorded check every Uticks cycle. By default, the control ranges are: above 5%, between 2% and 5%, and below 2%, with the maximum allowed variations being: 1%, 0.5%, and 0.2%, respectively.

4.2.7. Typical behavior

The charts below show the typical behavior of chamber conditions. Exposure was set to *Talvo* of 23,0 °C and *Ualvo* of 75.4%. After reaching equilibrium, the internal temperature oscillated by $\pm 0,3^{\circ}\text{C}$ while relative humidity remained around 75.2%. It's interesting to observe that the most expressive variations occur smoothly after the equilibrium point, ensuring that the exposure environment does not undergo thermodynamic shocks that could compromise crystal formation.

The relative humidity evolution of the silica gel container is also presented, which is useful to determine when the desiccant is saturated. With the trapdoor closed, there is minimal air transfer, leading to a gradual increase in the humidity. When open, the response is rapid, justifying the constant need to adjustment of the opening angle.

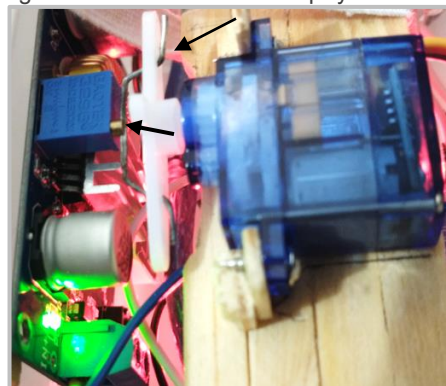




4.3. Thermostat adjustment

The cooling power is controlled by a servo (*thermostat*) physically connected to the power supply that feeds the TEC. The servo's angle determines the output voltage of the power supply, which in turn determines the cooling power associated with a specific temperature difference between the external environment and the interior of the chamber (*DT*).

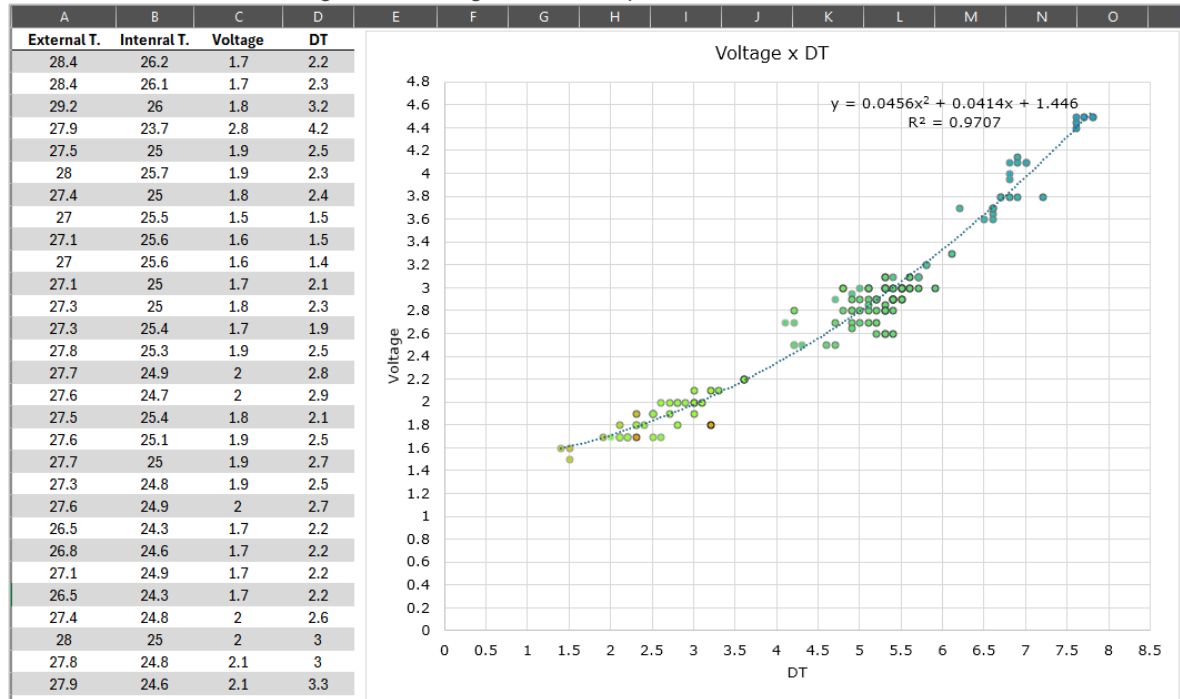
Figure 3 – Thermostat servo physical setup



It is important to note that the physical arrangement of the chamber, especially its insulation capacity, is the primary factor related to the system's thermal performance and its ability to respond to variations in ambient temperature.

A period of observation and data collection is required to correlate the servo angle with the TEC power and the internal equilibrium temperature: First, the equilibrium values of the internal temperature must be observed according to the circuit's supply voltage, logging the ambient temperature, internal temperature, and applied voltage. This allows for plotting a graph that relates the voltage to the temperature difference (between external and internal) and obtaining a regression curve (**voltage(DT)** function in quadratic regression using Excel at Figure 4).

Figure 4 – Voltage versus Temperature difference chart



Second, to incorporate the thermostat control, it must be remembered that the servo's position varies between 0 and 180°, so the physical arrangement of the power supply should be positioned as close as possible to the region of interest concerning the temperature difference, with some margin for adjustment throughout the daily variation.

For example, if the chamber is to be operated with the curve presented in Figure 4 at 23°C with an average ambient temperature of 28°C, resulting in a 5°C difference, the voltage adjustment screw should be positioned near the 2.8V setting, aligning the servo to 90° at this position.

Next, the relationship between the servo angle and the resulting voltage must be known. The simplest way to obtain this data is to vary the servo position, record the resulting values (see **thermostat sketch**), and determine the function that describes this behavior (Figure 5 – **degrees(voltage)** function obtained by linear regression).

Figure 5 – Linear regression of angular position x voltage for a thermostat servo setup

Servo		Degrees (voltage) - linear	
Degree	Voltage	a	b
0	2.5	369.7916667	-942.60417
60	2.7		
120	2.9		
20	2.7		
80	2.7		
140	2.9		
40	2.7		
100	2.8		
160	3		
180	3		
10	2.6		

By applying the **voltage(DT)** function in the **degrees(voltage)** function, the **degrees(DT)** function is obtained, which is then inserted into the Sketch in the **Thermostato()** function.

Figure 6 – Quadratic coefficients for a thermostat adjustment equation

	a	b	c
G(DT)	-19.1	310.3	-983.4

Observations

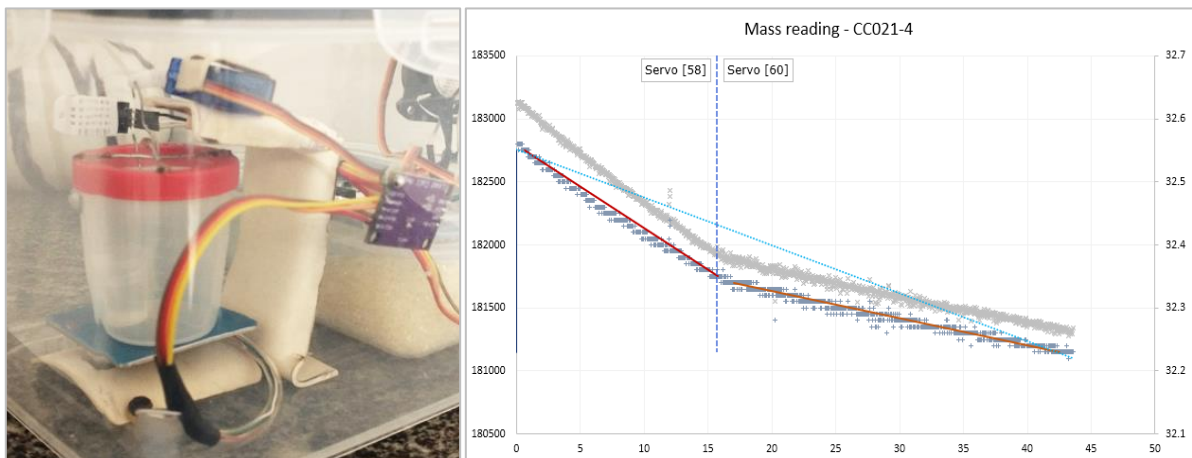
- ❖ Since the thermostat's position is limited by the physical arrangement, a new degrees(DT) curve should be obtained whenever the operating temperature range is changed beyond the current domain or when there is a seasonal change in the average external temperature.
- ❖ Despite the dynamic temperature adjustment (via the DTc difference), variability arises from the low precision of the power supply adjustment and other associated factors, which can delay the adjustment process. Therefore, it is recommended to regularly collect thermostat adjustment data (external, internal, and voltage) under equilibrium conditions (recommended VarV of 11xx or more stable). This allows for obtaining a more accurate voltage(DT) curve and, consequently, a more precise adjustment.
- ❖ The stability of the internal temperature depends on the thermal energy balance of the chamber (with particular emphasis on the thermal conductivity of the arrangement). Data collection at various voltages can generate a characteristic curve that helps determine if the desired working range is achievable. Similarly, if there are modifications to the chamber structure, a new curve should be calculated.

4.4. Operation with a scale

An alternative arrangement for the chamber consists in using a HX711 module with a Load Cell to measure the mass variation of the saline solution directly, observing the real evaporation rate in function of the exposure conditions.

Experience shows that to reduce the error associated with the Load Cell, it's necessary to calibrate the sensor before each reading, avoiding deviations and error accumulation over time. Thus, it's necessary to remove the solution container from the scale plate, turn it on, calibrate the scale, and only then place the container back and perform the weighting. The adopted answer to this was to utilize a servo to lift and lower the container during these stages.

This variation uses a specific sketch and requires almost all the Arduino available pins. Furthermore, there are requirements on the servo support, Load Cell support structure, and the suspension element for the container which are not part of this documentation. Below is illustrated the tested arrangement and a typical result curve of mass reading over time.



5. VARIABLES AND PARAMETERS

The system prioritizes the adoption of integer arithmetic logic during the calculations and verifications, to save memory and optimize the Arduino UNO processing. Floating-point calculations are used only at specific times. Default variable values are highlighted in bold in the description below.

5.1. Operation

Type	Name	Value	Description
unsigned char	intLim	0	Registers the amount of menu entries
	OPE	0 – Maintenance 1 – Temperature control 2 – Humidity control 3 – Only temperature 4 – Calibration 5 – Only humidity	Control operation state. Has requirements with the lid (<i>tampa</i>)
	Visor	0 – Off 1 – Operating 2 – Menu	Controls display operation mode
	SDticks	0	Delays SD card writing
	Uticks	0	Delays humidity control response
char	intSel	0	Operates the menu selector
bool	MAN	True/ False	Checks whether the system has come from a maintenance state to reset
	vSD	True/ False	Checks whether the SD card is inserted

5.2. Intervals

Type	Name	Value	Description
unsigned long	SDLT	0	Time of last cycle
	TECon	0	Time when TEC was turned on
	ULT	0	Time when humidity monitoring started
	RPMLT	0	Time the fan showed RPM below the limit
	varLT	0	Time the average variation was recorded
	intLT	0	Time of last interface action
	termLT	0	Time the thermostat was last changed
	buzzerLT	0	Time the buzzer alert was last activated
	tempResLT	0	Time of the last thermostat reset due to high temperature
const unsigned long	SDInterval	20000	Operation cycle interval (milliseconds)
	varInterval	900000	Recording of the average variation (15 minutes)
	intInterval	5500	Timeout for menu action and return to the standard screen
	termInterval	180000	Interval for thermostat functions
	buzzerInterval	600000	Interval to repeat the buzzer alerts

5.3. Moving Averages

Type	Name	Value	Description
unsigned char	pTin	0	Circular buffer position index of internal temperature
	pUin	0	Circular buffer position index of internal humidity
	pTex	0	Circular buffer position index of external temperature
	vTin	0	Filling of the internal temperature buffer
	vUin	0	Filling of the internal humidity buffer
	vTex	0	Filling of the external temperature buffer
int	mTin[6]	0	Circular buffer for internal temperature average
	mUin[6]	0	Circular buffer for internal humidity average
	mTex[6]	0	Circular buffer for external temperature average

5.4. Operation parameters

Type	Name	Value	Description
const int	Tsup		Upper limit of temperature range
	Talvo		Target temperature
	Tinf		Lower limit of temperature range
	Usup		Upper limite of humidity range
	Ualvo		Target relative humidity
	Uinf		Lower limit of humidity range
int	Tb	Talvo	Tolerance for TEC restart when returning from a shutdown below <i>Tinf</i>
	dUmax	10	Initial maximum humidity variation
	DTc	0	ΔT compensation on thermal control
const unsigned char	sClose	52	Humidity servo maximum closed angle
	sOpen	0	Humidity servo maximum opened angle

5.5. Monitoring

Type	Name	Value	Description
bool	intW	True/False	Keep display on while interacting with the menu
	OPT	True/False	TEC status monitor (on or off)
	tampa	True/False	If the lid is attached
	Useco	True/False	Registers if an excessive dry event happened
int	rpm	5000	RPM reading for the external fan, starts high to bypass the initial verification
	US[3]		Humidity status 0 – Variation (1: rising -1: falling) 1 – Trapdoor servo position 2 – Servo angle variation
	varM		Variation multiplier for the 15-minute status
	varV	9000	15-Minute indicator - temperature
	varU	9000	15-Minute indicator - humidity
	conT[3]		Monitoring of temperature values 0 – Previous temperature, for calculating variation 1 – Moving average of 5 readings 2 – Temperature 15 minutes ago
	conU[4]		Monitoring of humidity values 0 – Previous humidity, for calculating variation 1 – Moving average of 6 readings 2 – Module of the last variation 3 – Humidity 15 minutes ago
	conE		Monitoring of the moving average of external temperature
char	TS[2]		Temperature status 0 – variation (1: rising -1: falling) 1 – Occurrences below <i>Tinf</i>
	MoSt	1	<i>Monitor</i> status 0 – Normal 1 – Maintenance return 2 – Low cooling power 3 – Low drying capacity 4 – High cooling power 5 – Fan failure 6 – Temp. above <i>Tsup</i> 7 – DHT failure
unsigned char	sGaus		Servo thermostat angle
volatile unsigned long	counter	0	Counter for reading the RPM of the external fan

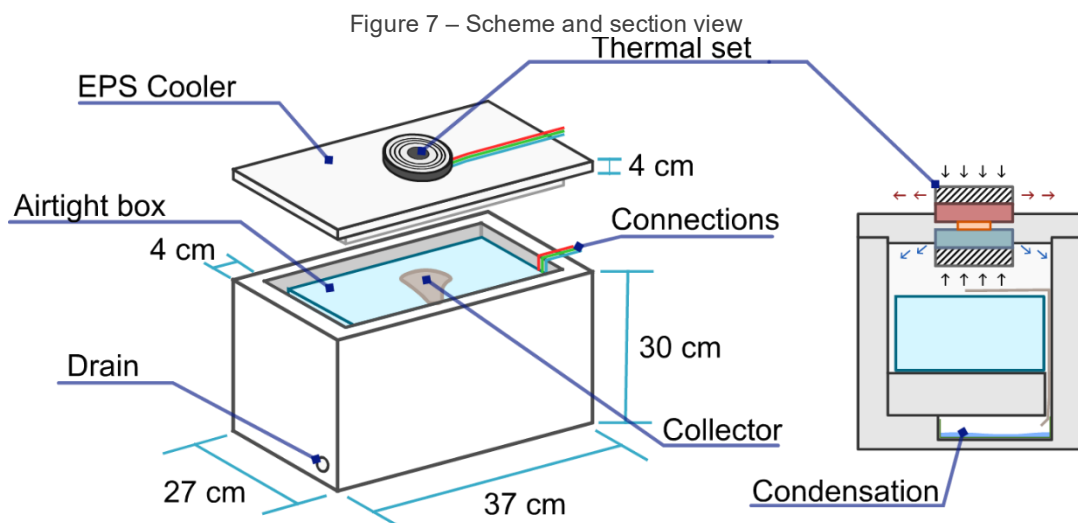
6. SCHEMATICS

6.1. Structure

The chamber's hygroscopic insulation is achieved by using an airtight polypropylene container. The wiring passes through a hole in the side of the box sealed with silicone. For the dimensions of this project, a 4-liter container with locks on the lid was used. The thermal insulation comes from the EPS cooler box (12 liters). The walls were originally 2 cm thick and were reinforced by another 2 cm by gluing new plates to improve the insulation.

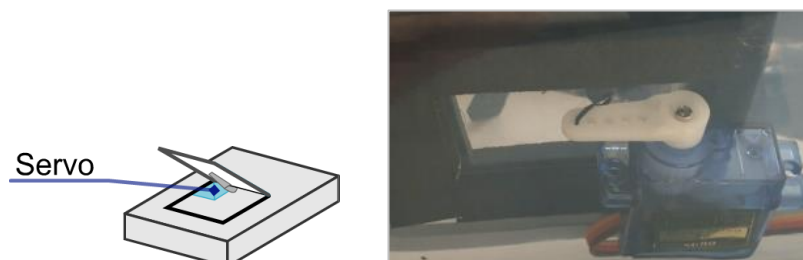
The thermal set is fixed in the center of the lid. A computer fan/heatsink is fixed to each side of the Peltier element. Thermal paste is used on all interfaces. It is possible for the internal temperature to reach the dew point on the cold heatsink, causing moisture condensation inside the chamber. Therefore, care must be taken with the arrangement of the internal heatsink to prevent water runoff from reaching the electronic parts of the fan and causing damage. In addition, dripping on the hermetic container is annoying and can interfere with operation. To prevent water accumulation, a fabric is used as a collector, directed along the side of the cooler to a reservoir at the bottom, where the water will be deposited by capillarity. A small hole in the front of the chamber acts as a drain for convenience, and it is advisable to cover it, since the volume of condensation water is usually small for short time periods.

The DHT sensors are positioned: 1) inside the silica gel container 2) inside the airtight container 3) on the outside of the chamber.

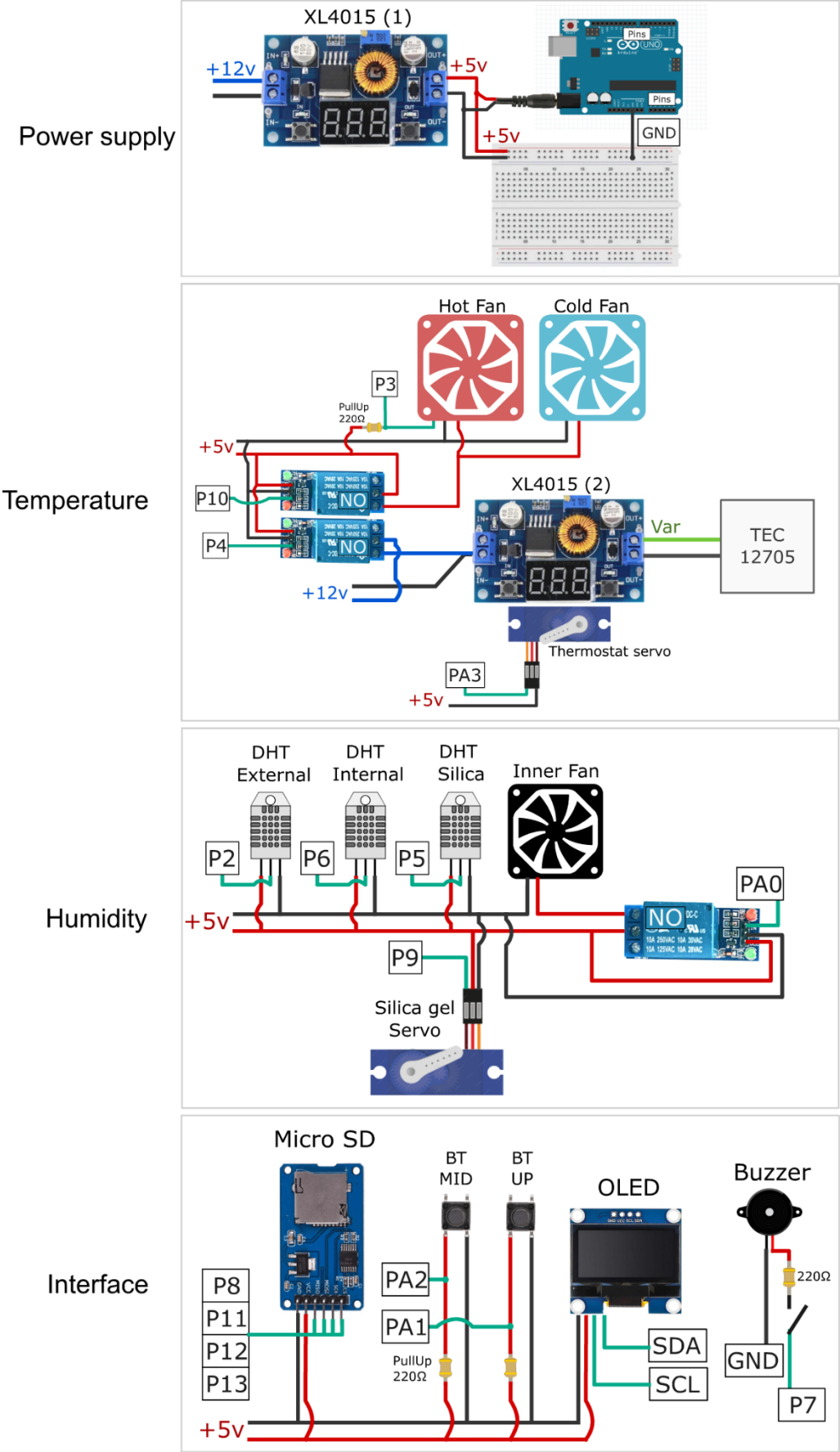


The desiccant trapdoor was also made using an airtight container, with a cut in the lid and positioning of the servo on the inside, attaching its arm to the lid so that in addition to pushing it open, it can pull it to close.

Figure 8 – Desiccant trapdoor scheme

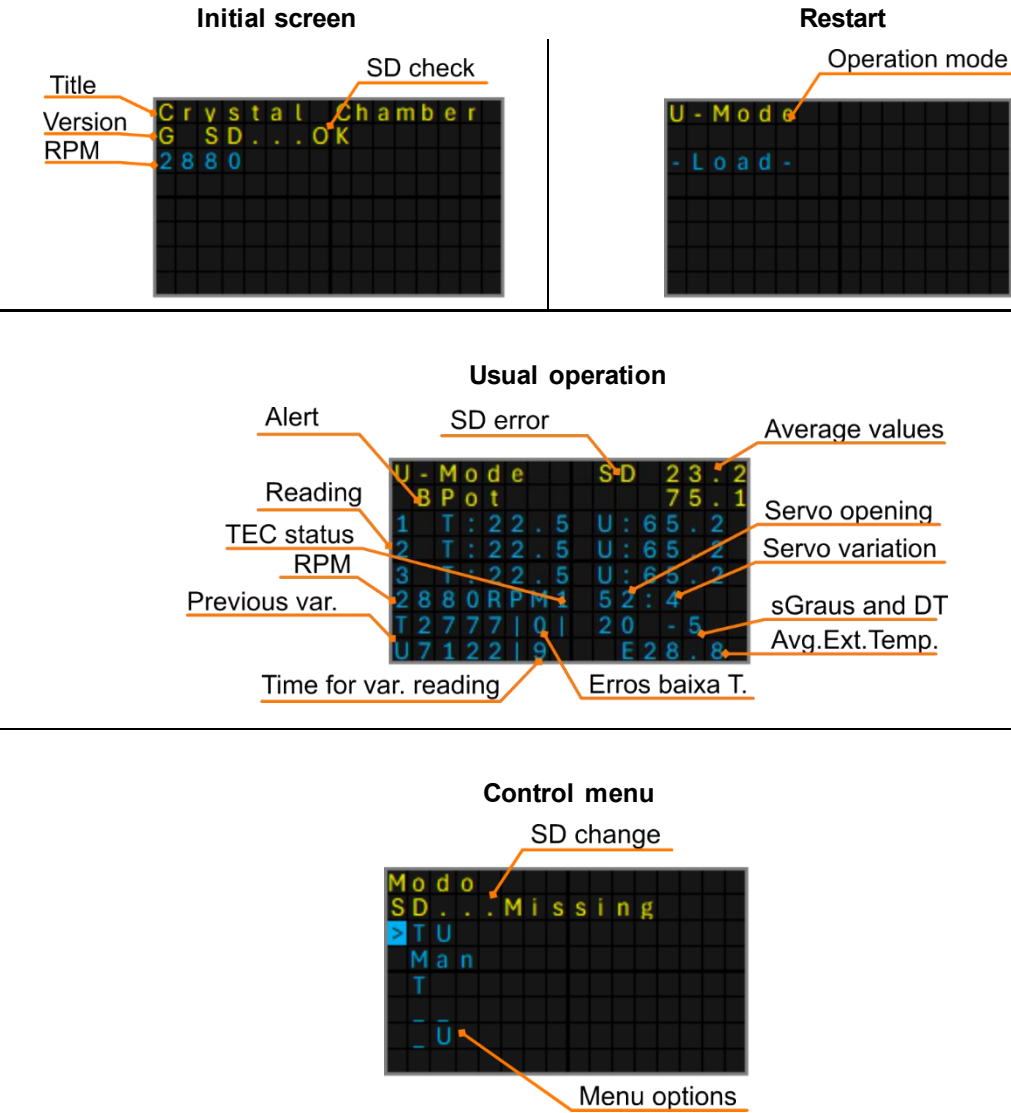


6.2. Connections

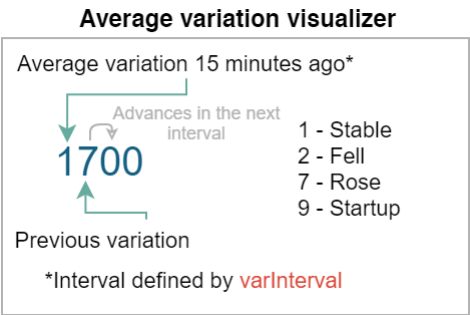


6.3. Display

The current sketch uses a 128 x 64 pixel display and the U8x8 library, where each character has 8 pixels resulting in a 16 x 8 grid to information arrangement with the following variations:



The average variation is periodically verified and displayed in a coded form, allowing for the monitoring of recent history:



6.4. Images

Figure 9 – External overview



Figure 10 – Side view

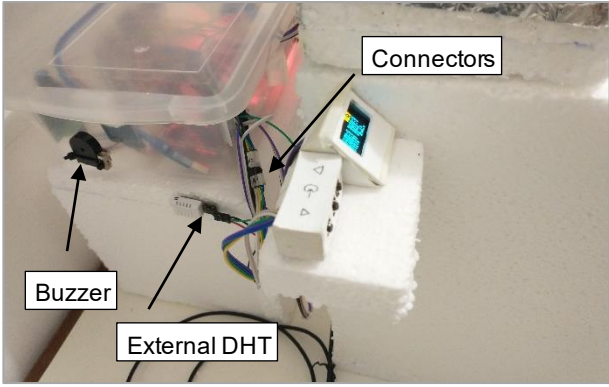


Figure 11 – Top view

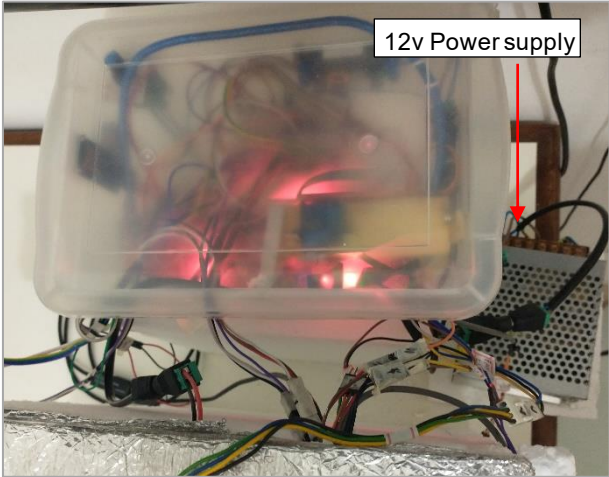


Figure 12 – Electronics box

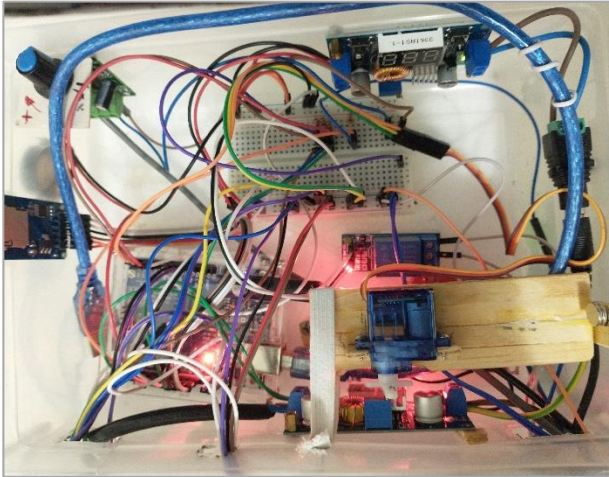


Figure 13 – Internal view

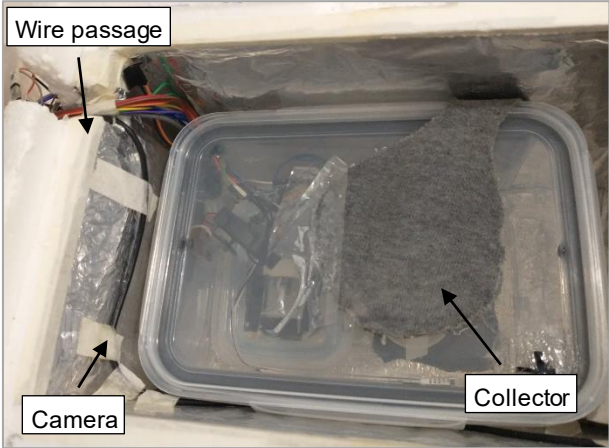


Figure 14 – Inside, silica gel and fan

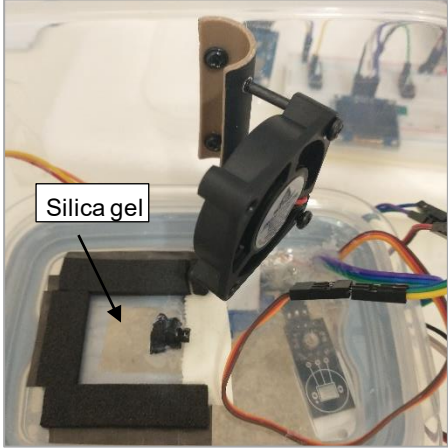


Figure 15 – Exposure environment

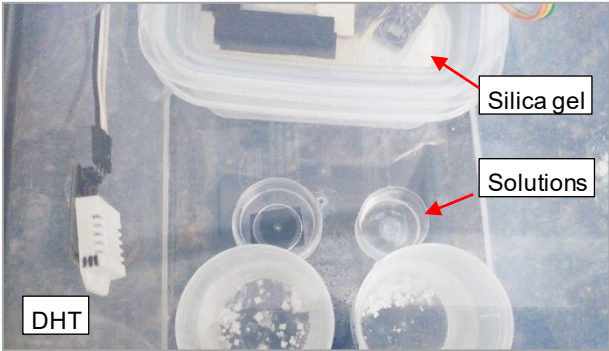


Figure 16 – Reservoir

