Control electronics for chemical sensors

A Degree Thesis
Submitted to the Faculty of the
Escola Tècnica d'Enginyeria de Telecomunicació de Barcelona
Universitat Politècnica de Catalunya
by
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In partial fulfilment
of the requirements for the degree in
TECHNOLOGIES AND SERVICES OF TELECOMUNICATIONS ENGINEERING

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Barcelona, January 2020

Abstract

The objective of this thesis is to develop an electronic implementation of a surface potential control for gas metal oxides (MOX) sensors. To go after the objective of this project, a PCB has been designed and fabricated which allows to apply different controls. To test the board and investigate the best way to control the sensor, different controls have been developed and applied in a controlled environment.

The document covers the description of the state of the art, the setup, the designed PCB, its controls, its main features, and the results obtained.

With this study, it has been proven that such electronic system can be implemented and good results have been obtained with a variety of controls. It has accomplished faster response times and a cheaper cost than the rest of the gas sensing systems in the market.

Resum

L’objectiu d’aquesta tesi és desenvolupar una implementació electrònica d’un control de potencial de superfície per a un sensor MOX. Per tal d’aconseguir aquest objectiu, una PCB ha sigut dissenyada i fabricada, que permet aplicar diferents controls sobre els sensors. Per testejar aquesta placa i investigar la millor manera de controlar el sensor, diferents controls han sigut desenvolupats i aplicats en un entorn controlat.

El document cobreix la descripció del “state of the art”, el setup que s’ha fet servir, la PCB dissenyada, els controls aplicats, les seves diferents característiques i els resultats obtinguts.

Amb aquest estudi, s’ha comprovat que el sistema electrònic anomenat anteriorment pot ser implementat, i s’han obtingut bons resultats amb una varietat de controls. S’ha aconseguit temps de resposta més ràpids i un cost més barat que la resta dels sistemes de mesura de gas del mercat.

Resumen

El objetivo de esta tesis es desarrollar una implementación electrónica de un control de potencial de superficie para un sensor MOX. Para conseguir este objetivo, una PCB ha sido diseñada y fabricada, que permite aplicar distintos controles sobre los sensores. Para testear esta placa, e investigar la mejor manera de controlar el sensor, diferentes controles han sido desarrollados y aplicados en un entorno controlado.

El documento cubre la descripción del "state of the art", el setup utilizado, la PCB diseñada, los controles aplicados, sus diferentes características y los resultados obtenidos.

Con este estudio, se ha comprobado que dicho sistema electrónico puede ser implementado, y se han obtenido buenos resultados con una variedad de controles. Se han conseguido tiempos de respuesta más rápidos y un coste más barato que el resto de sistemas de medida de gas del mercado.

This thesis is dedicated to my family and friends, who have celebrated with me every achievement during the degree and supported me during hard times. Specially I want to dedicate this degree thesis to my grandfather, who nowadays would be proud of my achievements, and this idea kept me studying and working hard during this degree.

Acknowledgements

I would like to express my deep and sincere gratitude to all the people who guided and helped me along the duration of this project. Specifically, I thank my research supervisor Professor Manuel M. Domínguez Pumar for introducing me to research, for teaching me many important concepts and for trusting me with this project. I also want to thank Professor Juan José Ramos Castro for the help and knowledge provided during the circuit design, and to the professor Vicente Jiménez Serres for the help provided with the digital side of the developed system.

Revision history and approval record

Revision	**Date**	**Purpose**
0	15/10/2020	Document creation
1	10/01/2021	Document revision

DOCUMENT DISTRIBUTION LIST

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1. Introduction

The measurement of gas concentrations is a very common practice in various environments. In industry it is used to double-check the quality of a certain product, or to check the compliance with environmental regulations, it is also widely used as a safety set-up where if there is any hazardous gas leakage it prevents people to stay in contact with it for too long. In the farming industry is used to maintain a healthy environment for the animals and therefore to increase production.

The concentration of a certain gas in the environment can be measured in different ways, the different gas sensors are classified into various types, based on the type of sensing element. The most used are:

- **Metal Oxide-based gas sensors:** as the name states, a combination of MEMS technology and MOX technology creates highly sensitive and responsive semiconductor sensors. This sensor technology is used in order to create the control developed through this thesis.
- **Optical gas sensors:** Those sensors, use the properties of absorption, fluorescence, scattering, deflection of light, and other phenomena for the detection of gas components.
- **Electrochemical gas sensors:** Those measure the concertation of a gas when it oxidizes or reduces the target gas at an electrode, by measuring the resulting current.
- **Capacitance-based gas sensors**: This technology is based on capacitive coupling, where it can measure a gas concentration using this gas as a dielectric.

1.1. Objective, requirements and specifications

The main objective of this project is to proof that an electronic implemented version of the electronic control designed and tested in “Open loop testing for optimizing the closed loop operation of chemical systems” [1] can be done. That can be applied to a variety of sensors, commercial ones as the CCS801 which we have been working with, this sensor has been used in the study named before, so we know that they are reliable, it can also be applied to specifically created sensors, developed in the (URV) Rovira and Virgili University in Tarragona by Eduard Llobet, Carlos Belmonte and Eric Navarrete.

As it is a continuation of the project developed by M. Dominguez-Pumar, J.M. Olm, L. Kowalski, and V. Jimenez, the setup and the technology used will be the same or very similar, and it will be described later in this thesis.

In order to accomplish the main objective, some sub-objectives must be accomplished:

- A precise control of the sensor’s temperature. In order to develop this control, a closed loop Wheatstone bridge is used, it is explained in 2.3 state of the art of temperature control.
- A closed loop control with an external digital board has been used in order to apply different types of control to the chemical sensor via the temperature, explained in the 2.2 state of art of the surface potential control.

This project is required to read chemical sensor's values in order to determine how much concentration of a certain gas is in the sensed atmosphere, it also needs to have a short time response, shorter than the other sensors due to our state of the art surface potential control and it has to be a relatively cheap system, cheaper than other gas sensing systems, which is accomplished by our colleagues in URV by developing cheap MOX sensors.

The circuit works from 0 to 5 Volts, and it has an adaptation circuit for communications with an external digital board, which works at digital levels of 0 to 3.3 Volts. To accomplish faster response times, temperature control is used. So the key value is not the resistance of the chemical sensor, indeed it is the required average temperature needed to maintain that resistance stable.

The designed system can be interesting for many companies or individuals, as it is a state-of-the-art device, it could be a very competitive product in the market.

1.2. Work Plan and personal contribution

In this chapter the work plan followed during the development of this project is explained, first, it describes the different work packages with its internal tasks, it is followed by the milestones reached and finally, a Gantt diagram is shown.

1.2.1. Work packages

Project: Control electronics for chemical sensors	WP ref: WP1	
Major constituent: Study	Sheet 1 of 5	
Short description: Study the approach of the project. Study how it has been done until the moment, know the state of the art, and understand it. Also learn the different techniques that will be used.	Planned start date: 15/07/20 Planned end date: 1/09/20	
	Start event: 15/07/20 End event: 1/09/20	
Internal task T1: Study the state of the art, through reading multiple papers related to the project. Internal task T2: Learn to apply the different techniques that will be used during the project. (PCB design, schematic construction…)	Deliverables: None	Dates

Project: Control electronics for chemical sensors	WP ref: WP2	
Major constituent: Design the test circuit	Sheet 2 of 5	
Short description: To develop a first schematic and design the PCB for a first circuit, in order to be able to test different sensors.	Planned start date:1/09/20 Planned end date:2/10/20	
	Start event:1/09/20 End event:2/10/20	
Internal task T1: Design a schematic for the circuit Internal task T2: Design a PCB Internal task T3: Print the PCB	Deliverables: Schematic PCB PCB printing	Dates: 15/09/20 28/09/20 2/10/20

Project: Control electronics for chemical sensors	WP ref: WP3	
Major constituent: Test the designed circuit	Sheet 3 of 5	
Short description: Use the PCB test circuit and find it's weak spots, also study which parts are unnecessary and which parts needs improving. Also test the difference between the different sensors.	Planned start date: 2/10/20 Planned end date: 19/10/20	
	Start event: 2/10/20 End event:13/11/20	
Internal task T1: Test the circuit thoroughly. Internal task T2: To characterize the CCS801 sensor. Internal task T3: Design a basic digital control. Adjust the circuit to the sensor and test it.	Deliverables: The system working properly.	Dates: 13/11/20

Project: Control electronics for chemical sensors	WP ref: WP4	
Major constituent: Control iterations	Sheet 4 of 5	
Short description: Design and test as many variations of the control as needed in order to end up with the best system possible.	Planned start date:19/10/20 Planned end date:15/01/21	
	Start event: 13/11/20 End event: 24/01/21	
Internal task T1: Test the different controls of the previous studies. Internal task T2: Create new controls and test them	Deliverables: Lessons learned from each control.	Dates: 24/1/21

1.2.2. Milestones

WP#	Task#	Short title	Milestone / deliverable	Date (week)
WP1	T1	Study the state of the art	-	Week 1
	T2	Learn PCB design techniques	-	Week 2, 3
WP2	T1	Design the schematic	Schematic	Week 4,5
	T2	Design the PCB	PCB design	Week 6,7,8
	T3	Print the PCB	PCB board	Week 8
WP3	T1	Test the circuit	The system working properly.	Week 9
	T2	Characterize the CCS801		Week 10-11
	T3	Adjust the circuit and test it.		Week 12-13
WP4	T1	Test the different controls of the previous studies.	Lessons learned from each control.	Week 14-21
	T2	New controls.		Week 14-21

1.2.3. Gantt diagram

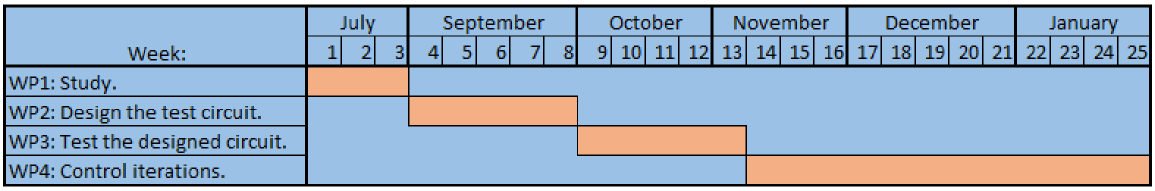


Figure 1: Gantt diagram

1.3. Deviations from the initial plan

Having to wait for months to receive the ammonia sensors from Tarragona, I had to go ahead with the commercial ammonia sensors, the CCS801 to be specific.

The time spent testing the circuit ended up being double what was expected, but it had no major repercussions.

A final version of the product was the last objective of the project, unfortunately as more research needs to be done in the control, a final design could not be done.

1.4. Structure of the document

Following the ideas raised above, this thesis collects the work done to create and develop the project. The information is structured as follows:

- Chapter 2: A literary review of the different techniques and technologies used during the development of this project.
- Chapter 3: This chapter includes the description of the different elements involved in the construction of the setup and the instruments utilized in order to take the different measurements. Also includes the different controls developed and applied.
- Chapter 4: Includes a recompilation of the different experiments done in order to test the system developed and the different controls applied, as well as an analysis of each one.
- Chapter 5: It contains a budget for the prototype designed and built.
- Chapter 6: Presents the conclusions of the project based on the results obtained.

2. State of the art of the technology used or applied:

In order to have knowledge about all the state of the art of the technology of the thesis, we will divide it into two parts. The state of the art of MOX gas sensors, the state of art of surface potential control, and the state of the art of the temperature control.

2.1. State of the art of MOX gas sensors

Chemoresistive gas sensors based on semiconducting gas metal oxides are widely used in many industries. The principle of their operation is rather simple as it is explained in "Fundamentals of Metal Oxide Gas Sensors" [2]:

"*In air, at temperatures between 150ºC and 400ºC, oxygen is absorbed at the surface of the metal oxides by trapping electrons from the bulk with the overall effect of increasing the resistance of the sensor, for n-type materials, or decreasing it, for p-type materials. The occurrence of a target gas in the atmosphere, which reacts with the preabsorbed oxygen or directly with the oxide, determines a change of the sensor resistance, which is recorded as a sensor signal and the magnitude of which is correlated to the concentration of the target gas*."

As it can be seen in the figure 2 these sensors are usually formed by two electrodes connected to the metal oxide in order to measure its electrical resistance, and a heated plate which use is to bring the sensing layer temperature up to its working range.

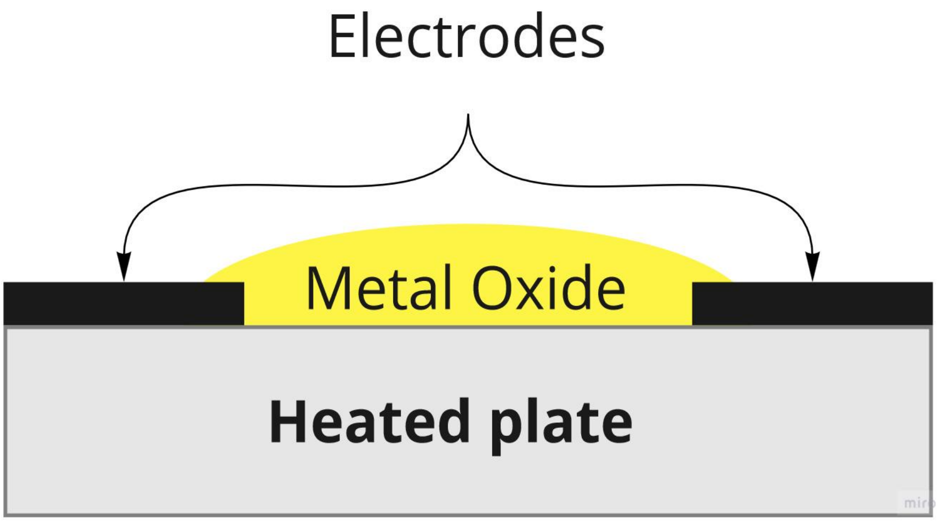


Figure 2: MOX sensor schematic

2.2. State of the art of surface potential control

As it has been stated before, the project developed during this final degree thesis is a continuation of the research done by M. Dominguez-Pumar, J.M. Olm, L. Kowalski and V. Jimenez, embodied in the published paper of Computers and Chemical Engineering “Open loop testing for optimizing the closed loop operation of chemical systems” [1].

This paper explains the methods required to obtain the bests results out of a Metal Oxide (MOX) gas sensor, by using a Surface Potential (SP) control. This set-up accomplishes low cost systems, with high surface-to-volume and therefore higher sensitivities, good stability, low power consumption, compatibility with semiconductor fabrication processes, and faster response time.

The paper explains the controls used. The system described is based in maintaining stable the electrical resistance of a MOX sensor’s sensing layer, by controlling its temperature. As a result of this control, the measuring information is no longer the sensing layer resistance, but its mean temperature.

In that study the controls applied are the sigma delta of first order and the sigma delta of second order, they can be seen in figure 3 and figure 4.

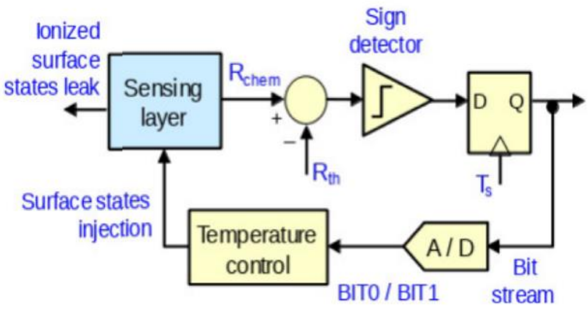


Figure 3: First order sigma delta scheme

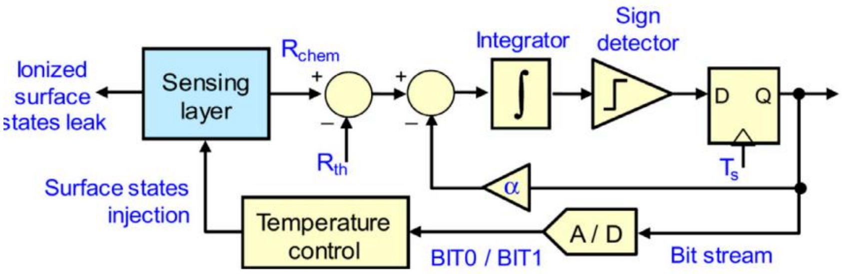


Figure 4: Second order sigma delta scheme

The experiments that took place in the named research, proved that such control can be applied and faster response time can be accomplished in a Metal Oxide sensor.

2.3. State of art of the temperature control

In order to control precisely the sensor temperatures, it has been used the method developed in "an investigation of the constant-temperature hot-wire anemometer" [3]. In this study, faster time response of the constant-temperature hot-wire anemometer was accomplished, by using a Wheatstone bridge with a feedback amplifier with both, high gain and high frequency response. The model circuit is the one represented in figure 5.

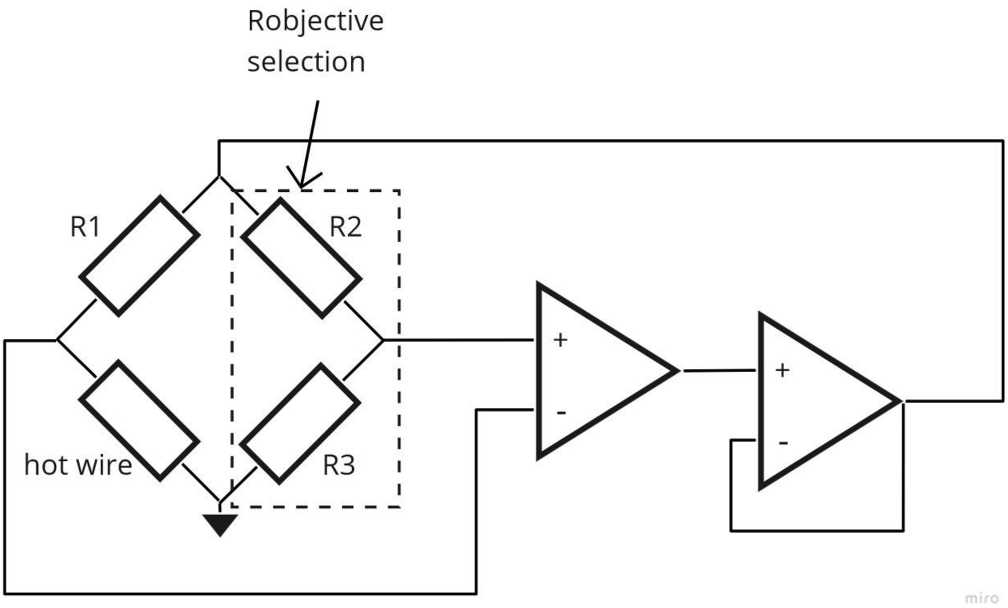


Figure 5: Closed loop Wheatstone bridge

The use of the Wheatstone bridge allows the user to choose the objective resistance of the hot-wire, and therefore its temperature. The feedback amplifier, corrects all the deviations of the hot-wire by applying more or less power to it.

3. Methodology / project development:

In this chapter will be included everything that has been used in order to develop the project, the sensors used, the gas control system, the control circuits designed, the measurements setup, the sampling and control protocols, and the characterization and calibration of the sensors.

3.1. Sensors used

3.1.1. CCS801

The sensor used mostly in this project is the CCS801 (figure 6), and as it is described in its datasheet it is an "*Ultra-Low Power Analog VOC Sensor for indoor air Quality Monitoring".* It contains a micro-hotplate technology, that is used to set the temperature of the sensing layer to the desired temperature, up to 500ºC. It can monitor Carbon Monoxide (CO) and a wide range of Volatile Organic Compounds (VOCs) such as Ethanol, which is the target gas used to develop this project.

Figure 6: CCS801 sensor

This sensor has been chosen as it was also used in [1], so a knowledge base of this sensor is already known. It was chosen due to its micro-hot plate, so a control of the temperature of the sensor can be designed and implemented. Also, this sensor has given good results for its ability to change its temperature in 5-10ms, which is very fast compared to other micro-hot plates.

3.1.2. WO3 nanoneedles

Those sensors were created in URV and they had tungsten trioxide (WO3) nanoneedles grown on top of two different hot plates, the "KMHP-100 MEMS Micro-Hotplate" which has a heating layer and two pads to sense the grown material, and the "CNR-IMM ULP-MOX hotplate" which is similar to the KMHP-100 but instead of having one sensing layer and one heating element, it has 4 of each that can be controlled separately. Those micro hotplates can be seen in figure 7 and 8. In figure 9, can be seen how these micro-hot plates are connected to a thin board with pads for testing purposes, and the growth of the sensing layer is done on top of this. At the end of the project, these sensors have not been tested yet.

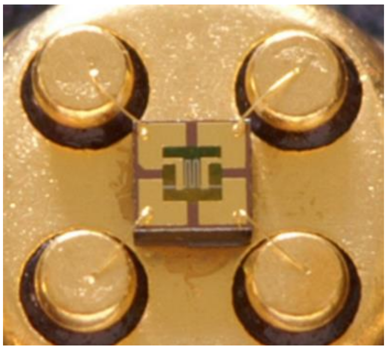


Figure 7: KMHP-100 MEMS Micro-Hotplate

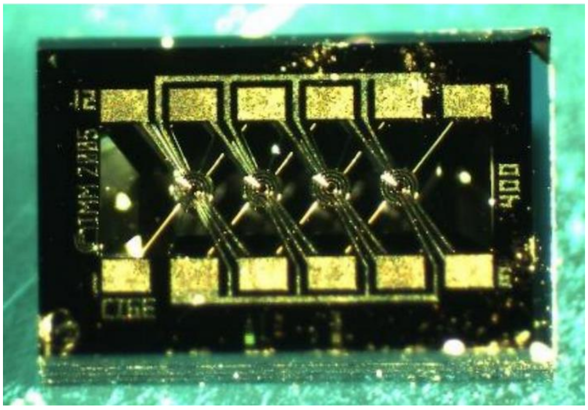


Figure 8: CNR-IMM ULP-MOX hotplate

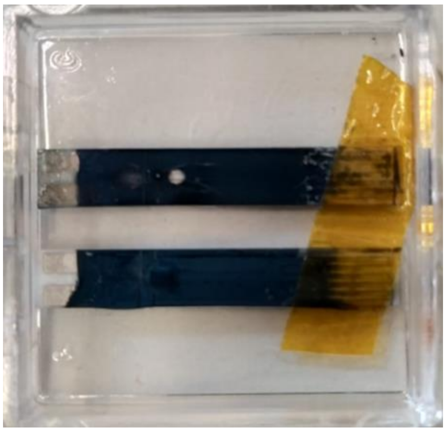


Figure 9: Both sensors mounted on a board with the WO3 layer

3.2. Gas control

In order to have a controlled atmosphere, we will reuse a set-up created by Lukasz Kowalski, which with a PC using LabView software and a NI PXIe-1073 with a PXI-7853R, (see figure 10) we can control different mass flow meters that control the flow of the desired gases, coming from gas bottles into a chamber designed to hold the sensor. In order to have a total control of the gas or combination of gases to which the sensor is exposed, and its concentrations. The set-up can be seen in figure 11.

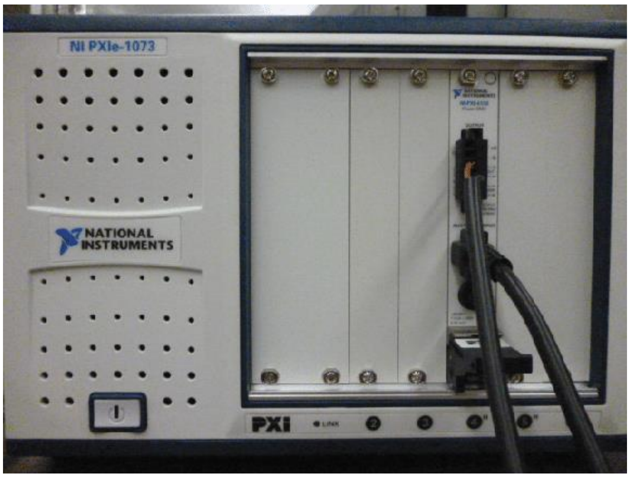


Figure 10: NI PXIe-1073 with a PXI-7853R

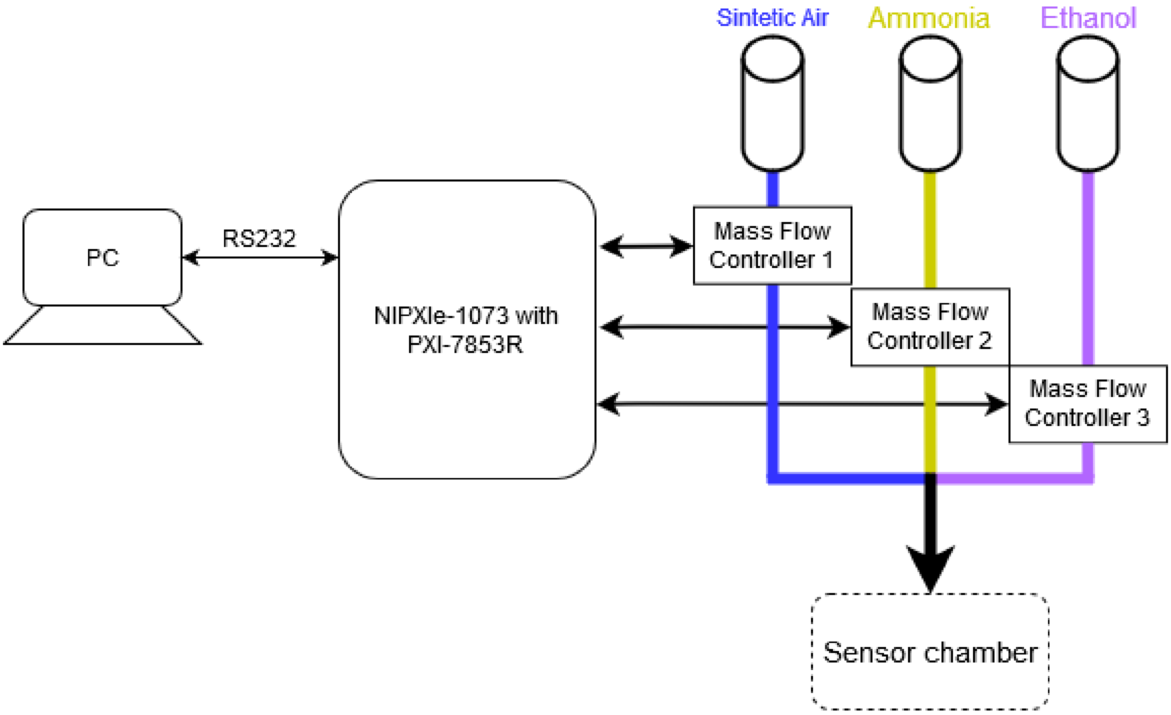


Figure 11: Gas control schematic

3.3. Control circuits

In order to develop the control and to record the measures of the sensor, a PCB was created, and it contains two sub circuits. The first one is designed for the control of the heater, and the second one is designed for the chemical sensor.

3.3.1. Temperature control circuit

As explained in the "State of art 2.2" the control circuit for the temperature is based on a two-wire anemometer control with a closed loop Wheatstone bridge. But in order to be able to choose between the two different temperatures necessary for the control a MUX (a SN74LV4052AD) is built in, so the circuit can alternate between two different resistance branches or the output of a DAC (the DAC8830). The circuit can be seen in the figure 12.

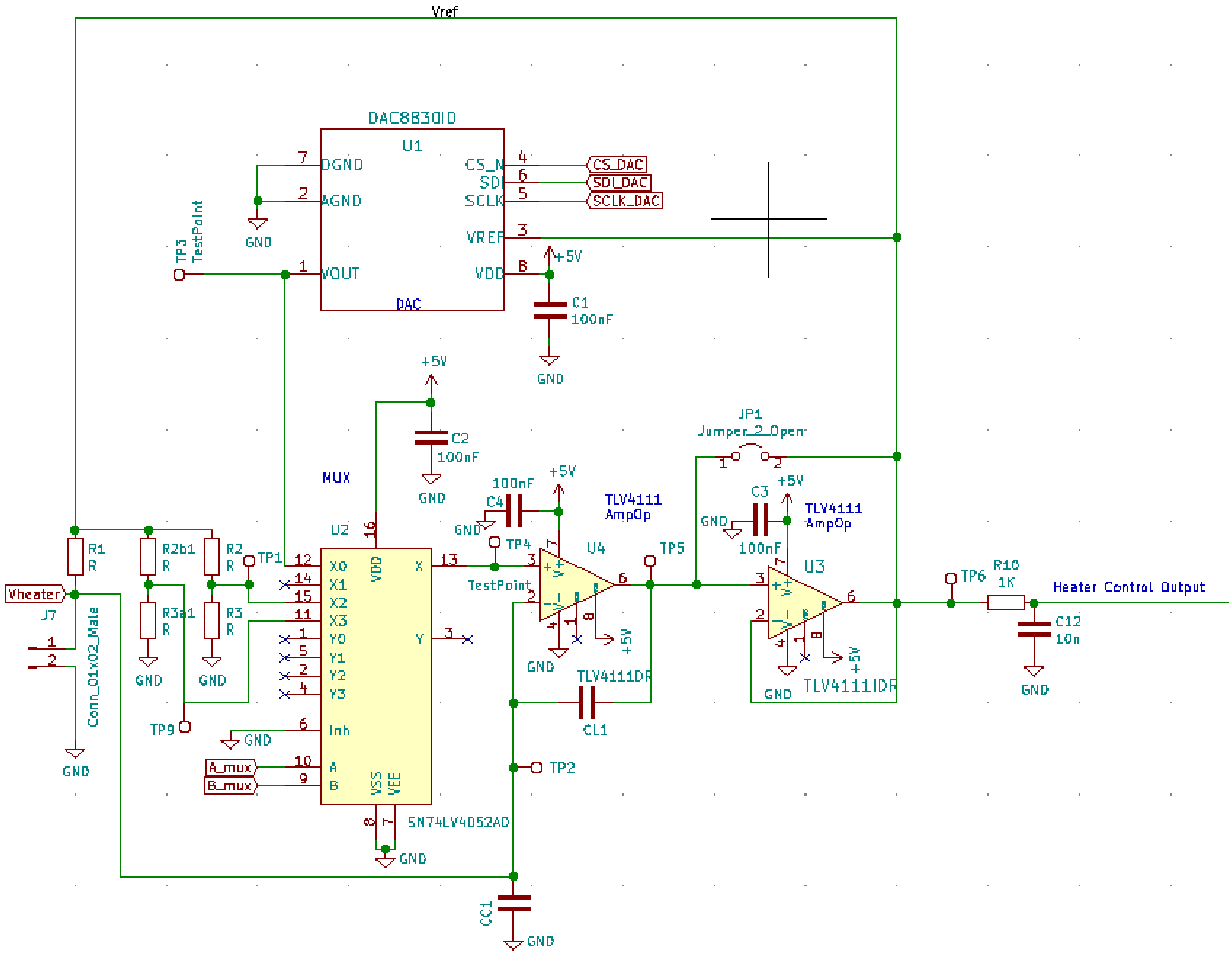


Figure 12: Temperature control circuit

The main feature of the designed circuit is that instead of comparing the output of two voltage dividers connected to the circuit's output, it can compare the ratio Vout/Vin of the DAC (which can be changed digitally) with the Vout/Vin of the voltage divider formed by the Rheater. So the control (digital board) has a wide range of values to set the resistor of the heater and therefore, a wide range of Temperature values that can be chosen using the digital SPI signal.

After comparing the two signals (the one from the MUX and the one from the Rheater voltage divider), it passes through a voltage follower and then, this signal named Vref, outputs to the ADC, this signal is also the one that flows back to the circuit, closing the loop of temperature control. It also has another ADC, which can monitor the voltage drop at the heater resistance, whit those two measures (Vref and Vheater) the Heater resistance can be calculated easily.

3.3.2. Chemical resistance acquisition circuit

In order to apply the different controls, the signal from the Rchem has to be compared with an objective resistance, it will be done using a INA188IDR operational amplifier. Also, it will be applying an offset of 2.5V, which is achieved using a voltage divider and an OpAmp voltage follower. The resulting signal will be outputted to the ADC in order to be received by the digital part of the circuit and take the control decisions from it.

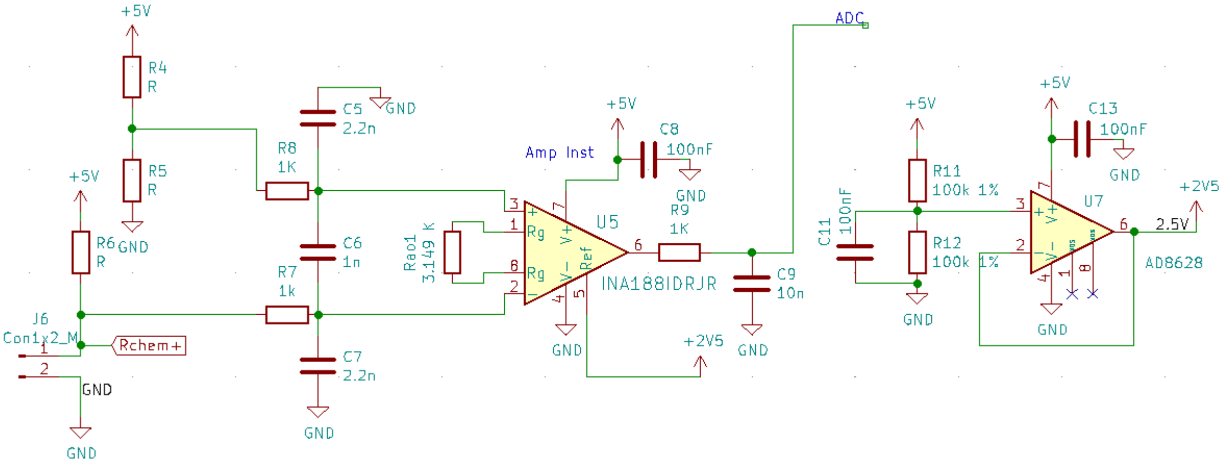


Figure 13: Chemical resistance acquisition circuit

The system has another ADC connected to Rchem, so it always knows the current resistance of the sensing layer. The output of the circuit can be described by the next equations:

$$V_{out\_Chem} = (V^{+} - V^{-}) \cdot G + 2.5$$

Rao=3.149 kΩ then $G = 1 + \frac{50k}{Rao} = 16.878$

$$V^{+} = 5 \cdot \frac{R_{obj}}{R_{obj} + R_4} \qquad V^{-} = 5 \cdot \frac{R_{Chem}}{R_{Chem} + R_6}$$

$$V_{out\_Chem} = 5 \cdot 16.878 \cdot \left(\frac{R_{obj}}{R_{obj} + R_4} - \frac{R_{Chem}}{R_{Chem} + R_6} \right) + 2.5$$

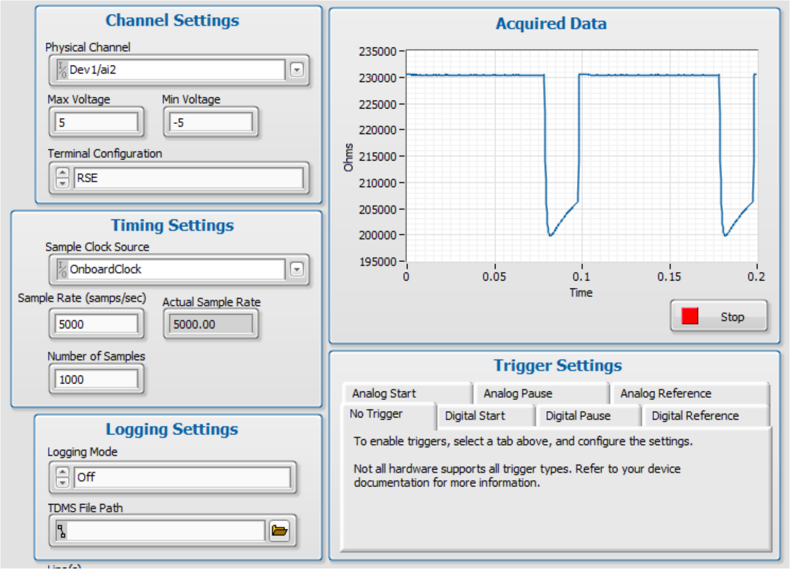
With that equation in mind, if the Robj is higher than the Rchem, the output of the circuit will be superior to 2.5 Volts, and inferior to 2.5V if the Robj is lower than the Rchem.

3.4. Measurements

3.4.1. National Instruments USB-6002

In order to accomplish the firsts measurements in the tests of the sensor and the designed PCB board we used a NI USB6002 (Figure 14), which controlled by a LabView program, was able to collect the data (in figure 15 can be seen an example of that software). It was chosen due to its 16-Bits resolution and its ADC capable of 50kS/s (enough for the firsts measurements).

Figure 14: USB-6002


Channel Settings

Physical Channel: Dev1/ai2
Max Voltage: 5
Min Voltage: -5
Terminal Configuration: RSE

Timing Settings

Sample Clock Source: OnboardClock
Sample Rate (samps/sec): 5000
Actual Sample Rate: 5000.00
Number of Samples: 1000

Logging Settings

Logging Mode: Off
TDMS File Path:

Trigger Settings

To enable triggers, select a tab above, and configure the settings.

Not all hardware supports all trigger types. Refer to your device documentation for more information.

Figure 15: LabView GUI for USB-6002 control

3.4.2. PCB measures and control

In order to establish the measures and the control over the sensor, two ADCs (LTC1865) and a DAC is used.

- **LTC1865**: The LTC1865 is a converter that operates on a single 5V supply and is used to take information from the sensor in order to take decisions over the control. It has been chosen for its 16-bits which give us a great resolution (gap=0.076mV). Also, it is ideal for our setup because it can achieve sampling rates of 1.25 MS/second. It communicates via SPI with the digital board. The ADCs have 2 channels, so we used one of each LTC channel for the chemical circuit (which is necessary for the control of the circuit) and the other two channels for the heater circuit (which is useful in order to monitor the operation of the Heater circuit).
- **DAC8830**: The DAC8830 is the part of the system that is controlled by the digital board, each decision of the control will be communicated to the DAC via SPI. This DAC has been chosen due to its high resolution (16-Bit), and its fast SPI Interface (up to 50MHz).

3.4.3. Digital board

The first board used for a first digital control and readings is the board NUCLEO F030R8 (figure 16). This board allows programing the hardware and the software, it integrates an STM32 microcontroller. Also, it has numerous programmable input and output ports, which has been very useful to establish the SPI communication with the designed PCB board. It is chosen as a first board due to its easy programming, as an example, all the designed functions are programmed with the open source compiler of Mbed in the C++ programming language.

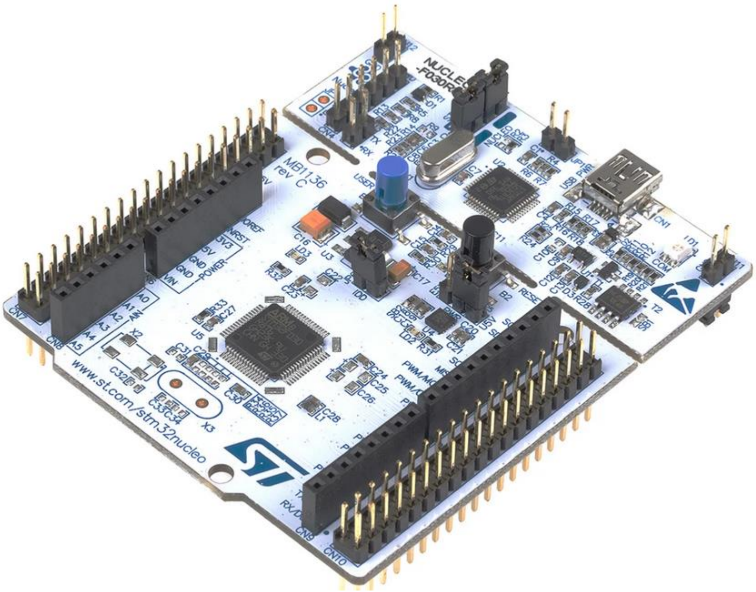


Figure 16: NUCLEO F030R8

This board will use the SPI ports in order to communicate with the PCB, and the USB in order to communicate with the PC. The PC receives the readings of the Heater control circuit and from the chemical circuit, it also receives the control information.

In order to process the information received by the board, a simple serial reader program was developed using MATLAB, which gave us the ability to record the different data sent by the board and to plot it.

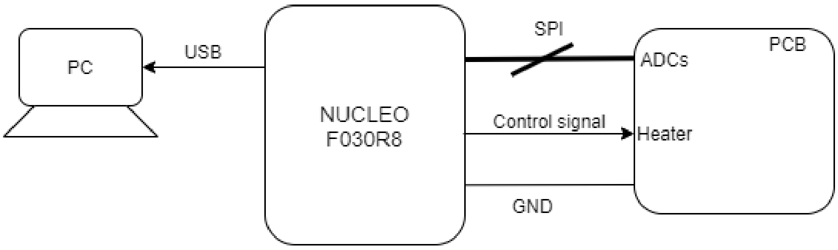


Figure 17: NUCLEO connections diagram

3.5. Setup Architecture

For a better understanding of how everything is assembled, the interconnection of all the previously described parts is represented in a basic schematic, in the figure 18.

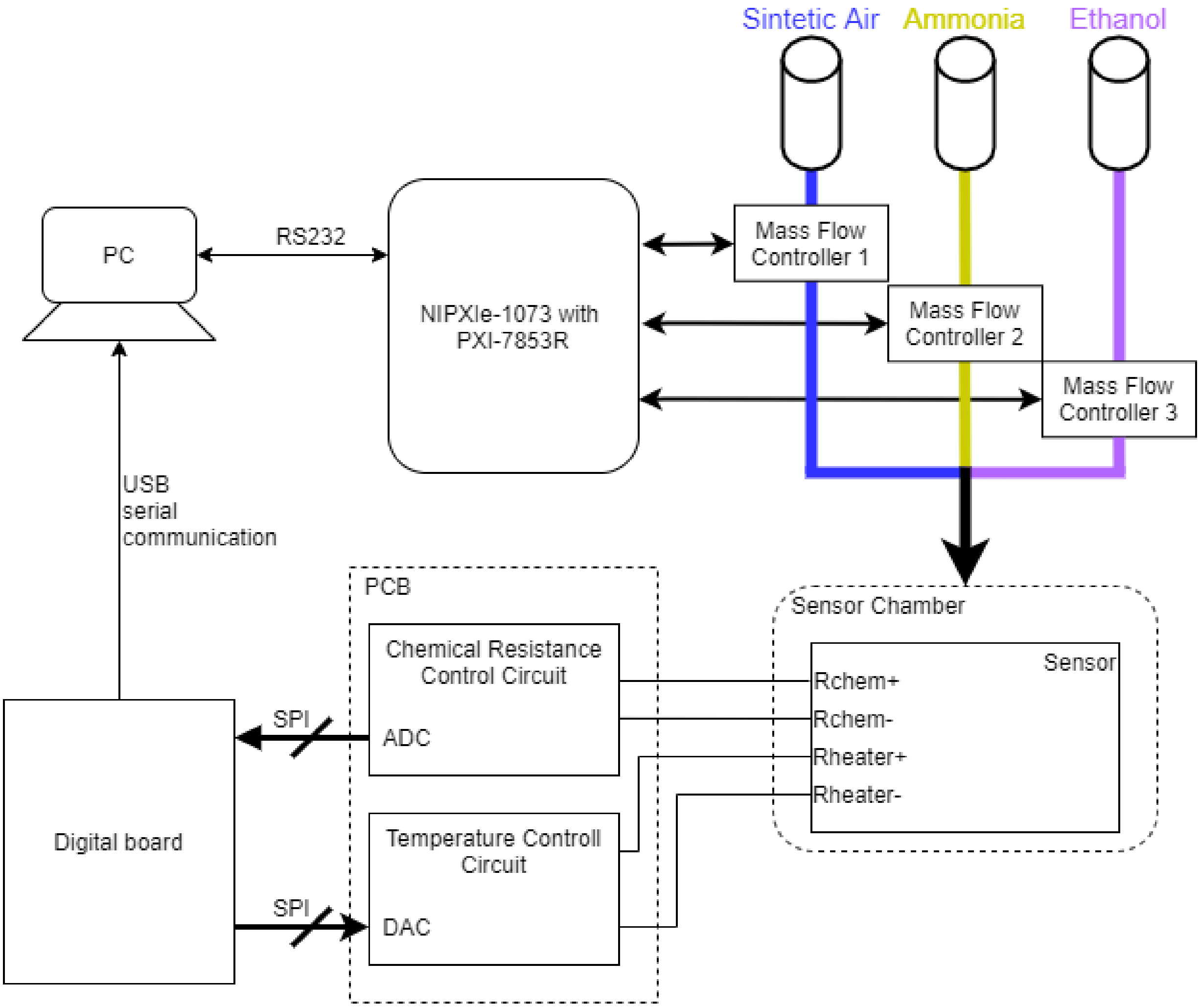


Figure 18: Full setup diagram

3.6. Sampling and control protocol

The sampling and control protocols used were designed with the digital board NUCLEO, using the open source compiler of MBED.

All communication with the PCB is done through SPI, the board has two ADCs to send information to the digital board which is configurated in order to read two new values each millisecond. The digital board sends, also each millisecond, the control decisions to a DAC located in the PCB.

Also, in order to receive the information, the digital board is connected to the PC and it receives via USB serial all the information needed. All the procedures that take place at the digital board, could be summarized in the figure 19 flow chart.

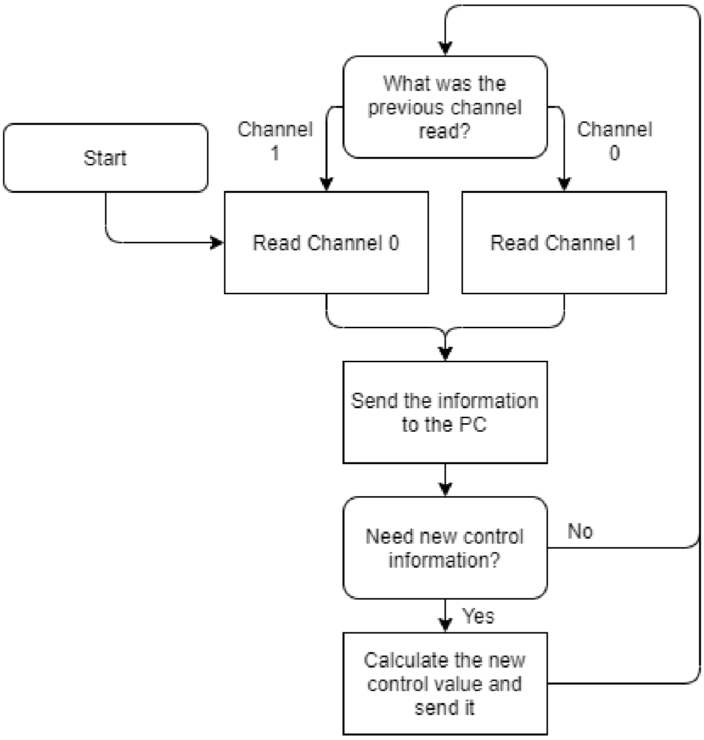


Figure 19: Sampling and control schematic

3.6.1. Sampling Protocol

As stated previously, the sampling of the circuit uses two analog to digital converters placed on the PCB, each one has two channels. The NUCLEO board is programmed in order to receive information from both ADCs each millisecond, and it alternates between the two channels.

3.6.2. Control Protocol

In order to test and to try to improve the circuit, different controls had been developed:

3.6.2.1. First order sigma delta control:

The control of this circuit is a continuation of the [1] controls, so to begin with it will work with a bit0 and a bit1. Bit1 is the system response when control is fixed at high temperature (Thigh, which is in the working range of the sensor) during all the duration of the bit, and the bit0 is when the system response is fixed at low temperature (Tlow, outside the working range of the sensor) during three quarters of the bit and at Thigh during the last quarter, a graphical representation of the different bits can be seen in figure 20. Each bit will have a length of 160ms.

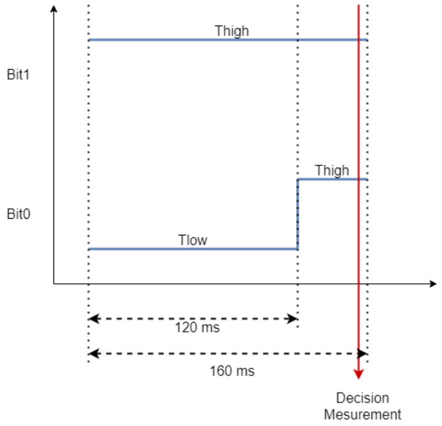


Figure 20: Sigma delta bits

This length per bit has been chosen because as the bit0 needs to be at temperature high at the end in order to make the decision measures always at the same temperature (Thigh), a 40ms heating time is mandatory due to the heater Thigh set-up time (we have been chosen this time because it what took the slower sensor (the one from URV) to change to Thigh). As the sensor is 40ms at high temperature at bit0, at least the 75% of the time needs to be low, so it is three quarters of the time at the low temperature, and in total it means that a bit length of 160ms is needed.

The control of the system is rather simple, at the end of each bit, always at Thigh, it compares the sensing resistance or chemical resistance (Rchem) with the objective resistance (Robj), if the Rchem is higher than the Robj, it takes the decision to make the next bit a bit0, and bit1 if Rchem is lower than the Robj. A diagram of this control can be seen in figure 21.

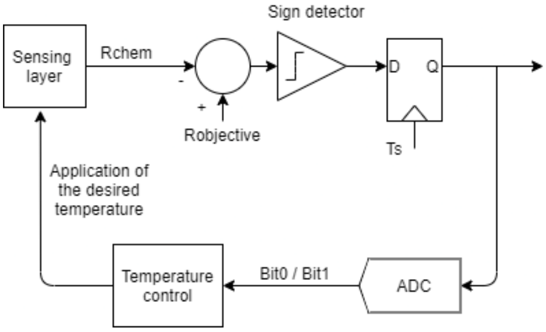


Figure 21: First order sigma delta schema

3.6.2.2. Second order sigma delta control

In order to try to make a less noisy control of the surface potential, while avoiding some undesired effects of the first order control, such as the presence of plateaus, a second order sigma delta control is applied.

It is the same concept of the first order sigma delta, but instead of taking the control decision with the last measurement of the last bit, we include an integrator for all the measurements made at the end of all the bits processed. As a result of this configuration, the decision is taken with the output of that integrator.

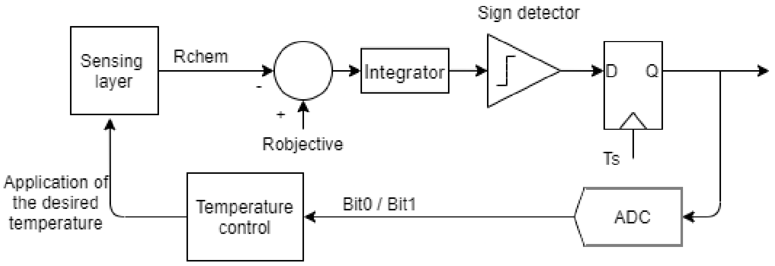


Figure 22: Second order sigma delta schema

3.6.2.3. First order sigma delta control with PWM

This control is similar to the first order sigma delta control, but instead of having a bit0 or bit1, the system takes the decision of increasing or decreasing a PWM value, which determines the time that a bit will be at Thigh or Tlow. With this control, the system has the ability of fine tuning the mean temperature through the PWM signal, therefore a higher resolution is accomplished. Also, the sensing resistance turns out to be more stable around the objective resistance.

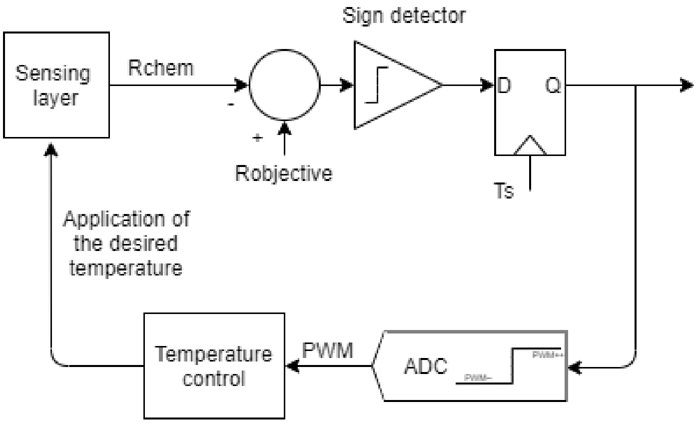


Figure 23: First order sigma delta with PWM

3.6.2.4. First order sigma delta control with PWM and Robjective control

As we continued developing and testing the previous controls, we observed that our different sensors had a temporal drift in the Chemical resistance which could not be controlled, so we always ended up with a saturation of all the controls. In order to prevent that from ruining our measures, an actualization of the first order sigma delta control with PWM was developed, in which the objective resistance would change its value every time that the PWM is at saturation (PWM=100% or PWM=0%), with this change we sacrifice the part of the PCB which objective was to compare the sensing layer resistance with the objective resistance, and from now on the decision will be taken with the resistance of the sensing layer, which can be measured with the voltage that drops in that layer.

Making those updates, the circuit no longer saturated and we were able to appreciate its drift while having a control applied.

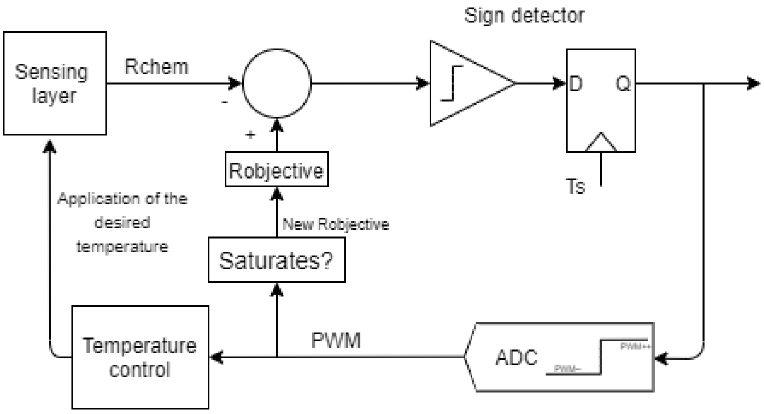


Figure 24: First order sigma delta with PWM and Robj control

3.7. Calibration/Characterization of a sensor

As the sensor used has two parts, the chemical resistance which reacts to the gas, and the heater resistance used to set the temperature. First, an independent characterization of the heater needs to be done, and afterwards the couple heater-sensor can be characterized jointly.

In order to explain the characterization of the sensors, we will use as an example the characterization of the CCS801.

3.7.1. Characterization of the heater resistance

For an optimal use of the heater, it is needed that the heater applies a desired Temperature into the chemical resistance. In order to know which temperature the circuit is providing, a good characterization of the heating element is needed.

To characterize it we measure its resistance at various known temperatures inside an oven. Also, we know from "*CCS801 Application Note Design Guidelines"* [4] the formula of the resistance of the heater is:

$$R = (R_0 - CR_0 + DR_0) \cdot [1 + \alpha(T - T_0) + \beta(T - T_0)^2] + CR_0 - DR_0$$

Being:

R the target heater resistance at target temperature T

$\alpha = 2.05 \cdot 10^{-3} \mathrm{K}^{-1}$

$\beta = 0.3 \cdot 10^{-6} \mathrm{K}^{-2}$

R_0 is the resistance of heater at a known temperature

C=0.24

D=0.063

Which for ease of use, can be simplified as:

$$R(T) = R_0 \cdot [1 + \alpha(T - T_0)]$$

The measures taken in the oven gave us a $R_0 = 39.3\Omega$ for a $T_0 = 57^{\text{º}}C$ or $331.15K.$ So the result of the characterization of the system is:

$$R(T) = 39.3\Omega \cdot [1 + 2.05 \cdot 10^{-3} \mathrm{K}^{-1}(T - 57^{\text{º}}C)]$$

3.7.2. Calibration of the chemical resistance and its combination with the heater resistance

Once the heater is characterized, the Rchem must be characterized. First it must be characterized for different temperatures, and its variations. Our control will work with two temperatures, one in the recommended range of the Chemical at 335ºC, and one temperature out of the recommended range, but close enough to make the conversion sufficiently fast, which is 240ºC, those values it has been chosen the used in the “Open loop testing for optimizing the closed loop operation of chemical systems” [1], but it is different for the other sensors, in which in order to choose them, we needed to comply with the datasheet or with the instructions from our colleagues at URV and to make trial and error tests in order to end up with the best suiting temperatures.

In order to characterize the sensor response to different temperatures and different gas concentrations, we measure its resistance at different commuting rates to accomplish different average temperatures, and with different ethanol concentrations. The results are shown in the figure 25.

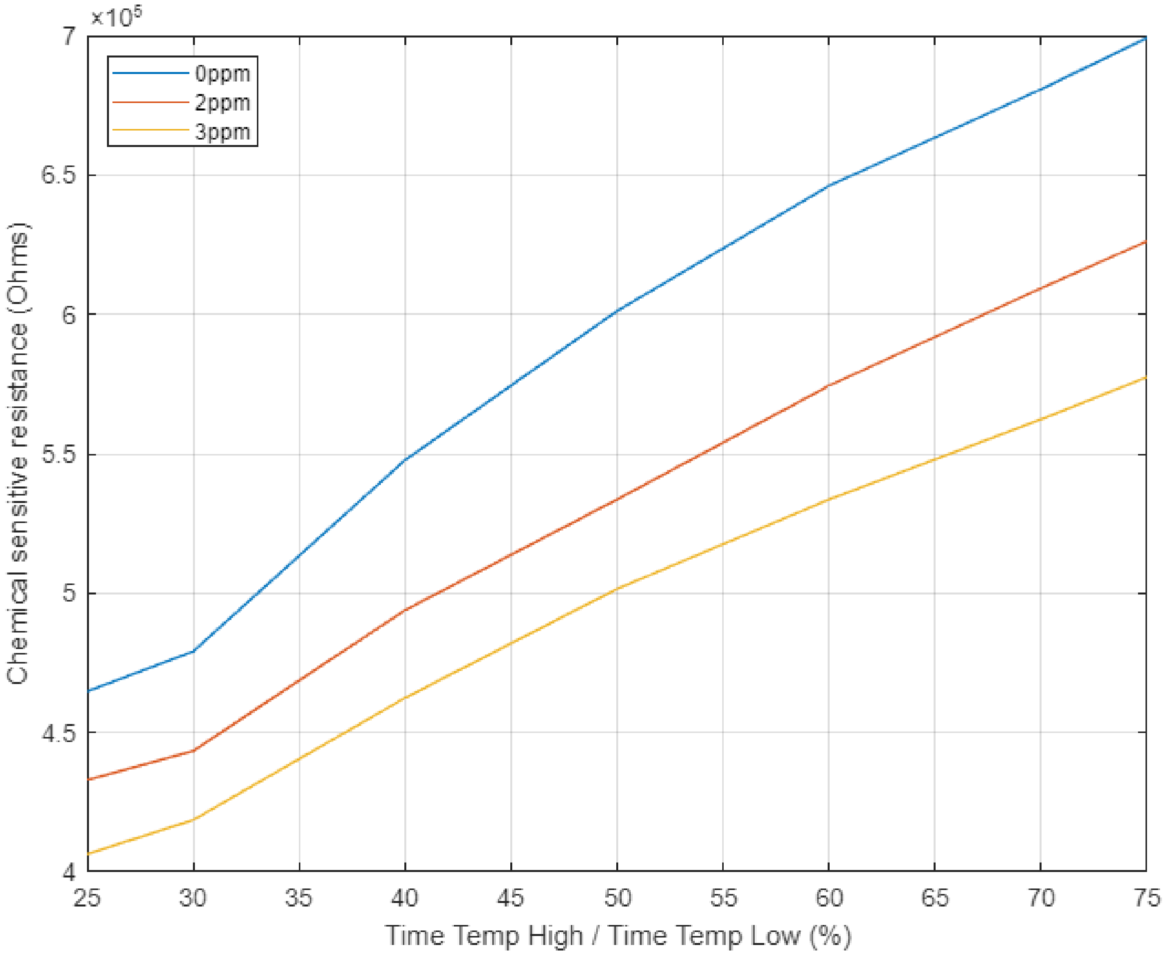


Figure 25: Characterization of the CCS801

Observing the previous graph, we are interested in an objective resistance of around 500k Ohm, so we are in range to measure all the desired concentrations of the gas. The selection of this value could be higher, but as the datasheet of the CCS801 claims that the lifetime at a continuous tension of 1.4V is superior to 5 years, the lower average temperature was chosen so less power is needed. A higher objective resistance could improve the resolution of the measurements, as the average temperature for the different gas concentration spreads at higher resistances. In any case, higher objective resistances have not been chosen for the experimental phase of the project as a higher resolution was not needed.

4. Results

4.1. Temperature control

The first experiment was to detect if the closed loop Wheatstone circuit with the CCS801 sensor, which was designed in order to control the temperature by controlling its resistance, was working properly. In order to do this test, a first simulation of the first control was applied. In that simulation, the system sent the information of 4 bits periodically (3 bit0 and 1 bit1). To take the measurements, the USB-6002 was used and the voltages on the heater resistance (Vheater) and on the feedback wire (Vref) were measured. The objective temperature values were 335ºC and 240ºC.

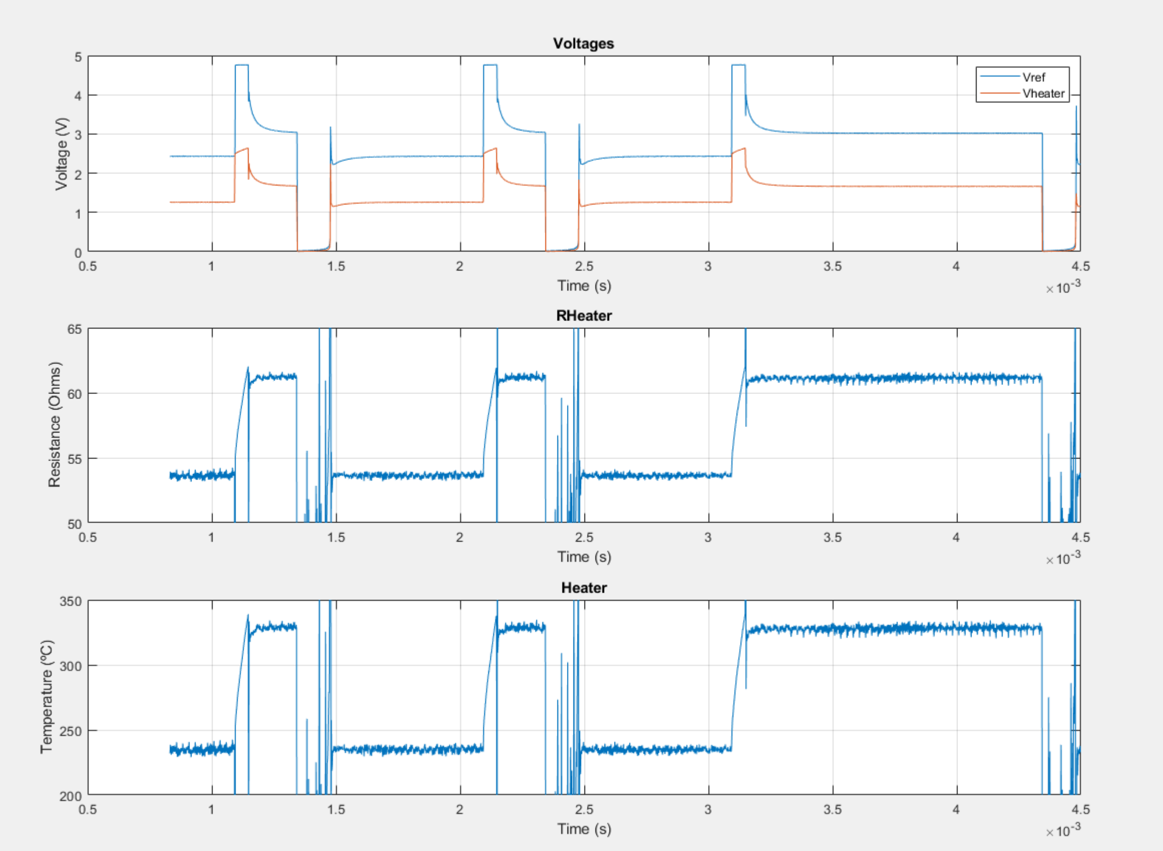


Figure 26: Result of temperature control test

As it can be seen in the previous figure 26, the first plot has the two measured voltages. With those voltages and knowing that the resistance between Vref and Vheater was fixed, the heater resistance was easily calculated and therefore, thanks to the characterization of the sensor, the sensor temperature could be known and plotted.

This test was accomplished successfully and the result proved that a correct control of the temperature was accomplished.

4.2. Open loop analysis

In order to test the sensor CCS801 with an open loop configuration and to be able to compare its response to the next experiments with a control applied, a test was developed using a constant temperature, 335ºC which is at its optimal working point. In order to maintain this temperature, the feedback Wheatstone bridge was used, and the measures were taken by the inboard ADC which was controlled by the NUCLEO F030R8 and logged the voltage differential at the chemical layer. In that example, different gases were applied, first it was used 50 sccm of synthetic air, at 11h 2ppm of Ethanol where applied while conserving a total flow of 50 sccm and 15 minutes later, the gas was set to be only synthetic air again.

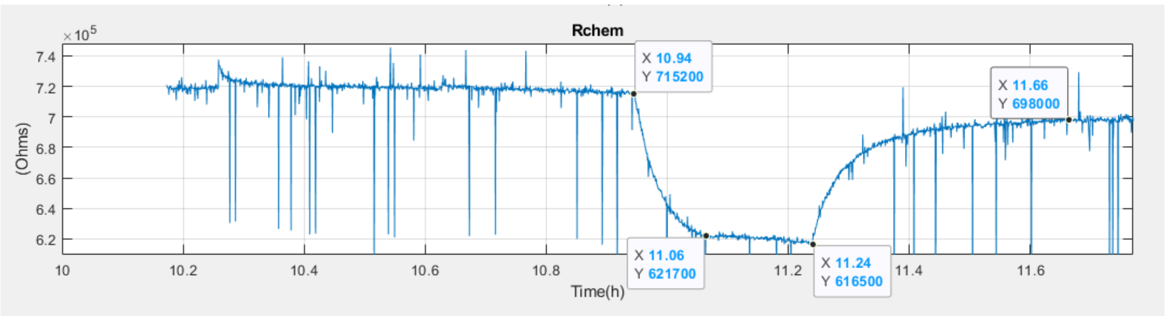


Figure 27: Result of the open loop test

Knowing that one terminal of the chemical layer is connected to the ground and the other terminal to a 500 kohms resistor which has its other terminal connected to 5V, it acts as a voltage divider, and therefore it's resistance can be calculated and plotted as it can be seen in figure 27.

This experiment gave interesting information, the reaction time (the time that takes to the sensor to stabilize after a change in the gas concentration) is calculated to be of 7.2 minutes from 0ppm to 2ppm of ethanol, and 25.2 minutes from 2ppm to 0ppm of ethanol.

This result gave us the opportunity to compare future results, and to see that there is margin of improvement with the reaction time of the sensor.

NOTE: The glitches seen in the results showed us that more work needed to be done on the sampling procedures.

4.3. Sigma delta First order

In order to test the first order sigma delta with the CCS801, the control was programmed and applied to the NUCLEO F030R8 board and a Rchem objective of 200 kohms was chosen, this Rchem is different from the one calculated on the 3.7.2 (Characterization of the sensor), because it was not the same sensor and their sensing layer electric resistance varies with different sensors. For this test, the same concentrations of the previous experiment were applied, while maintaining a constant gas flow (50 sccm), during the first 15 minutes the system applied only synthetic air, later on it applied 2 ppm of ethanol during 30 more minutes and finally during 1 hour the system applied just synthetic air again.

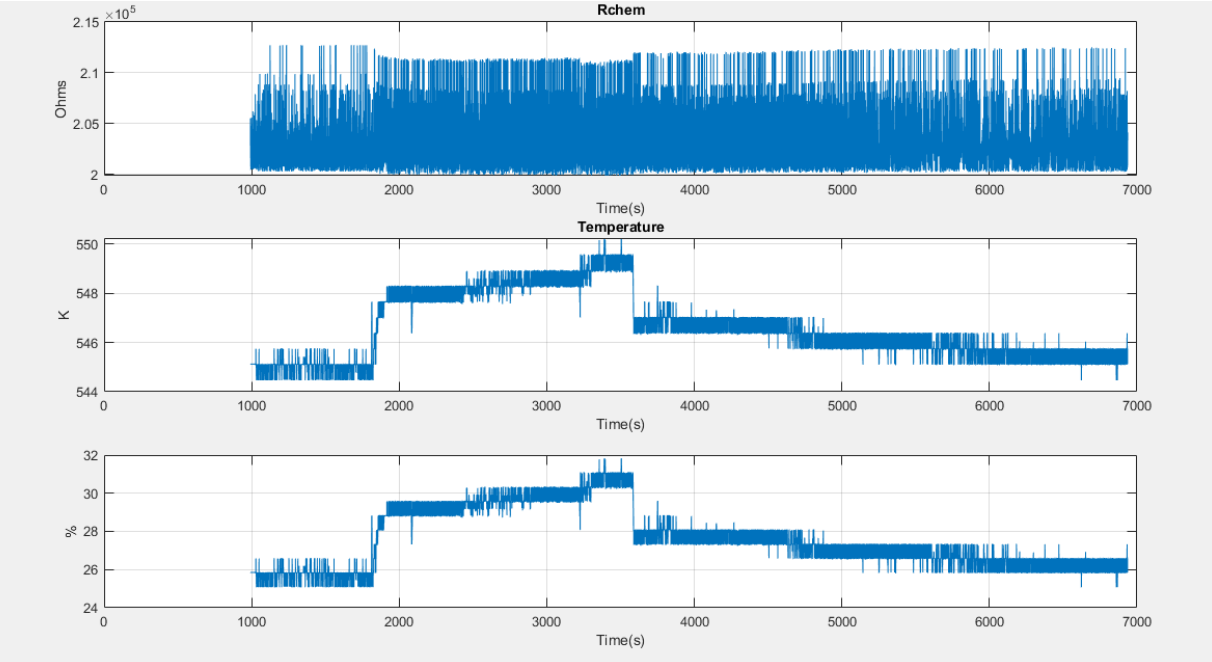


Figure 28: First order sigma delta test

As it can be seen in the images the response of the system is faster with the control applied, from 0 to 2 ppm of ethanol it took 2 minutes to stabilize while with open loop it took 7.2 minutes. But for the system to stabilize from 2ppm to 0ppm, the response was almost immediate, around 10 seconds.

What can be improved is the output resolution, the output value (mean temperature) is the mean value of the last 100 bits. So, if we want to improve the resolution, more bits must be used to calculate the mean, therefore the system can become slower at its output.

4.4. Sigma delta second order

In order to test the second order sigma delta with the CCS801 the experiment developed was similar to the previous experiments, with the control programmed and applied to the NUCLEO F030R8, a constant flow of gas was applied to the sensor (50 sccm), and different concentrations of ethanol where applied 0ppm-2ppm-0ppm. The output temperature was calculated using the mean of the last 100.

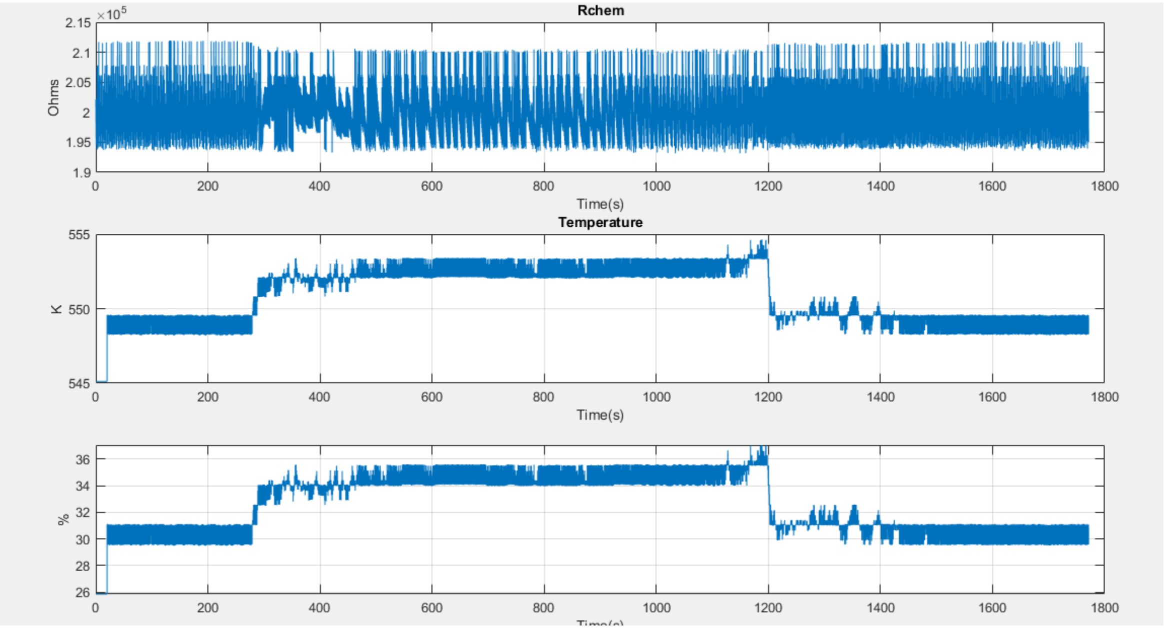


Figure 29: Second order sigma delta test

As a result of applying the second order sigma delta with less resolution, the system is faster than before, to stabilize from 0 ppm to 2ppm it just takes 20 seconds. And from 2ppm to 0ppm it takes even less, 10 seconds. But the resolution is unacceptable, so the knowledge acquired with this test is that we need to improve that aspect of our control.

4.5. First Order Sigma delta with PWM

To test the 1rst Order Sigma delta with PWM using the CCS801, we did the same experiment done in 4.4 but with the new control. A constant flow of gas was applied (50 sccm), and we started with 45 minutes of synthetic air, then 2ppm of ethanol where applied for one and a half hours, finally just synthetic air was applied for one hour.

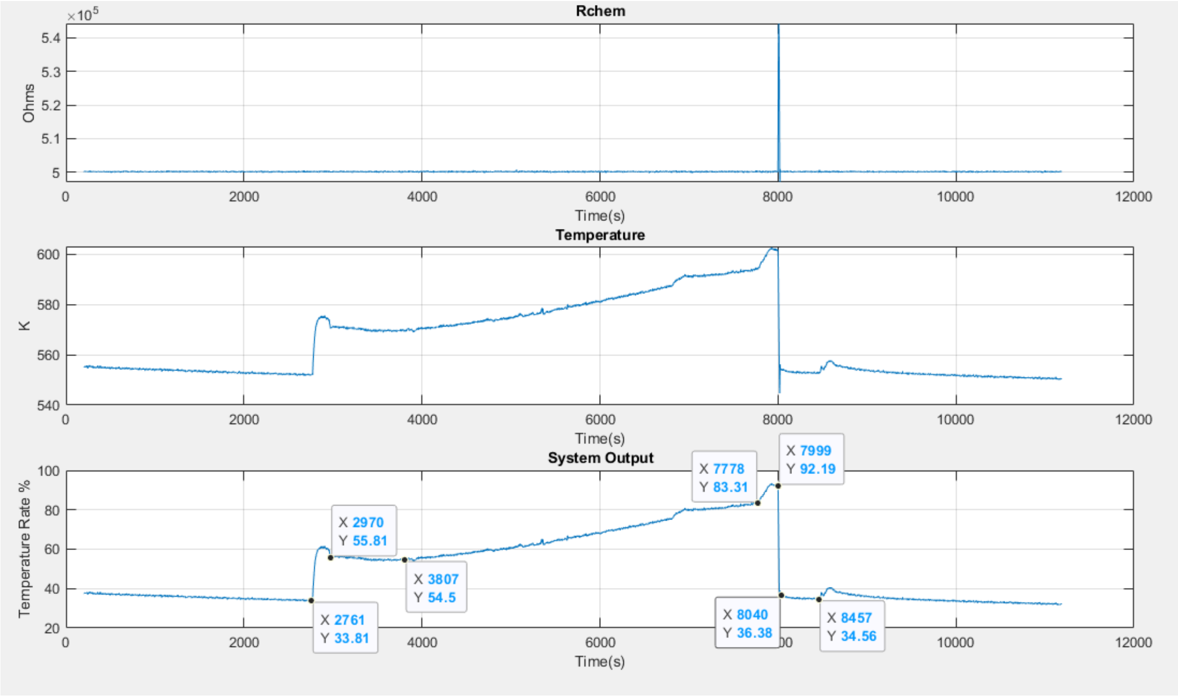


Figure 30: First order sigma delta with PWM test

As it can be seen in the previous graphs the resolution is improved, and the response of the system from 0ppm to 2ppm takes 3 minutes, which is a good trade-off if we consider how bad was the resolution with the previous controls. Also from 2ppm to 0ppm it takes 40 seconds.

In this figure can also be seen the temporal drift of the sensor (it can be clearly seen from the second 3807 to the 8000), and it made us realize that the Robjective could not be fixed if we did not want the system to saturate.

4.6. First Order Sigma delta with PWM and Robj change

In order to test the first order sigma delta with PWM and Rchem target change, the test was the same that was done with the rest of the controls. First 5 minutes with 0ppm of ethanol, then 10 minutes with 2 ppm of ethanol and finally 0ppm again.

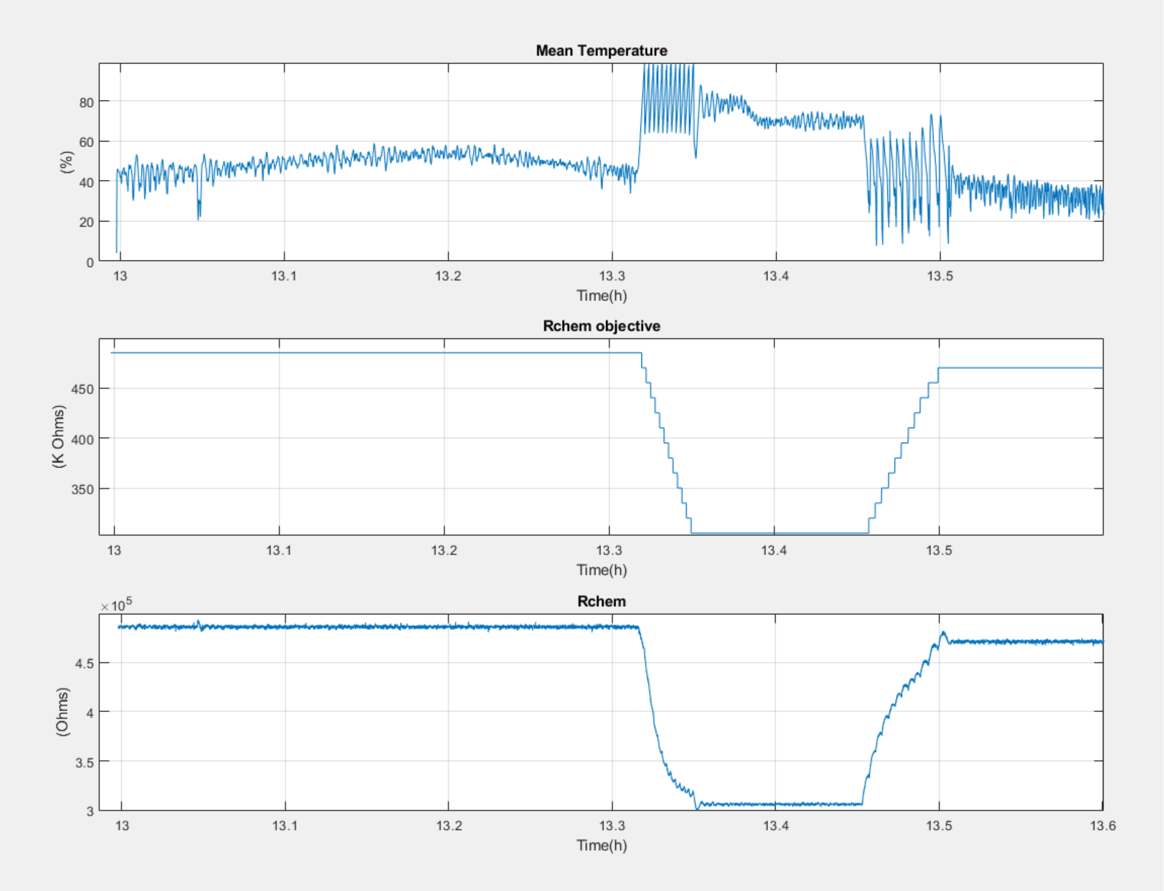


Figure 31: First order sigma delta with PWM and Robj change test

With this experiment the result presented the response to saturation and a stabilization time from 0ppm to 2ppm of 5 minutes, due to the time that the system needs to reach the proper sensing layer target resistance. And from 2ppm to 0ppm it took 3 minutes, this difference is due to the new target, which is closer to the target resistance of the 2ppm.

Also, as it can be seen the resistance target at 0ppm is different at the start of the experiment to the one in the end of the experiment. This can be due to the sensing layer drift and due to the fact that the system can be stable at different resistances while being exposed to the same gas concentration.

5. Budget

This chapter contains all the components that have been needed in order to create the testing prototype with which this project has been done. It also includes the hours that have been dedicated to the thesis, evaluated at cost of junior engineer.

List of components:

Component name	**#**	**Price**
SMD and THT Resistances and condensers	-	50€
TXB0108PWR (Level shifter)	1	1.38€
TXB0104PWR (Level Shifter)	2	1.86€
TLV4110ID (OpAmp)	2	5.06€
90 degrees pin connector	1 bundle	3.47€
IC socket	1 bundle	4.40€
Pin connectors	5 bundles	2.82€
Jumper	5	1.86€
Test points	10	3.04€
DAC8830IBD	1	12.73€
SN74LV4052AD (MUX)	1	0.47€
INA188 (OpAmp)	1	3.48€
AD8628ARZ (Op amp)	1	1.36€
LTC1865ACS8 (ADC)	2	36.96€
NUCLEO F030R8 (Digital board)	1	15.22€
	TOTAL:	**144.22€**

For a European junior engineer, the salary is around 2500 €/month, as I have been working in this project around 8h a day for 5 months, the budget destinated to pay a junior engineer to do this work should be around **12500 €.**

6. Conclusions and future development:

The hardware developed, the application of the different controls, and the results obtained, demonstrates that a good electronic implementation of the temperature-controlled gas sensor has been accomplished, and faster response times and lower price objectives have been obtained.

For future investigation, I would focus on the idea of making the system commercially viable, for this reason, more research should be done in the field of the controls. I would recommend researching ways to eliminate the electrical resistance drift from the equation, for example, it can be a good idea to try to make a commutation between two objective resistances and taking the difference mean temperature between the two resistances as the output value. The two objective resistances should be able to change their values in case that one of them saturates, and the difference between those resistances should always be the same [5].

Redesigning the control to apply a UV light [6] instead of temperature can also be a good approach.

To summarize, this thesis has proven that such a system can be implemented, and it provides a first step towards a future commercialization of this type of controls for gas sensing systems.

Bibliography:

[1] M Dominguez-Pumar, J.M. Olm, L. Kowalski, V. Jimenez, "Open loop testing for optimizing the closed loop operation of chemical systems," 2020.

[2] N. Barsan, U. Weimar, "Fundamentals of Metal Oxide Gas Sensors," in *The 14th International Meeting on Chemical Sensors* , 2012.

[3] J. H. Watmuff, "An Investigation of the Constant-Temperature Hot-Wire Anemometer," *Elsevier,* 1994.

[4] AMS, "Application Note and CCS801 Design Guidelines," 2010. [Online]. Available: https://www.sciosense.com/wp-content/uploads/2020/01/CCS801-Application-Note-Design-Guidelines.pdf.

[5] R. Ionescu, E. Llobet, S. Al-Khalifa, J.W. Gardner, X. Vilanova, J. Brezmes, X.Correig, "Response model for thermally modulated tin oxide-based microhotplate gas sensors," *ELSEVIER,* 2003.

[6] L. Kowalski, E. Navarrete, E. Llobet, M. Dominguez-Pumar, "Control of surface potential in WO3 gas sensors using UV light," *IEEE,* 2019.

Appendices:

In these appendices the final designed circuit and the programmed control will be added for more information.

1 Software appendix

The code has been written in C++ using the online compiler of os.mbed.com, the code as it can be seen, is commented:

```
#include "mbed.h"

/*************************************************************

 C H E M 2 0 2 0 _ F 0 3 0

 Code for closed loop operation of chemical sensors

 System has two outputs
    sample (D9+D8): Generates sample signal for LTC1865 ADC
                        Both D9 and D8 shall connected together
                        as D8 provides ISR launch feedback
                        Signal period is SAMPLE_PERIOD
                        Signal is high during SAMPLE_TIME_UP

    dout   (D2) : Digital heater control
                    State is updated in time TIMEOUT1 after
                    sample falling edge

 Data from the ADC is read using SPI signals:
    MOSI (D4) : Signal from MCU to ADC (SDI)
    MISO (D5) : Signal from ADC to MCU (SDO)
    SCLK (D3) : Serial clock            (SCK)

 ADC readings are sent trough serial over USB
 **************************************************************/
```

```
// Constants ------------------------------------------------------------------

// Timing data
#define SAMPLE_PERIOD  0.001     //   1ms Main period
#define SAMPLE_TIME_UP 0.00004   //  40us Time up for sample
#define TIMEOUT1       0.0005    // 400us Timeout 1

// ADC SPI frequency
#define ADC_CLOCK    1000000     // 1 MHz clock

// ADC MOSI Inputs
#define SPI_SINGLE_0 0x8000      // Single Ended / Channel #0
#define SPI_SINGLE_1 0xC000      // Single Ended / Channel #1
#define SPI_DIFF_01  0x0000      // Differential (+)0 (-)1
#define SPI_DIFF_10  0x4000      // Differential (+)1 (-)0

// State constants
#define FRAME_READS      160       // Number of reads in a frame
                                   // Shall be even as we need two
                                   // ADC reads for one full reading

// Hardware Configuration -------------------------------------------------

Serial pc(SERIAL_TX, SERIAL_RX, 115200);   // Serial link with PC
SPI spi(D4,D5,D3); // mosi, miso, sclk
SPI spi2(PB_15,PB_14,PB_13); // mosi, miso, sclk


PwmOut      sample(D9);   // Timer Hardware Sample Signal on D4
InterruptIn isample(D8);  // Sample feedback from D4 to D5 to issue ISR

DigitalOut dout(D2);

// Objects ---------------------------------------------------------------------

// Timeout object
Timeout tout1;    // Timeout 1

// Global Variables ------------------------------------------------------

// Current frame cycle
int frameCycle = 0;

// State for the system output
int state = 0;

//DecisionData
int decidata = 0x0000;
int PWM = 60;
int Rchem_obj = 0x01F4;//500
int Rstep = 0x00F;//15
float Vch_obj=5*((float)Rchem_obj)/(((float)Rchem_obj)+500);
float Vch=0;
```

```
// Current read data
int data = 0;
int data2 = 0;

// Decision Code -------------------------------------------------------
// Determine new state from current reading
static inline void computeNewState(void)
 {

 // In the last 40 frames the state is always 1
 if (frameCycle > (FRAME_READS-41)) {
    state = 1;
    }
else{ // The decision is taken in function of the PWM value
    if (120-frameCycle < PWM){
        state = 1;
        }else{
        state = 0;
        }
    }
 }

// Kernel Code ---------------------------------------------------------
// Timeout 1 ISR
// This function is called on timeout 1 from sample ISR
void timeout1ISR(void)
 {
     //The bytes that will be sent to the computer
 int byte0, byte1, byte2, byte3, byte4, byte5, byte6, byte7;

 // Set new state value on dout output
 dout = state;

  if (frameCycle == FRAME_READS-2){
    decidata=data2;
    Vch=(float)decidata*5/65535;
    if (Vch<Vch_obj){
            PWM++;
            if(PWM>120){ //If the control saturates
                PWM=40;
                Rchem_obj=Rchem_obj-Rstep;
                if (Rchem_obj<1)Rchem_obj=Rstep; //So Rchem_obj is never 0
                Vch_obj=5*((float)Rchem_obj)/(((float)Rchem_obj)+500);
                }
        }else{
            PWM=PWM-1;
            if(PWM<0){ //If the control saturates
                PWM=40;
                Rchem_obj=Rchem_obj+Rstep;
                Vch_obj=5*((float)Rchem_obj)/(((float)Rchem_obj)+500);
                 };
        }

 }
```

```
// Send serial data

// Framing protocol for sending 8 bytes
// 1rst byte has msb(most significant bit) at "1"
//        Bit 7 : "1"
//        Bit 6 : "1" on frameCycle 0, "0" elsewhere
//        Bit 5 : Current state
//        Bit 4 : Reading channel
//        Bit 3 : Reading bit 15 (of the 1rst ADC)
//        Bit 2 : Reading bit 14 (of the 1rst ADC)
//        Bit 1 : Reading bit 13 (of the 1rst ADC)
//        Bit 0 : Reading bit 12 (of the 1rst ADC)
// 2nd byte has msb at "0"
//        Bit 7 : "0"
//        Bits 6..0 : Reading bits 11..5 (of the 1rst ADC)
// 3rd byte has msb at "0"
//        Bit 7 : "0"
//        Bits 6..2 : Reading bits 4..0 (of the 1rst ADC)
//        Bits 1..0 : Reading bits 15..14 (of the 2nd ADC)
// 4th byte has msb at "0"
//        Bit 7 : "0"
//        Bits 6..0 : Reading bits 13..7 (of the 2nd ADC)
// 5th byte has msb at "0"
//        Bit 7 : "0"
//        Bits 6..0 : Reading bits 6..0 (of the 2nd ADC)
// 6th byte has msb at "0"
//        Bit 7 : "0"
//        Bits 6..0 : Reading bits 15..9 (of the  Rtarget)
// 7th byte has msb at "0"
//        Bit 7 : "0"
//        Bits 6..0 : Reading bits 8..2 (of the  Rtarget)
// 8th byte has msb at "0"
//        Bit 7 : "0"
//        Bits 6..5 : Reading bits 1..0 (of the  Rtarget)

  // First ADC
  byte0 = (data>>12)&0x0F;
  byte1 = (data>>5)&0x7F;
  byte2 = (data<<2)&0x7C;
  //Second ADC
  byte2 |= ((data2>>14)&0x03);
  byte3 = (data2>>7)&0x7F;
  byte4 = data2&0x7F;
  //Rtarget
  byte5 =((Rchem_obj>>9)&0x7F);
  byte6 =((Rchem_obj>>2)&0x7F);
  byte7=((Rchem_obj<<5)&0x60);

   // Add the rest of bits
  byte0 |= 0x80;                              // Bit 7 : Always "1"
  if (!frameCycle) byte0 |= 0x40;             // Bit 6 : "1" on frameCycle 0
  if (state) byte0 |= 0x20;                   // Bit 5 : Current dout state
  if (!(frameCycle&0x01)) byte0 |= 0x10;      // Bit 4 : Reading channel
```

```
     // Send serial data
      pc.putc(byte0);
      pc.putc(byte1);
      pc.putc(byte2);
      pc.putc(byte3);
      pc.putc(byte4);
      pc.putc(byte5);
      pc.putc(byte6);
      pc.putc(byte7);


 // Update frameCycle
 frameCycle++;
 if (frameCycle >= FRAME_READS) frameCycle = 0;

 }

// Sample ISR (Interruption Service Routine)
// This function is called on the falling edge of isample
void sampleISR(void)
 {

 // Set timeout to set new state
 // Includes 40us error equalization
 tout1.attach(&timeout1ISR,TIMEOUT1-0.00004);

 // Read SPI data
 // As sample is used also as CS, no explicit CS is needed
 // We just read a 16 bit SPI word
 // We read different data on even and odd cycles
if (frameCycle&0x01){
     data=spi.write(SPI_SINGLE_1);
     data2=spi2.write(SPI_SINGLE_1);
    }else{
     data=spi.write(SPI_SINGLE_0);
     data2=spi2.write(SPI_SINGLE_0);
    }


 // Update state value from current reading
 computeNewState();
 }

// Initialization Code --------------------------------------------------

// Initialize timings
void timingInit(void)
 {
 // Generate sample signal using hardware PWM
 sample.period(SAMPLE_PERIOD);
 sample.write(SAMPLE_TIME_UP/SAMPLE_PERIOD);

 // Set isample feedback ISR
 isample.fall(&sampleISR);
```

```
 }

// Initialize SPI
void spiInit(void)
 {
 // Setup the spi for 16 bit data, high steady state clock,
 // second edge capture (rising edge), with a ADC_CLOCK clock rate
 spi.format(16,3);
 spi.frequency(ADC_CLOCK);
 spi2.format(16,3);
 spi2.frequency(ADC_CLOCK);
 }

// Program Main -------------------------------------------------------

int main()
 {
 spiInit();      // Configure SPI
 timingInit();   // Start timers and operation
 while(1) {};   // Do nothing while waiting for events
 }
```

2 Hardware appendix (Designed Circuit)

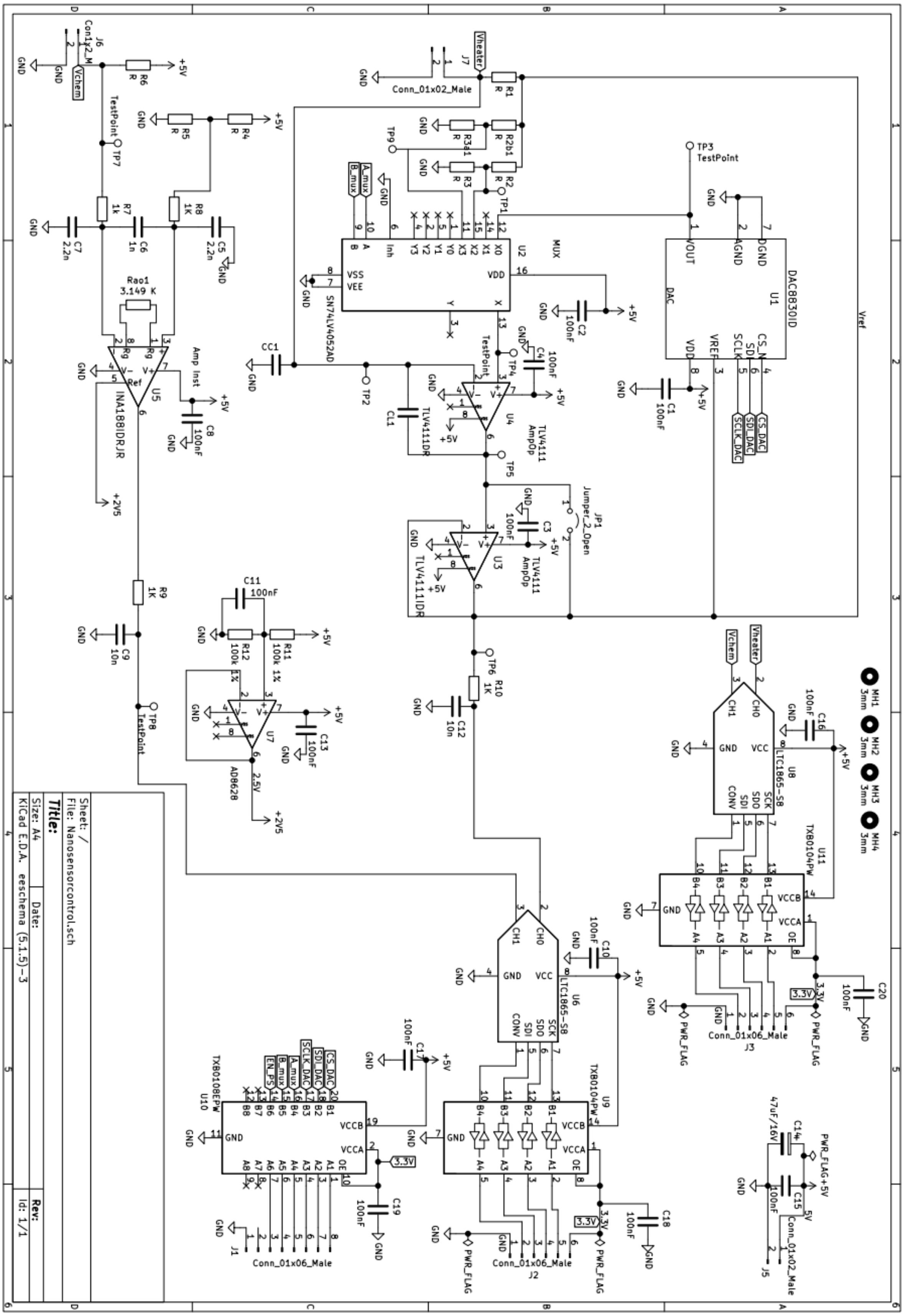


Figure 32: Designed circuit

3 Hardware appendix (Designed PCB)

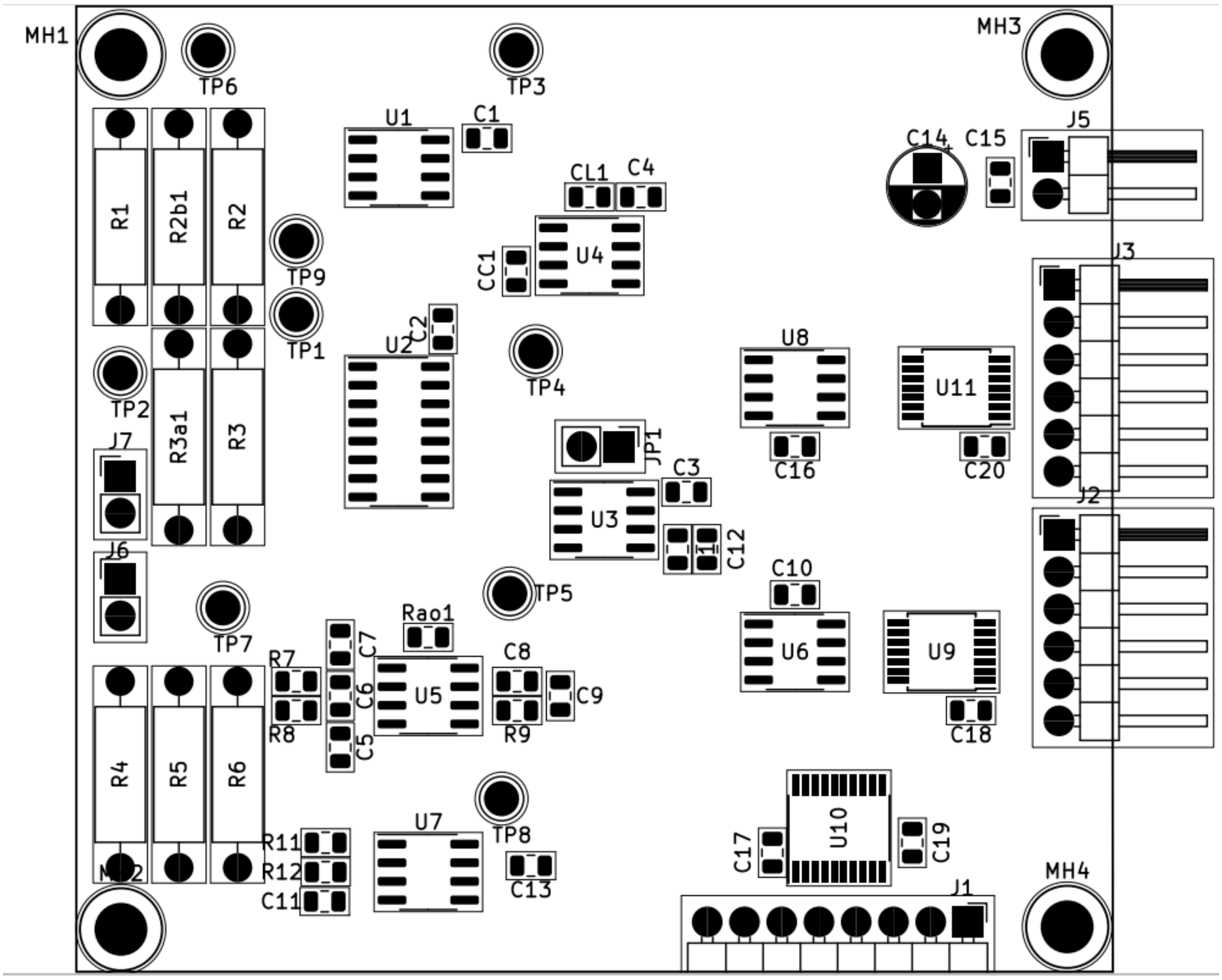


Figure 33: PCB layout

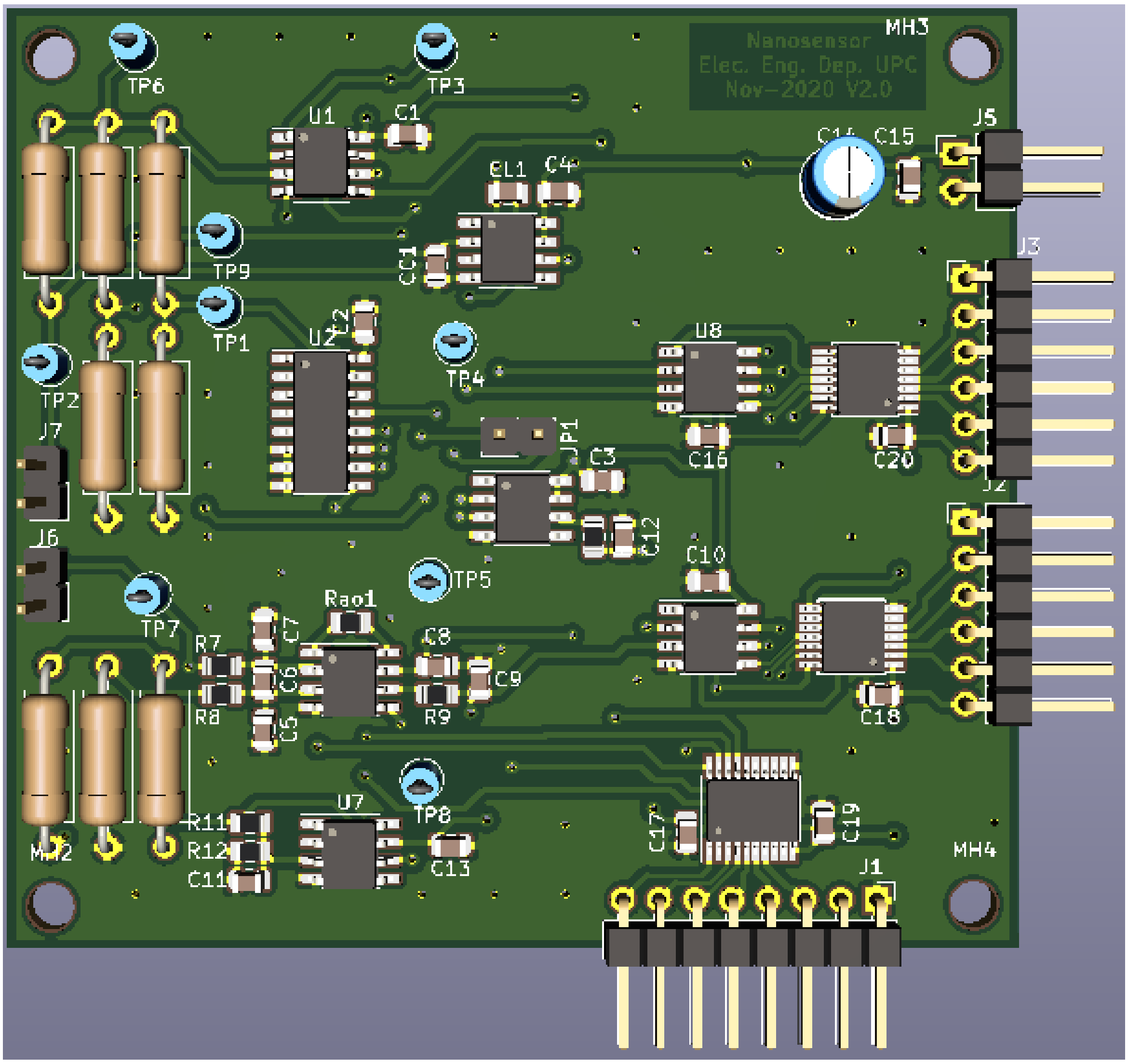


Figure 34: 3D PCB preview

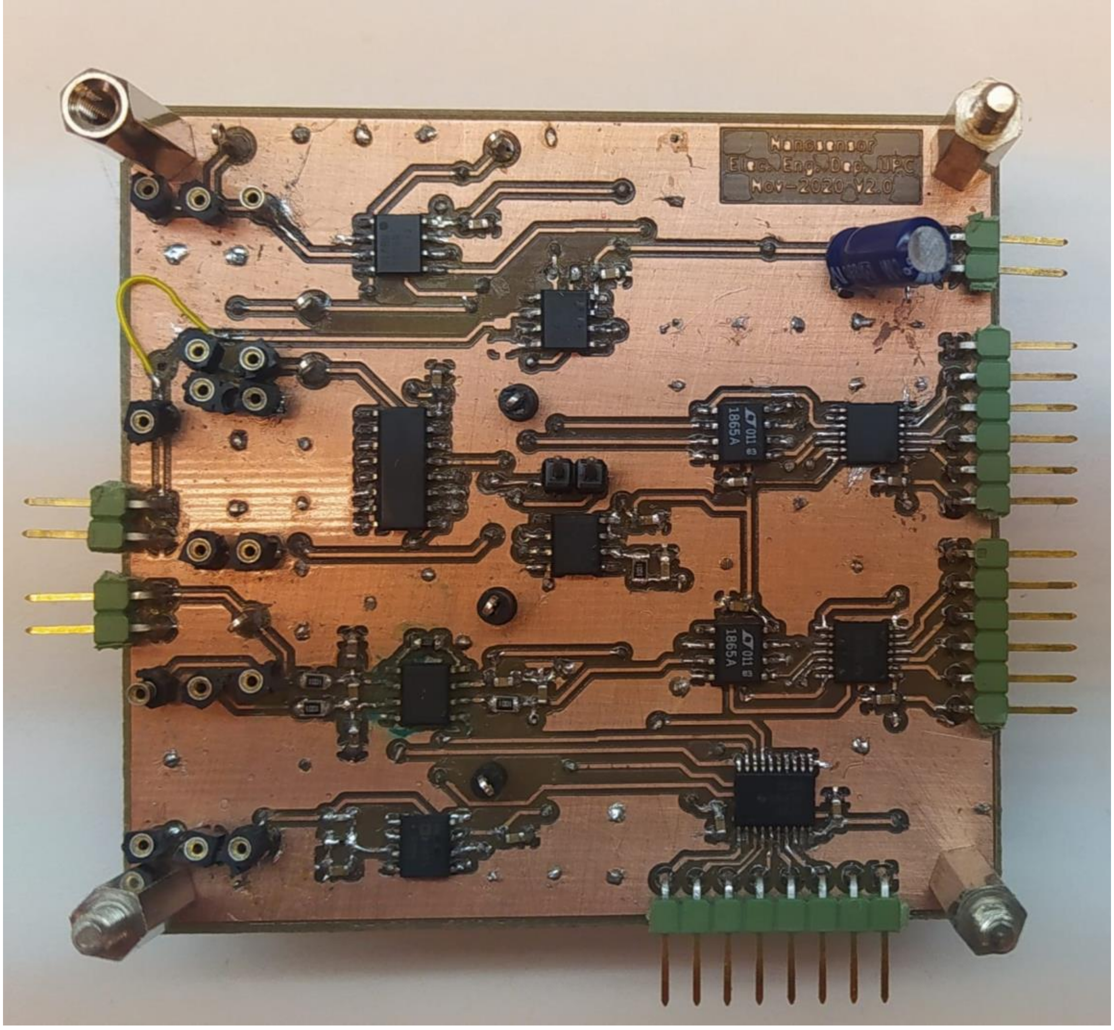


Figure 355: Printed PCB

Glossary

MOX sensors: Metal Oxide sensors.

MEMS: Micro-Electro-Mechanical Systems**.**

VOCs: Volatile Organic Compounds.

PCB: Printed Circuit board.

MUX: Multiplexer.

DAC: Digital to analog converter.

ADC: Analog to digital converter.

Vout: Voltage at the output.

Vin: Voltage at the input.

Rheater: Electrical resistance of the heating layer of the MOX.

Vheater: Voltage difference between the two electrodes of the heating layer of a MOX.

Vref: Voltage of the wire that closes the loop in the Wheatstone bridge.

Rchem: Electrical resistance of the sensing layer of a MOX.

OpAmp: Operational amplifier.

Robj: The target electrical resistance of the sensing layer.

Vout_chem: Output of the analog treatment of the sensing layer.

Thigh: High temperature, in the optimal working range of the MOX.

Tlow: Low temperature, outside the optimal working range of the MOX.

PWM: Pulse width modulation.

sccm: Standard cubic centimeters per minute.

ppm: Parts-per million.