



## 1. The first step before beginning to work with the PTGA is to open the gas bottles

Near the lab where the PTGA is located there is a Specific room where the bottles are contained. The following bottles are needed:

- Nitrogen to purge the head and the heated body of the thermobalance
- Air to perform oxidation
- Reduction gases (among which  $N_2$ ,  $CO_2$  and  $CH_4$  will be used in these tests).



Figure 1: The valves of the bottles in the specific room

Botellas para la PTGA					
N2	Nitrógeno técnico F50 P200	35 €	Messer		
Aire	Aire sintético 5.0 sin HC F50 P200	65 €	Messer		
CH4	Metano 2.5 F50 P200	169 €	Messer		
CO2	Dióxido de carbono técnico F50 37,5 kg	45 €	Messer		

Día	Hora	N2	5	6	7	8
		N2 CAHN	CH4	N2	CO2	Aire
7/2/22	10:40	70	190	160	45	160
8/2/22	10:20	60	190	155	50	160
9/2/22	9:15	50	190	140	45	160
11/2/22	9:45	30	190	125	50	150
15/2/22	9:15	25	190	120	50	150
17/2/22	11:45	15	185	105	45	140
22/2/22	14:00	200	190	100	45	150
23/2/22	10:05	190		80		
24/2/22	07:45	170		65		
7/3/22	10:30	115		45		
8/3/22	10:40	150		200		
15/3/22	12:00	140	200	170	45	150
16/3/22	11:30	140	195	160	50	150
17/3/22	9:32	140	200	150	50	150

Figure 2: Example of paper used to check the bottles state



Figure 3: The valves of the bottles in the wall near to the flow meters

**2. When beginning to work with the PTGA, as a first thing check always check that it is horizontal and correctly installed.**



Figure 4: Level indicators

If the balance is not horizontal or has to be adjusted in its position, this can be done through the following screws.





Figure 5: Screw to adapt balance position

**2. Check that the pressure inside the balance is zero before opening.**

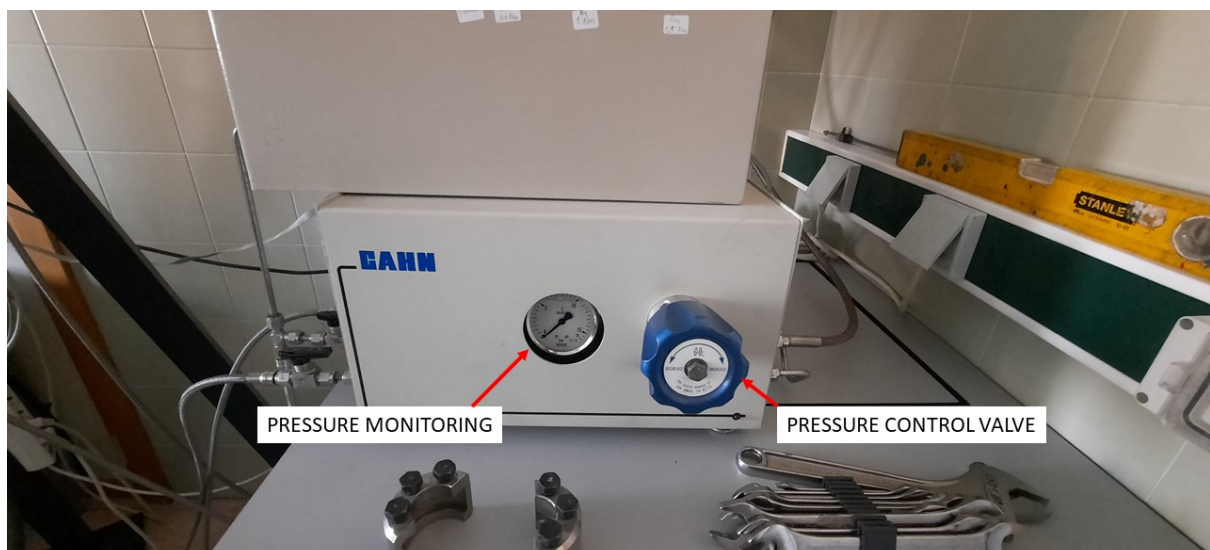


Figure 6: Pressure control valve



3. Check also that the valves at the outlet of the PTGA are correctly placed.

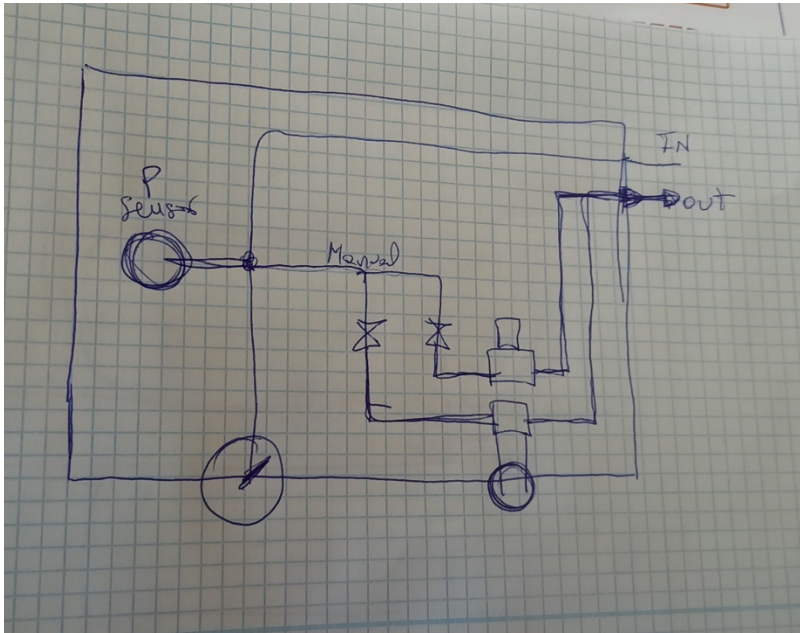


Figure 7: Position of the outlet valves

From figure 4 we see that there are two ways to regulate the pressure inside the PTGA: one is manual and one is automatic. The manual control will be the one which is used in most of the cases. So for this reason the valves have to stay in the position which is reported in figure 8.

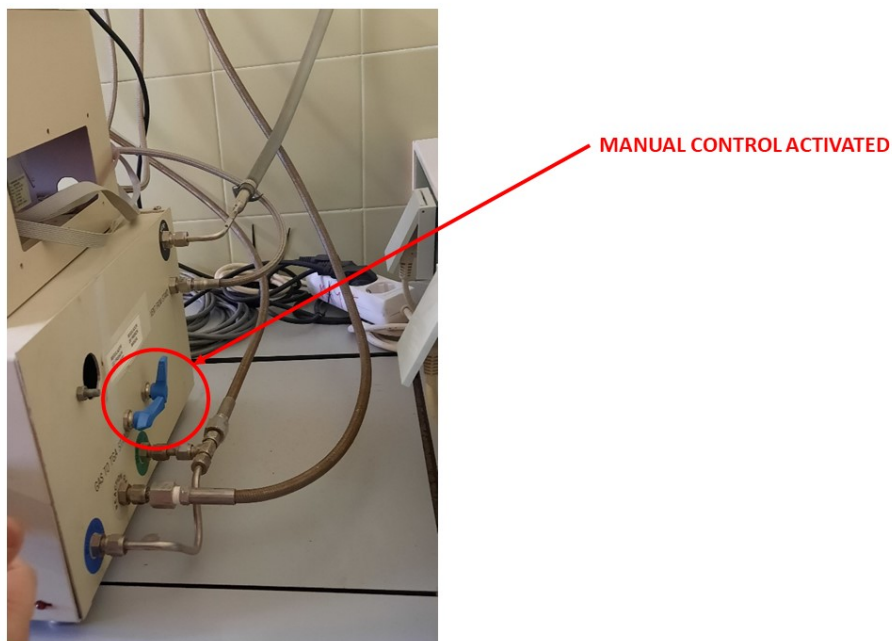


Figure 8: Outlet valves from the PTGA

The outlet of the PTGA is connected to the pressure control apparatus by using 2 valves.

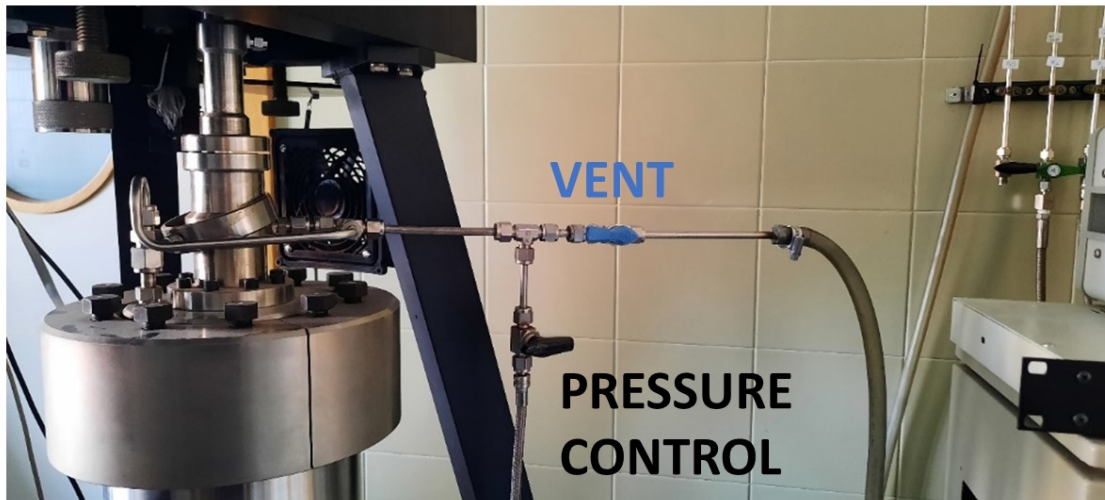


Figure 9: Gas flow regulation system

**4. Now to open the reactor the first thing is to put under the reactor a support to maintain its position even when the connection between the balance head and the heated reactor is not present anymore. See figure 10.**



Figure 10: In the left part of the figure it can be seen the PTGA when it is opened (with the support put in place); in the right part of the figure it can be seen the PTGA once it is closed

This is done by pushing the button which moves the sustain.

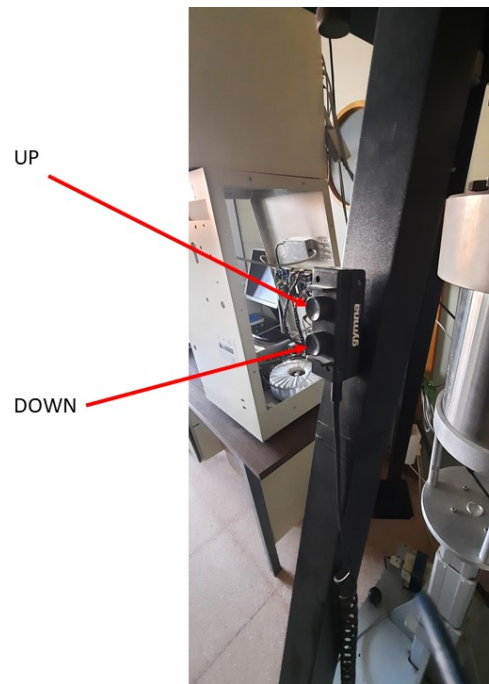


Figure 11: Command that moves the support for the heated body

**5. Once the heated body is now supported carefully, the two semi cylinders that close the connection between the balance head and the heated body have to be unlocked. See figure 12.**

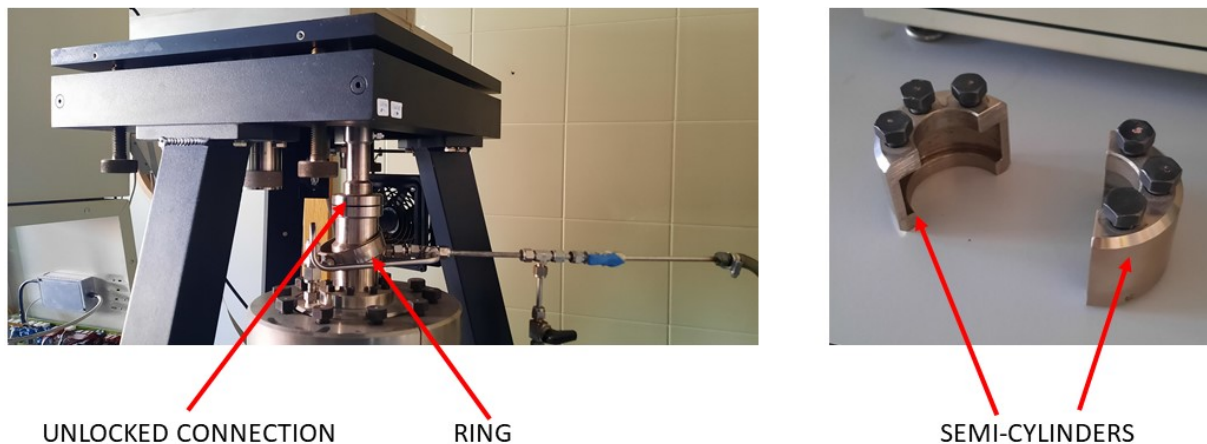


Figure 12: How to unlock the connection of the balance head from the heated body

The two semi-cylinders are also united by a ring with a screw. This has to be also unscrewed and when it is free the screw has to be placed again in the cylinder to avoid losing it.

## 6. Insertion of the sample.

Once the connection between the balance head and the heated body is removed, then the heated body can be moved down with the button shown in figure 11 to free the sample, which is hold on a basket of platinum and is supported by quartz wool. The basket needs to have same distance from the



walls of the electric furnace and does not have to touch the walls. Besides this the bottom of the basket is directly sitting on a thermocouple so it is important to maintain always the same length of the wire that holds the basket. The length of the wire and of the thermocouple are shown in figure 13.

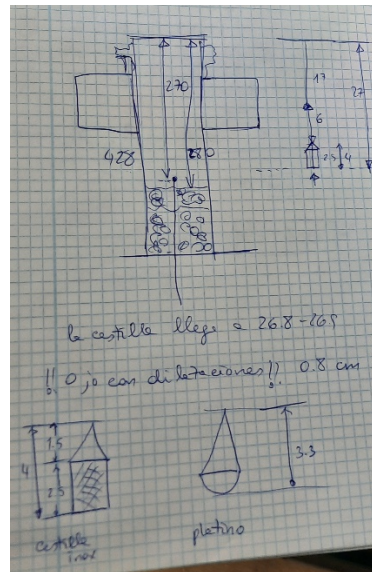


Figure 13: Dimensions of the wire and of the thermocouple

Once the balance it is opened it will look like in figure 14.

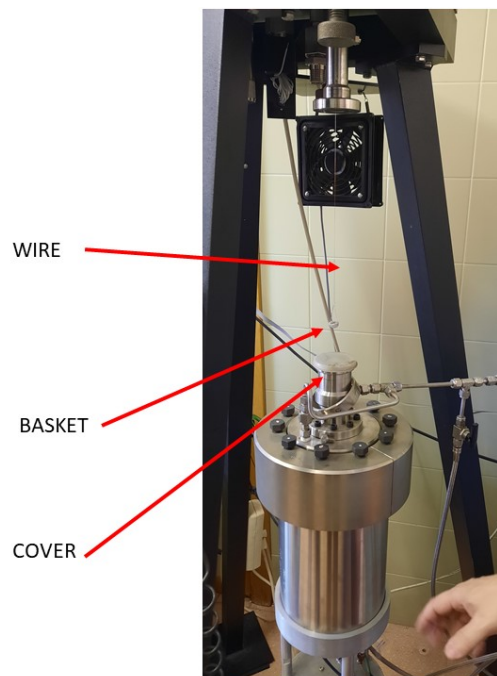


Figure 14: Opened PTGA

To position the sample inside the thermobalance a cover has to be put in the hole which is below the basket and at the entrance of the furnace in fact if some part of the sample will fall in the whole this will join the bed material in the furnace which is represented by quartz and inert materials.

Once the sample is placed in the balance the PTGA has to be closed repeating back the processes which have already been described when it was firstly opened. Then the furnace and the gas flow have to be controlled.

## 7. Switching on the furnace

To switch on the furnace we have 3 switches: two on the back and one in the front. See figure 15.



Figure 15: Switches of the furnace

## 8. Switching the gas control

To switch on the gas control the procedure is:

- Switch on the pc
- Switch on the gas measurement
- Switch on the pressure controller

While switching on the pc is done through the power button of the pc, obviously; the buttons to switch on the gas control apparatus is the one shown in figure 16.

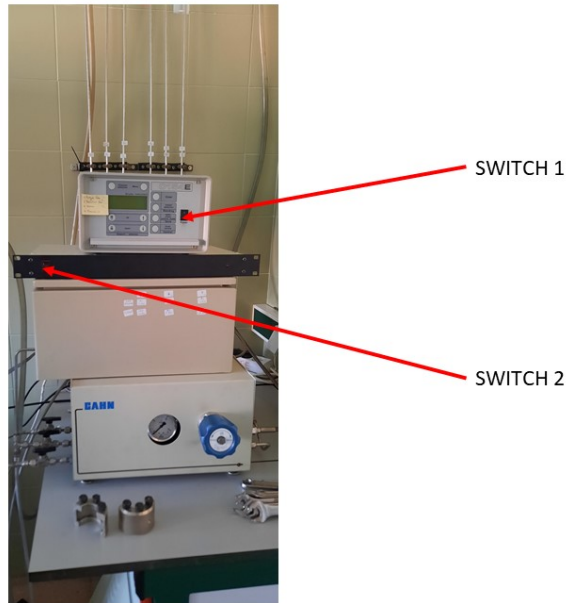


Figure 16: Switches for the gas apparatus (the third switch is in the back of the pressure controller)

## 9. Setting the heating method for the furnace

To set the method it has to be open the software “Cahn TG System software” in the pc. See figures 17,18 and 19.

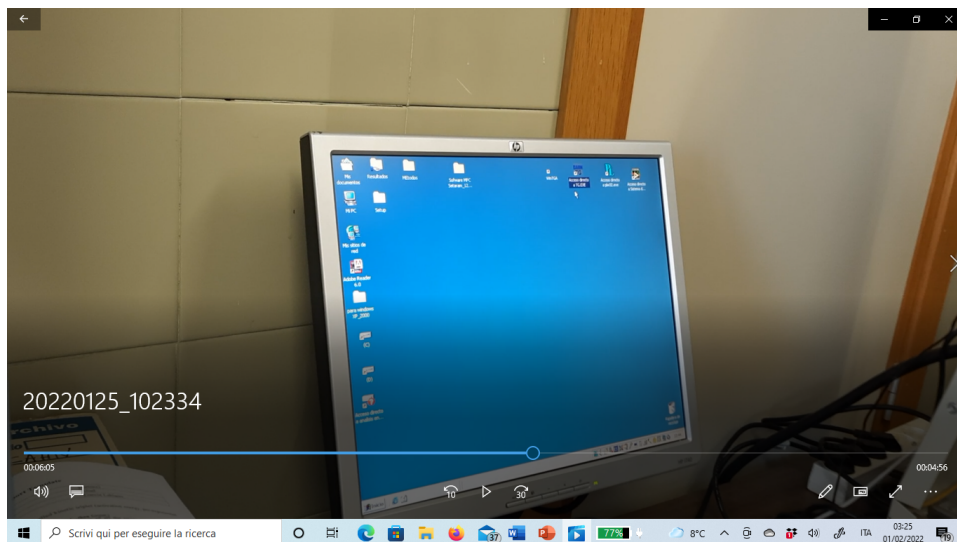


Figure 17: “Cahn TG System software” on the desktop



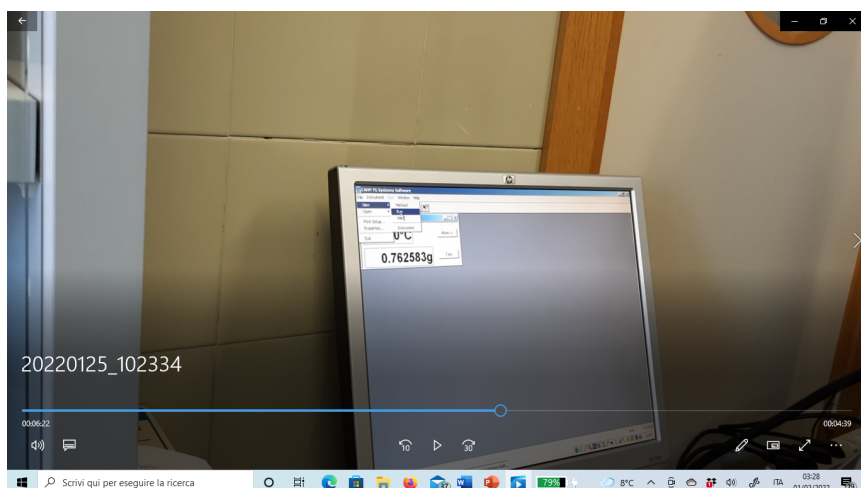


Figure 18: “Cahn TG System software” how to chose the new, method

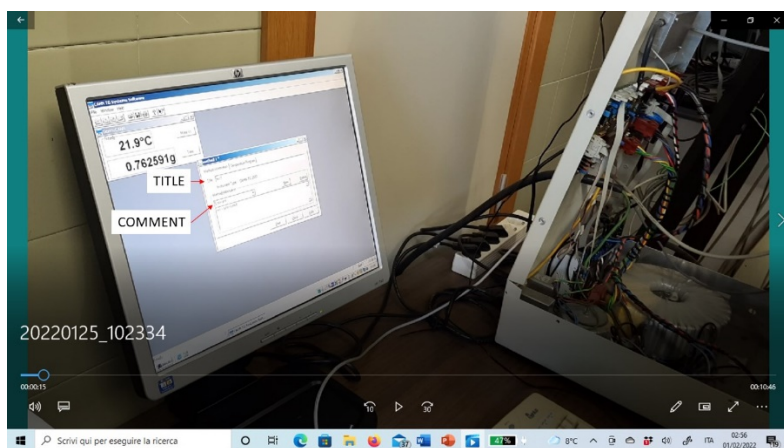


Figure 19: Cahn TG System software once opened

As a first step we have to build a new method which consists of all the heating ramps which we want to achieve for our sample. To do this we initiate a new method and then we insert: title, comments and date and operator. See figures 20 and 21.

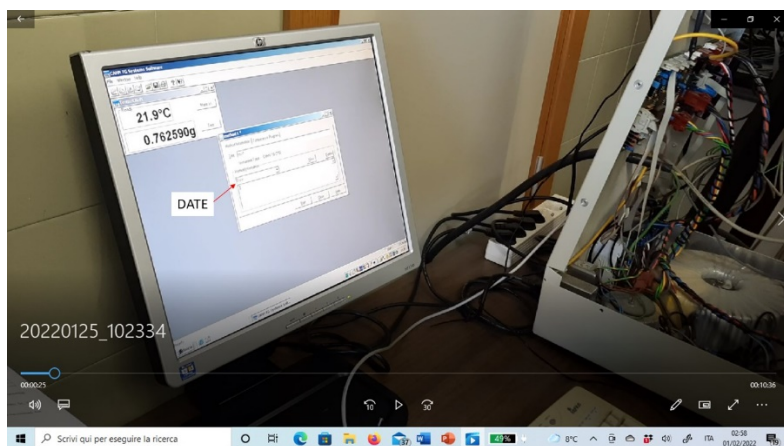


Figure 20: Insert the date

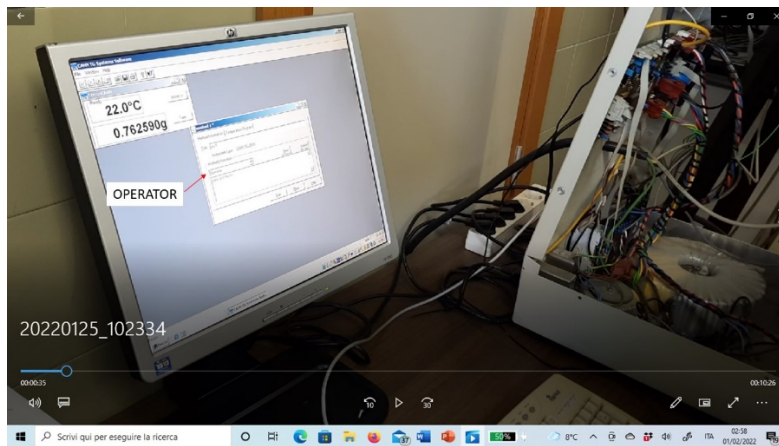


Figure 21: Insert the operator

Then the next step is to insert the temperature program. First we have to choose if it is dynamic or isothermal. We chose in the first step the option “isothermal” and we set the following parameters (see figure 21):

FIRST IS ISOTHERMAL -> 30 DEGREES FOR 2 MINUTES,

SAMPLE PERIOD = 1 SECOND

AC POWER LINE ON

COOLING FAN ON (Always control it is working)

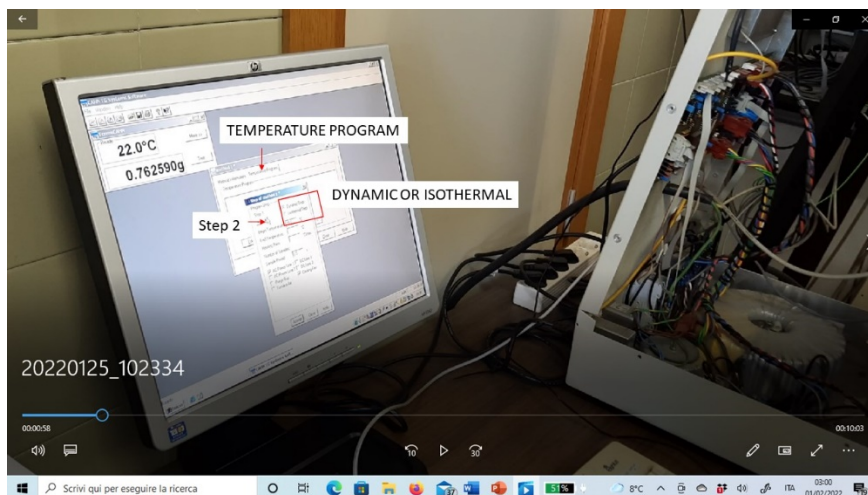


Figure 22: Setting the temperature program

Then we can switch to the second step with the command highlighted in figure 16. The second step will be in this case dynamic heating for this reason we have to choose not only the beginning temperature but also the final and the heating rate. So, beginning temperature is 30°C, final temperature will be 600°C and heating rate will be 20°C. We chose also to use power and cooling, see figure 23.

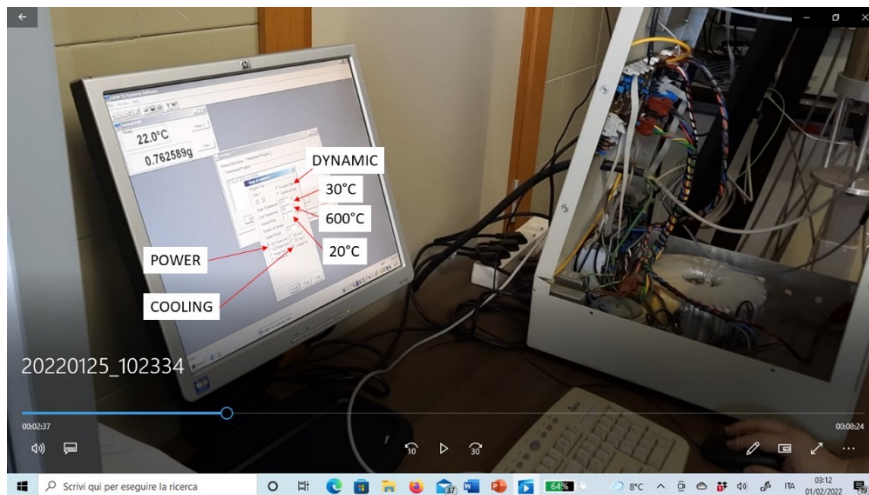


Figure 23: Dynamic heating

Then we chose another isothermal step which we chose to maintain constant for 5 minutes.

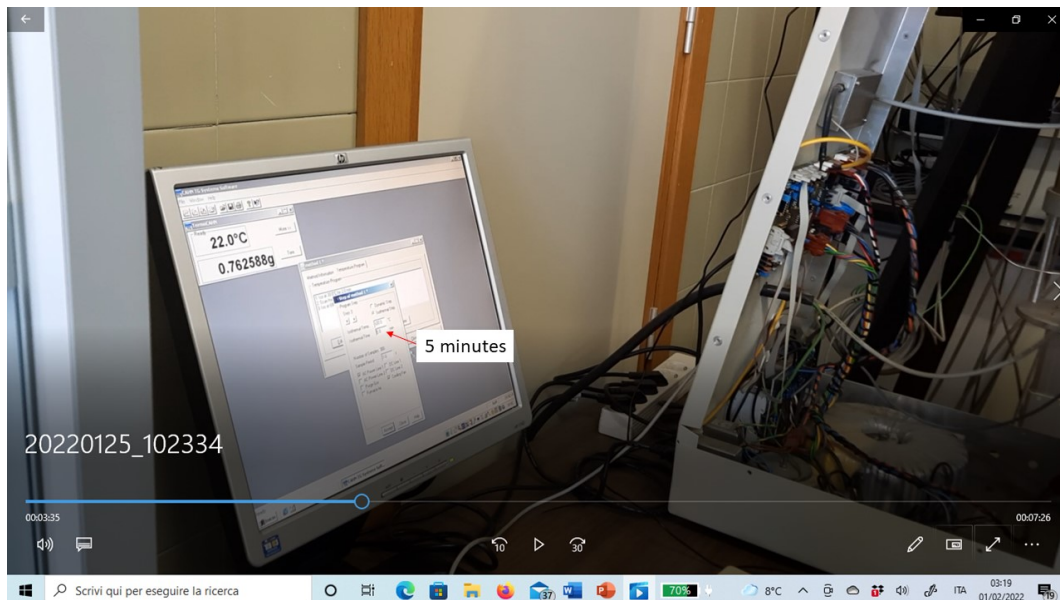


Figure 24 Isothermal step

Then we make another dynamic to reach 900°C with heating rate of 20°/min. And then we make isothermal at 900°C and we insert 6 hours we can cut then whenever we want. It is not decided the cooling step. The cooling step is only when I stop the power. Once defined the method is accepted. Once accepted we can see it and it can be still edited according to our preferences. Once the method is ready the user has only to press start, obviously.



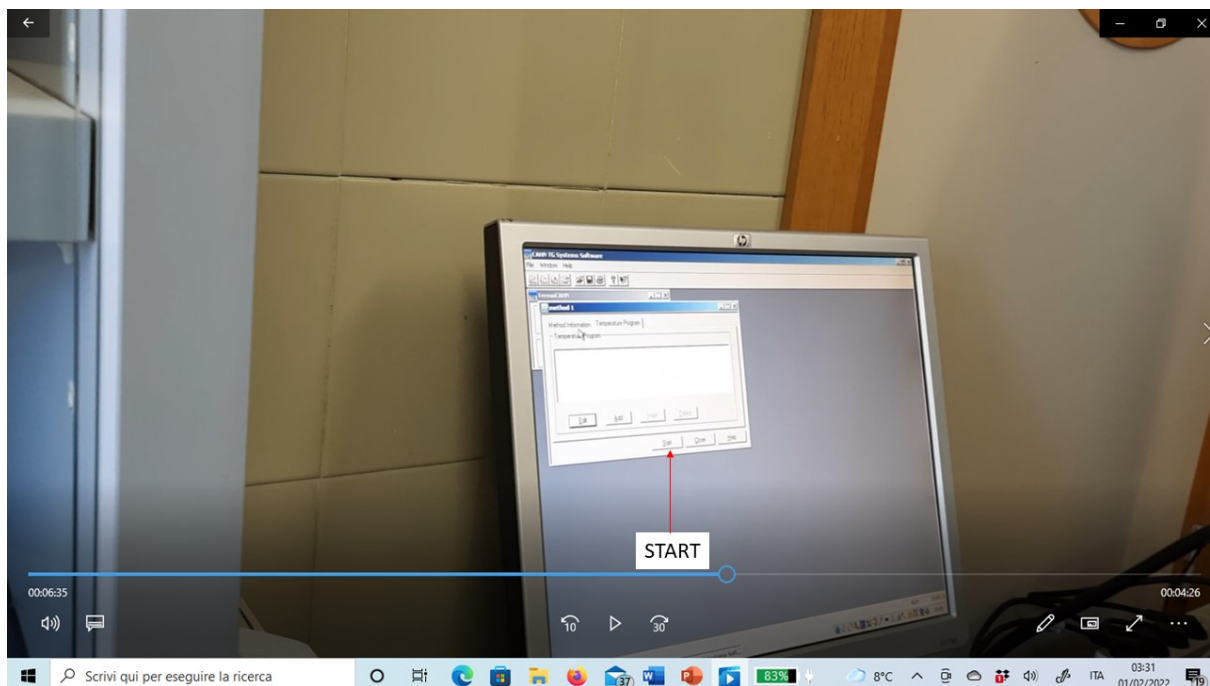


Figure 25: Method -> START

In alternative an already existing method can also be opened, see figure 26.

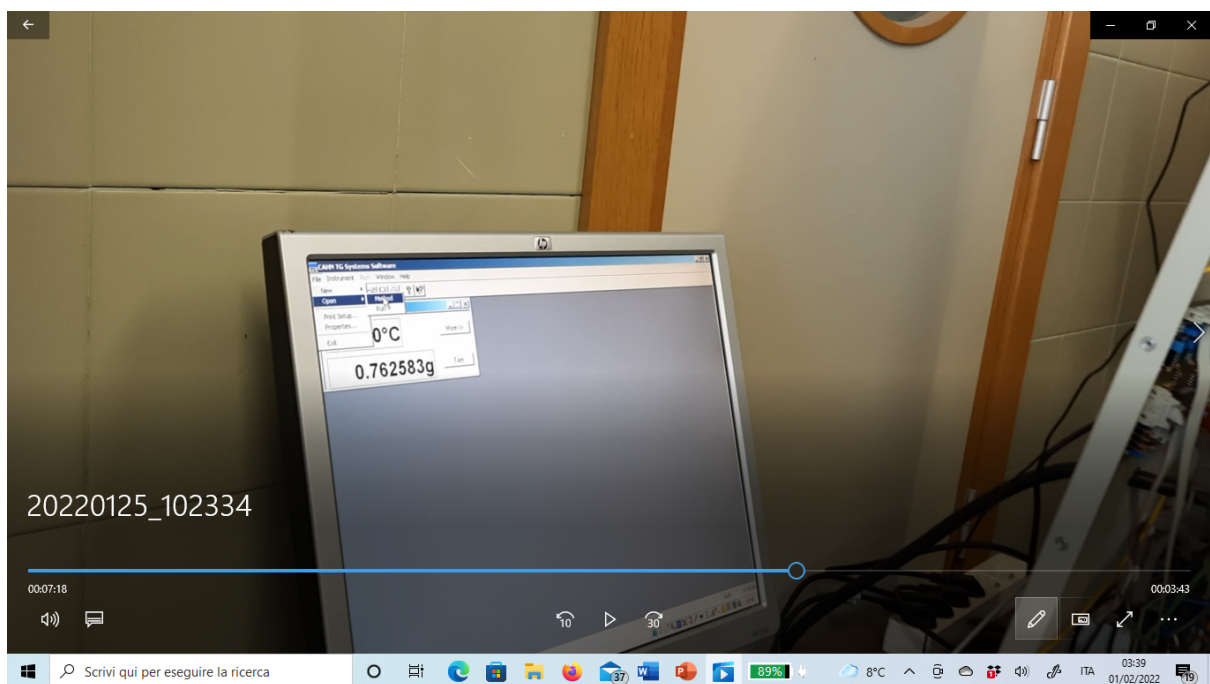


Figure 26: Method -> OPEN

The method files have usually the extension "mtp", see figure 27.

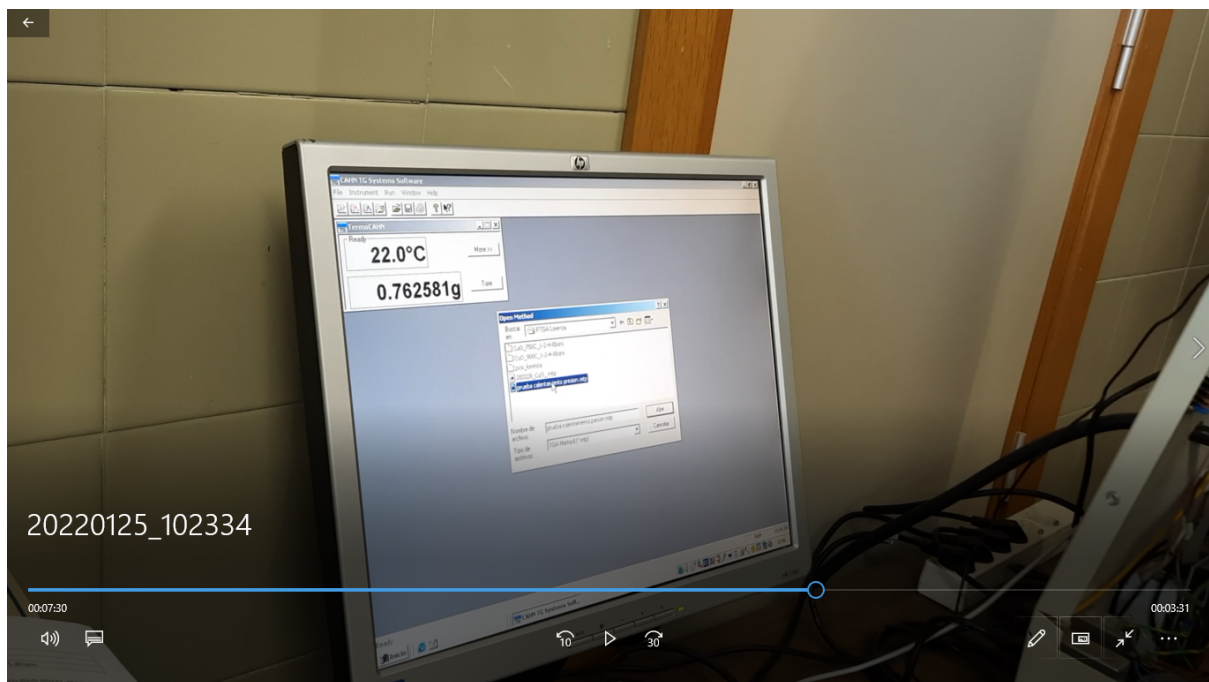


Figure 27: How to open the method files

Finally to see the results you have to open a run the method, with the command “run”, see figure 28.

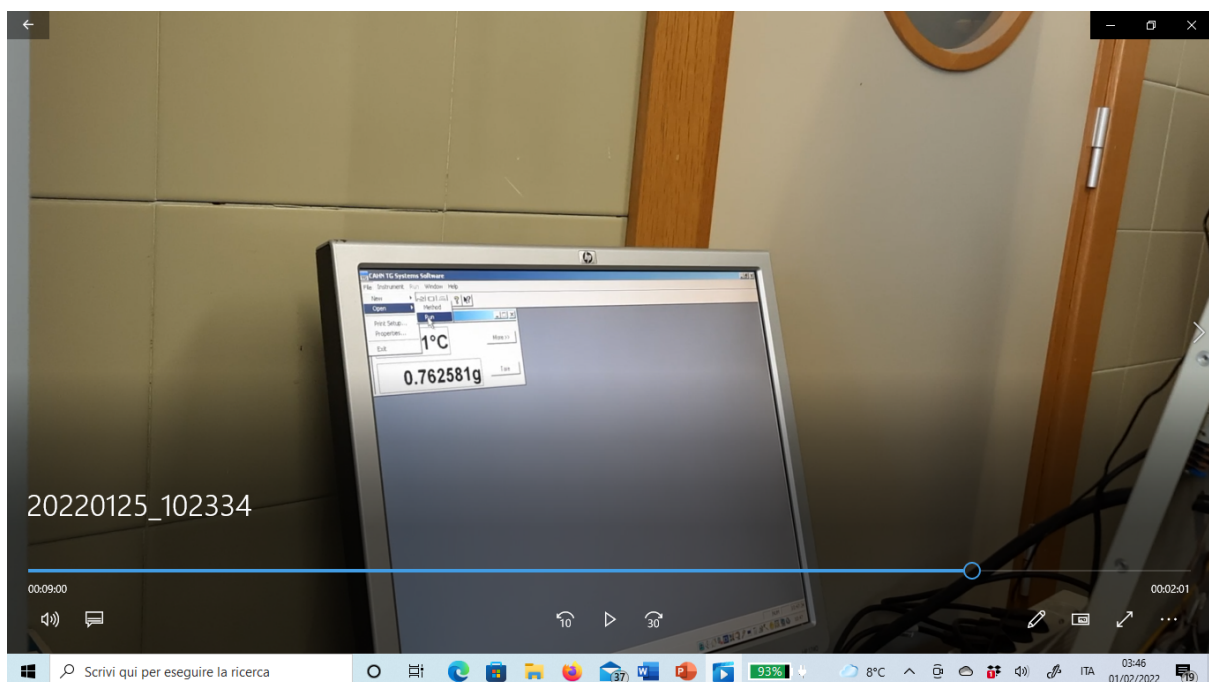


Figure 28: Method -> RUN

The results are contained in files with the extension “ctd” and can be opened also with the software named “winTGA”, see figure 16.

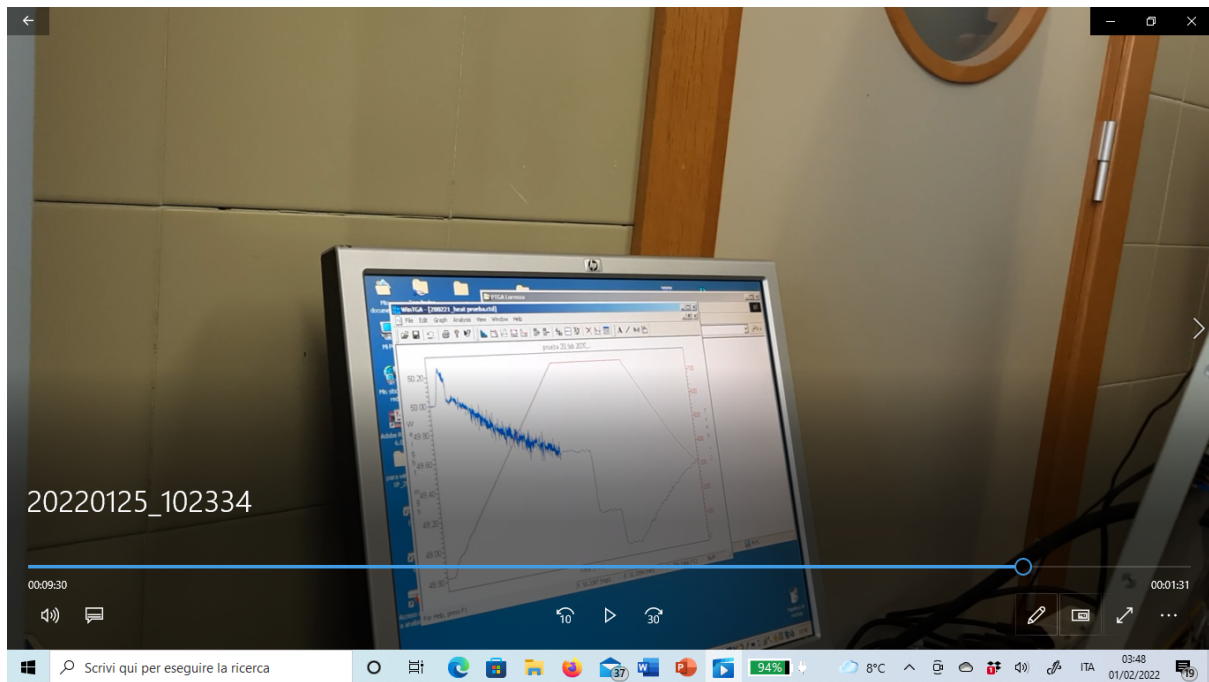


Figure 29: OpenTGA software.

From winTGA you can export to “.csv” and modify the data for using the raw data in calculations. See the export command in figure 30.

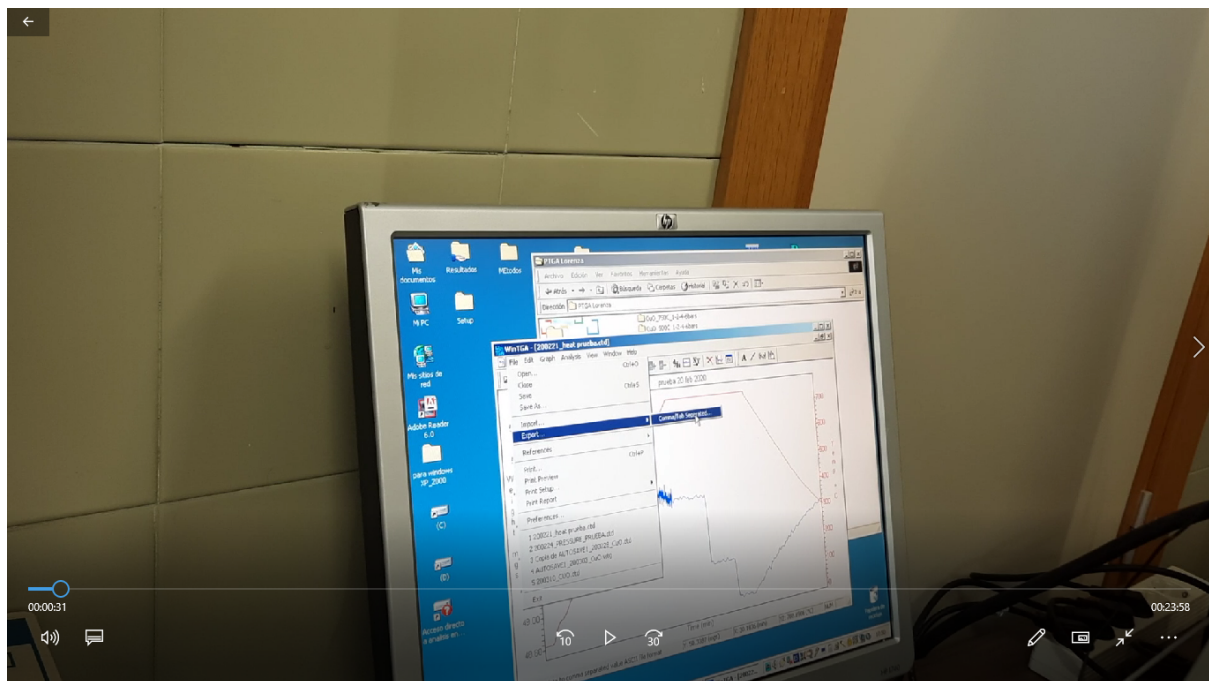


Figure 30: How to export data from WINGTGA

## 10. Controlling the pour gases

To control the gases 2 systems are adopted. The first one is manual and it is shown in figure 31.





Figure 32: Gas control manual system

In the system shown in figure 33, we have basically 4 gases:

1. is nitrogen -> pour gas for the bottom of the balance
2. is air for oxidation
3. is nitrogen -> furnace TGA
4. is the one which regulates the pressure

In the manual system we can first select the channel. Once you select the channel you can change the set point using the arrows. Arrow up is for increase and arrow down is to decrease. To modify the set point (which is expressed in percentage) you need to know the range of the mass flowmeter. To know this it can be useful to consider the schemes which are shown in figures 33, 34 and 35.

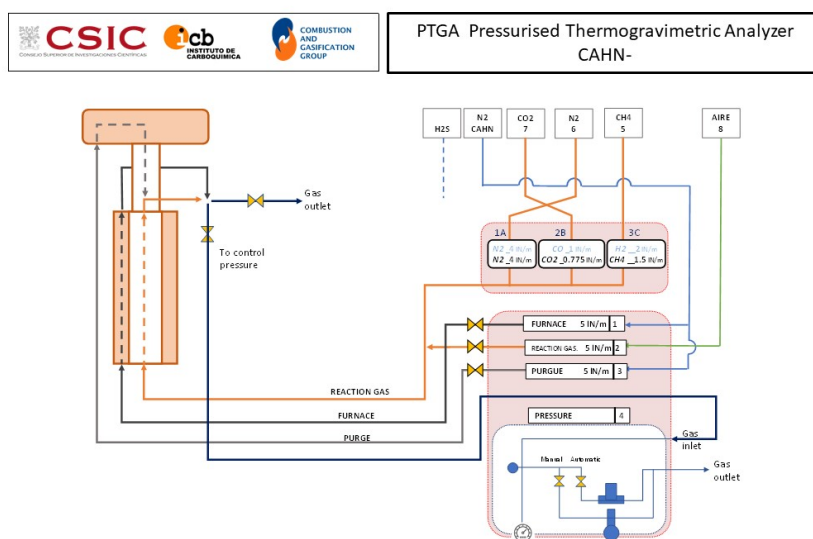


Figure 33: PTGA first scheme

[illegible]

Figure 34 says for example which are the nominal flows of the flowmeters. For nitrogen we have for example 5 normal liters per minute. So if the flowmeter is open 20% the flow will be 1 NL/min. Nitrogen at position 3 for the furnace HAS TO BE ALWAYS OPEN. Normally it is used 20% for the pour gases. So it should be 20% for the pour gas and 20% for the furnace gas.

WBC	7	8	5
G20.0mm	N1	C0	A2
C0.02mm	C02	A1	C03

For reaction gases we can consider:

- The reaction gases are controlled with the pc.

## 11. Controlling the reaction gases

The reaction gases are controlled with a LabView software, see figure 36.

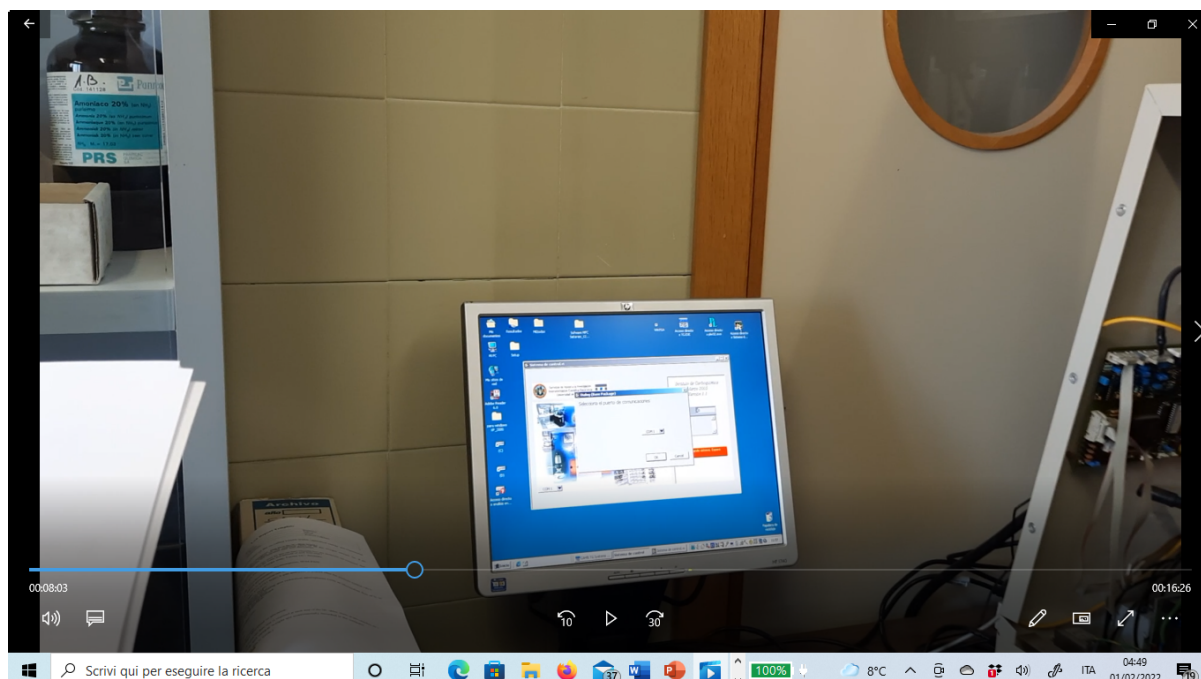


Figure 36: LabView software

The first thing to chose when connecting to the software is: COMM2. This has to be remembered because otherwise the data won't be visible. If the procedure is done correctly what will be shown is the screenshot displayed in figure 37.



Figure 37: Labview software screenshot



From the screen shown in figure 37 we can control the flow of the three reaction gases: CO<sub>2</sub>, N<sub>2</sub> and CH<sub>4</sub>, see also figure 38.

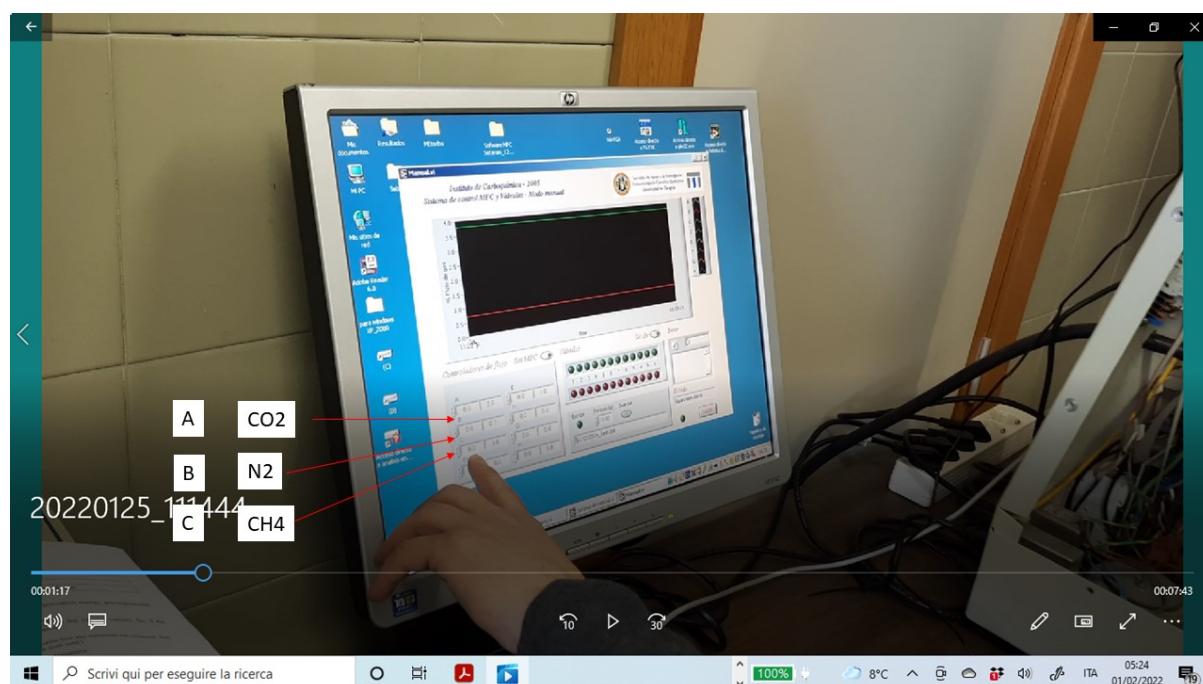


Figure 38: How to control reaction gases flow

CO<sub>2</sub> is A and it is number 7 on the other controller

N<sub>2</sub> is B and it is number 6 on the other controller

CH<sub>4</sub> is C and it is number 5 in the other controller

We have to write there only the percentage of the mass flow controller to open it at to have the desired flow. To close it I set it to zero. Air is regulated manually. While reduction gases are regulated through LabView. To set reaction gases we have to set:

- Total flow
- Composition.

If we have to pass from one cycle to another. We can switch off the flow of CH<sub>4</sub> and CO<sub>2</sub> and continue with nitrogen, we can increase the flow of nitrogen to compensate the decrease of pressure!! Also during oxidation nitrogen can be used.

## 12. Monitoring pressure

The monitoring of the pressure is done with the software PicoLog, see figure.

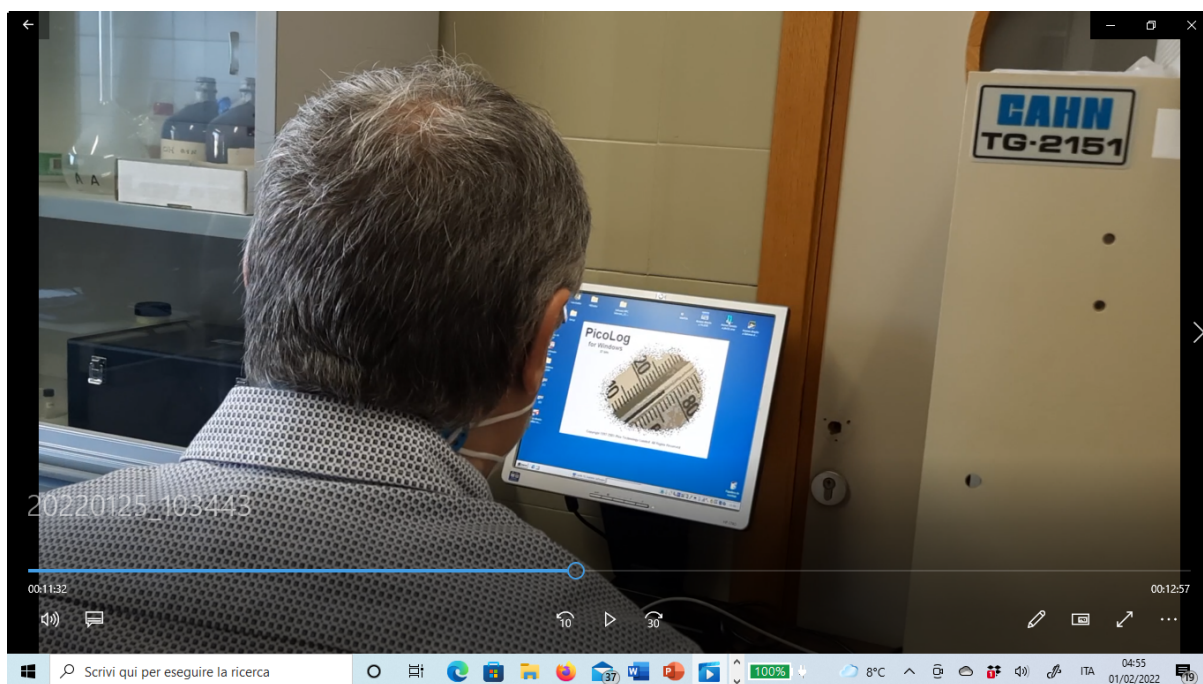


Figure 39: PicoLog software

For the control of the pressure is very important the equation reported in figure 40.

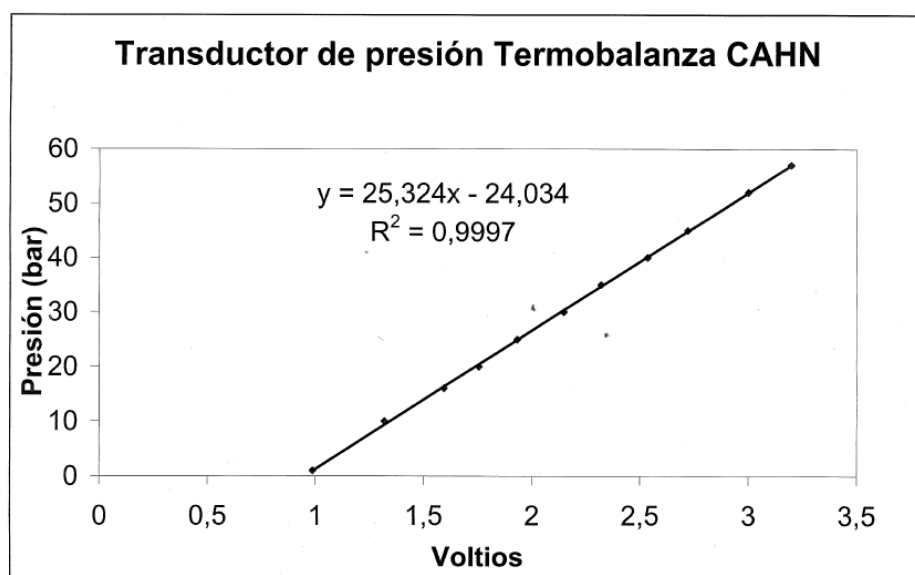


Figure 40: Regression line used for the conversion of Volts into pressure

Pressure can be also recorded but it is enough to see it on the screen. The whole procedure to set up the pressure is described in a doc file which can be found on the desktop, see figure 41

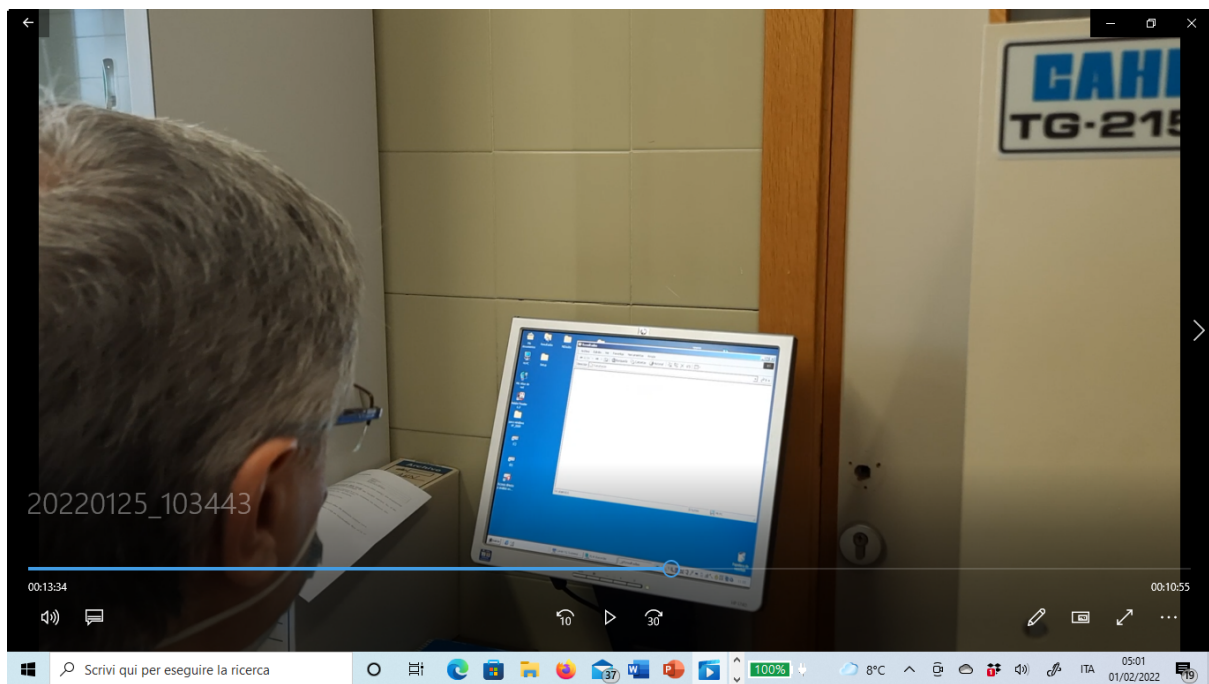


Figure 41: where to find the doc file describing how to set the pressure parameter

A screen shot of the word file is proposed in figure 42.

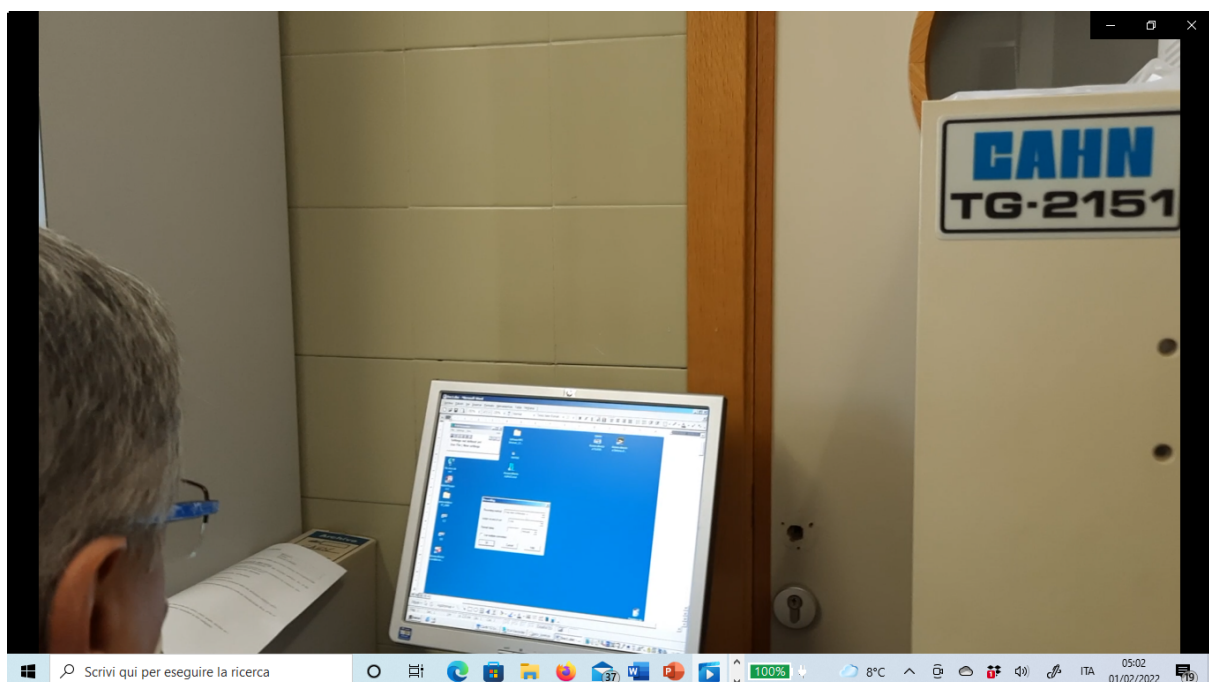


Figure 42: Screen shot of the procedure prepared by Lorenza

The steps are:

- Chose settings, chose Real time continues, chose stop and chose in 1 minute;



- Set the sampling rate: 1 second is ok
- You can define the maximum number to record but it is not important because we are not going to record the pressure;
- Set ADC converter
- Set the port
- Define the maximum, the minimum
- Define scaling, then chose pressure and then insert the equation

Other way can be to chose an already define program, see figure. It has just to be pushed the command: open.

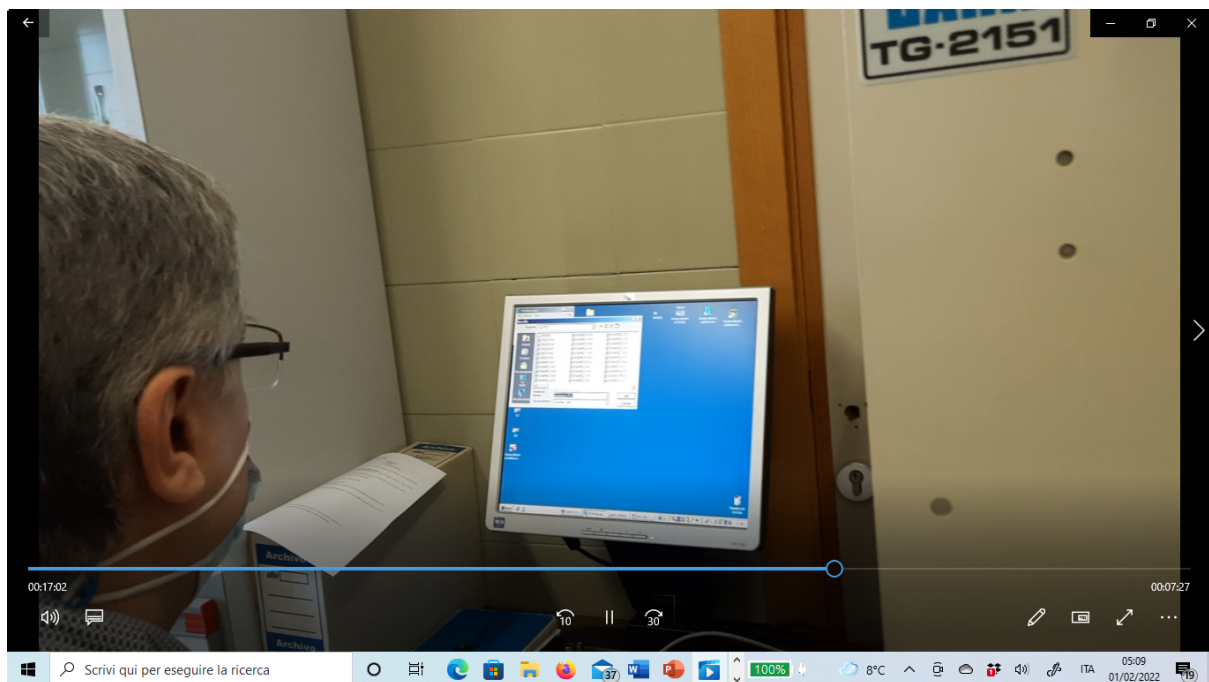


Figure 43: Open Pressure monitoring software

The final diagram that we expect to see is shown in figure 44.

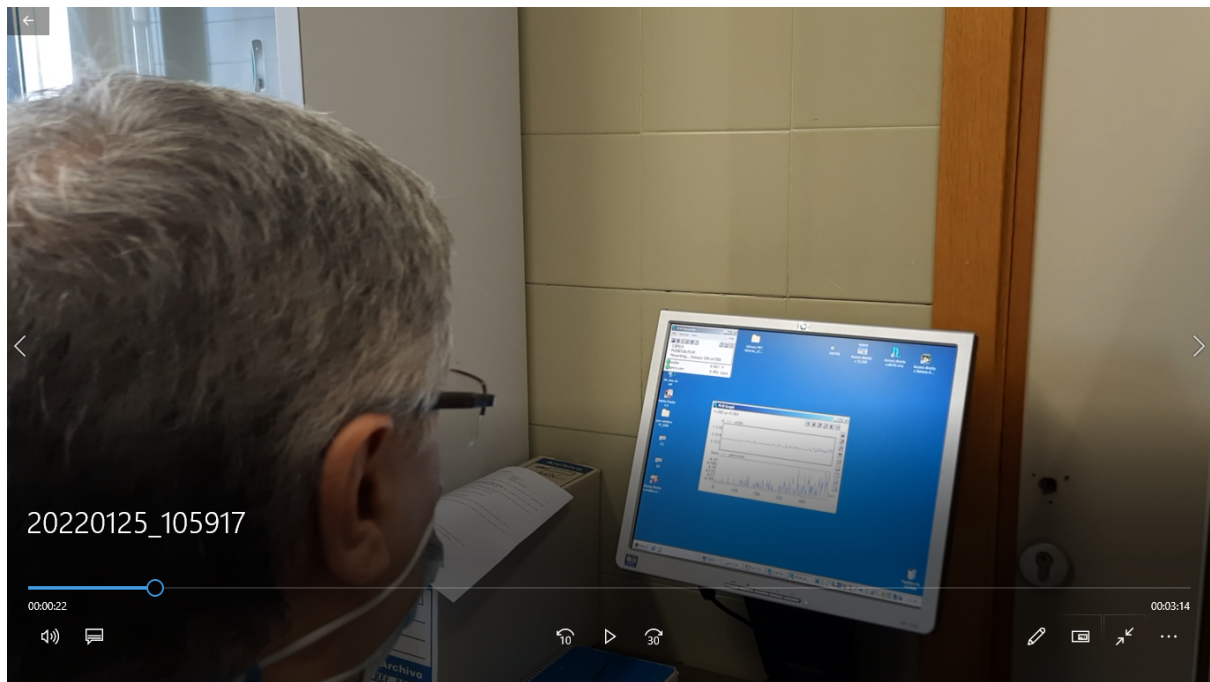


Figure 44: final diagram

## APPENDIX

