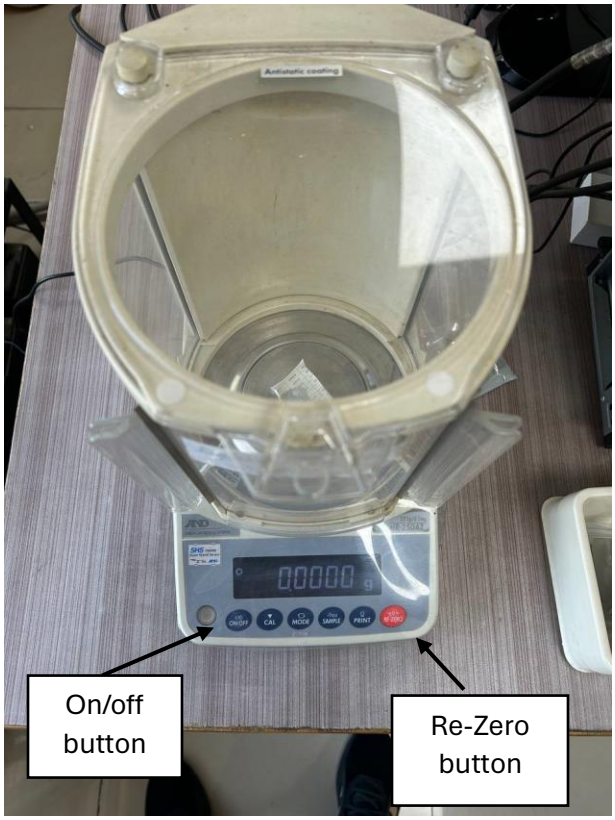
TPR analysis

Major Step One: Degassing

Step 1: **Start the machine.**

Note: while measuring your sample, turn on the digital balance main switch first, then press the **on/off button** on the beam balance and press the **Re-Zero button** to make the reading 0.000g.

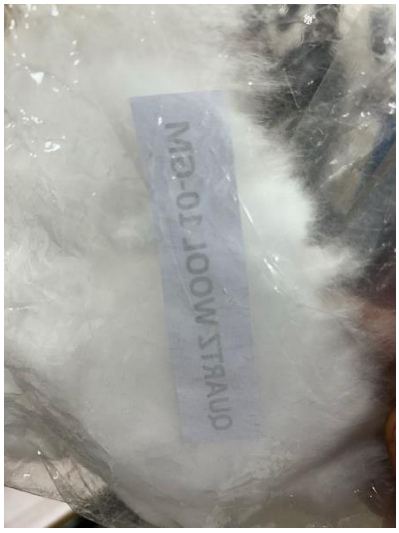


Step 2: **Measure your sample in the digital balance and note down the amount.**

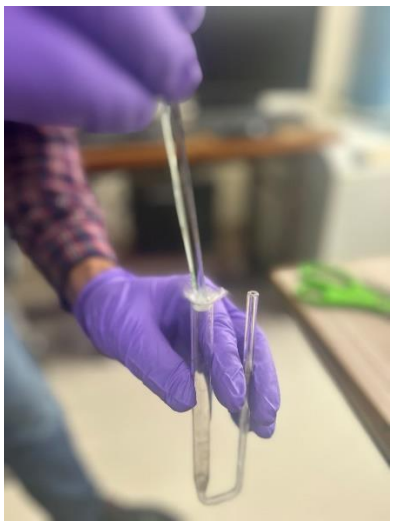
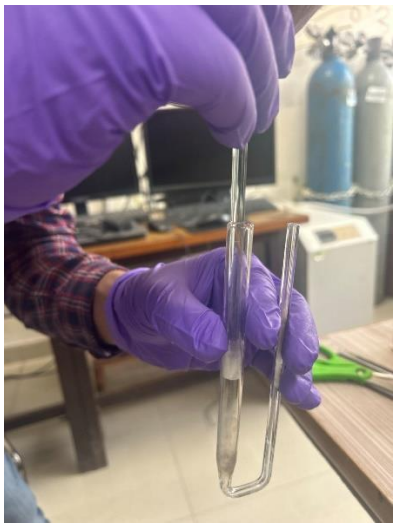
Note:

- The amount to measure is between 30-40mg.
- Whenever taking a sample take the sample from the corner of your plastic container or any place in the sample container with very fine powdered particles.

Step 3: **Put an appropriate amount of Quartz wool in the U tube using a glass road.**





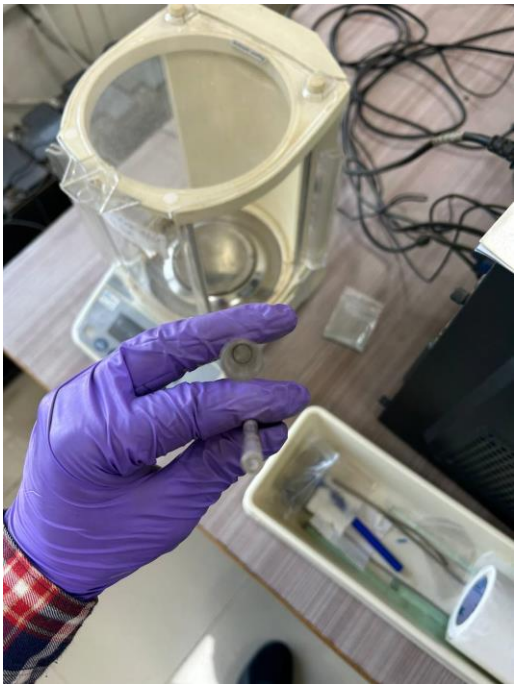

After putting the quartz wool in the bottom part using the glass road, use the metal road to gently push the cotton a little downward as shown in the picture.


Step 4: **By putting the glass tube in the circular carton, measure the mass and note down the reading.**

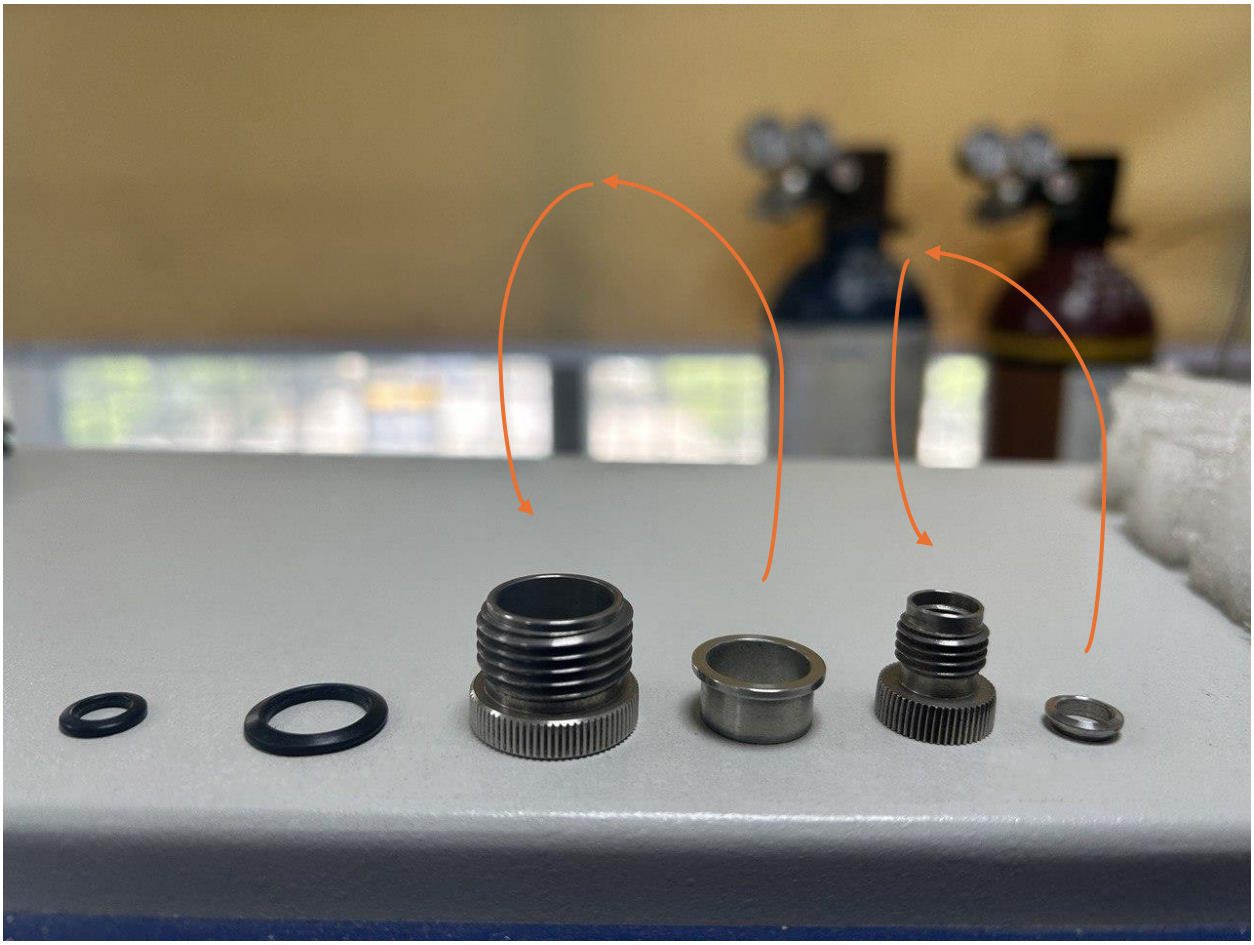
Step 5: **Put your measured catalyst inside the glass tube and note down the reading.**

Note:

- First, prepare a V-shaped paper to put the catalyst in the U-glass.
- Put the catalyst in the V-shaped paper.
- Arrange the U tube in the horizontal direction, put the V-shaped paper with the catalyst inside and slowly move it vertically in a way the catalyst will directly be on the prepared quartz bed.
- Make sure the catalyst is uniformly distributed. if not very slowly shake from the bottom of the U-shaped glass.
- When measuring make sure to close both glass doors of the weighing machine.
- If pc is running open the glass door in the other side to avoid Pc fan disturbance on the weighing balance.
- After measuring kind of crosscheck if the mass of the sample measured and the difference between the glass only and the glass with the catalyst difference are somewhat the same, if not take the smaller value which you find from either of the readings.



Step 6: **Take your U-shaped glass, hold it like it is shown in the picture gently, and put on the metal and rubber fittings carefully.**

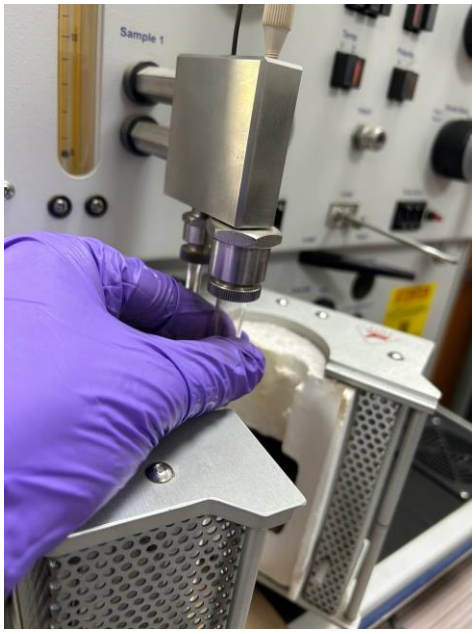


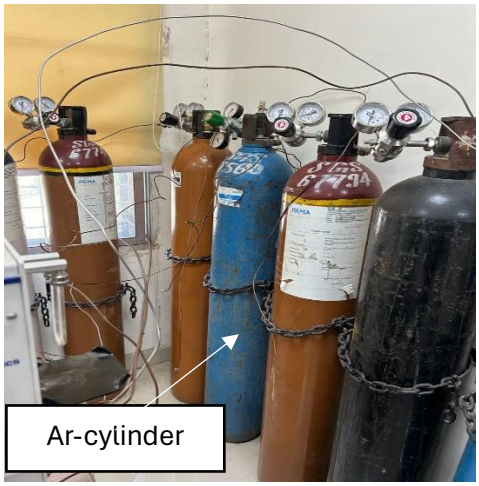
Then put the metal screws on the glass carefully by holding it like shown in the picture and put on the rubber accordingly and make it a little lower to avoid the probability of leaking.

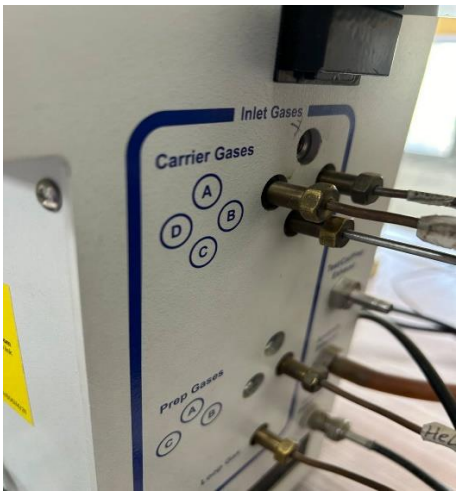
Step 7: **Put the Cardboard under the furnace before attaching the U-shaped glass to the machine.**

Step 8: **Put the glass road in the machine very gently by holding the U-shaped glass with your left hand and gently by supporting your hand on the furnace and tightening the screw nut on both glasses anticlockwise.**

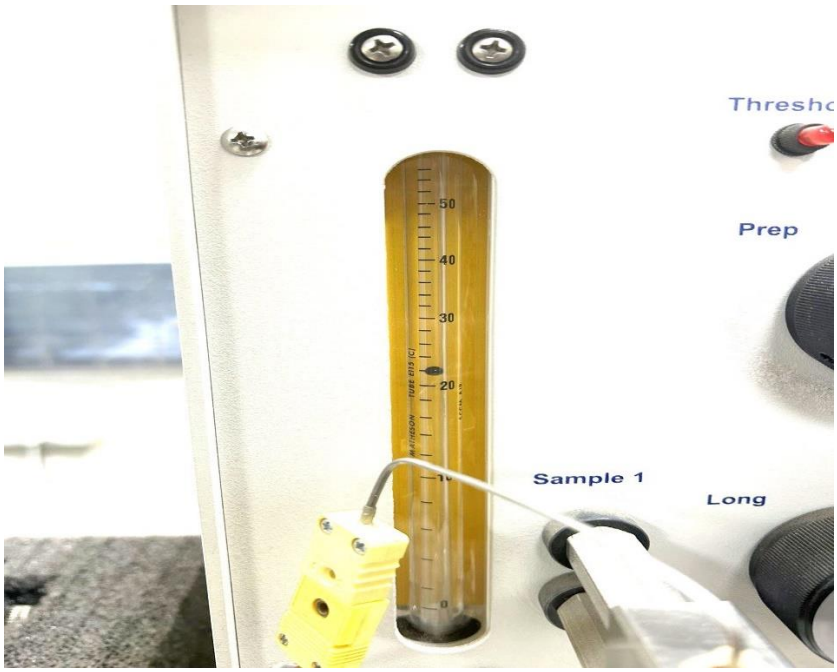
- While doing the rotation try to do it parallelly not one after the other.
- Open the Argon cylinder and press the respective button.


Sample 1


Ar-cylinder


Inlet Gases
Carrier Gases
A
B
C
D
Prep Gases

- Check if the flow is going on the flow meter, and it should show you 20 or slightly above. If it's not showing, that means a leak is there and you must open, properly attach the rubber rings, and tighten it again.



- If the rubber rings are stacked inside, use a metal spring metal road to tickle it and get it out.
  Link to video:
  https://drive.google.com/file/d/1kJjCfOiBOyZRTkff0Cuq_V3EedMMqXiz/view?usp=drive_link

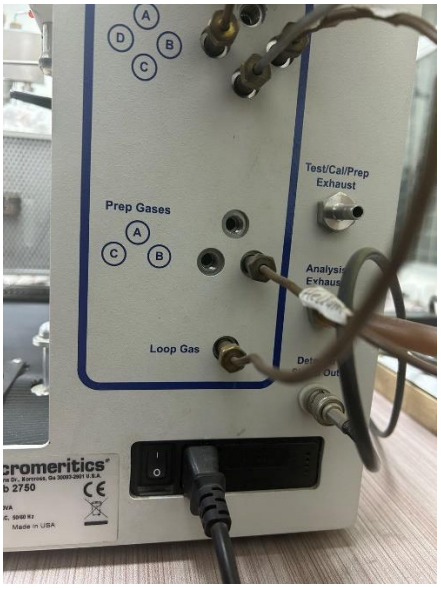
Step 9: **Put the thermocouple downward and clip it carefully.**

- First put the metal bar from the bottom side of the glass and clip it with the thermocouple.



Step 10: Close the furnace and put the glass wool on top for insulation.

Step 11: **Turn on the UPS and turn on the TPR machine from the right side and the Eurotherm from the back button.**



UPS **TPR machine** **Eurotherm**

Step 12 : **Start the eurytherm and hold it when it reaches 200 ºC by pressing the run button which you use to run till you see the hold LED light come.**

Step 13: After it reaches 200 then we will wait for 2 h degassing time by putting alarm and when the time is up we will cool it by long pressing the run button which we use to run the temperature. In the meantime to speed up the cooling process we turn on the fan.

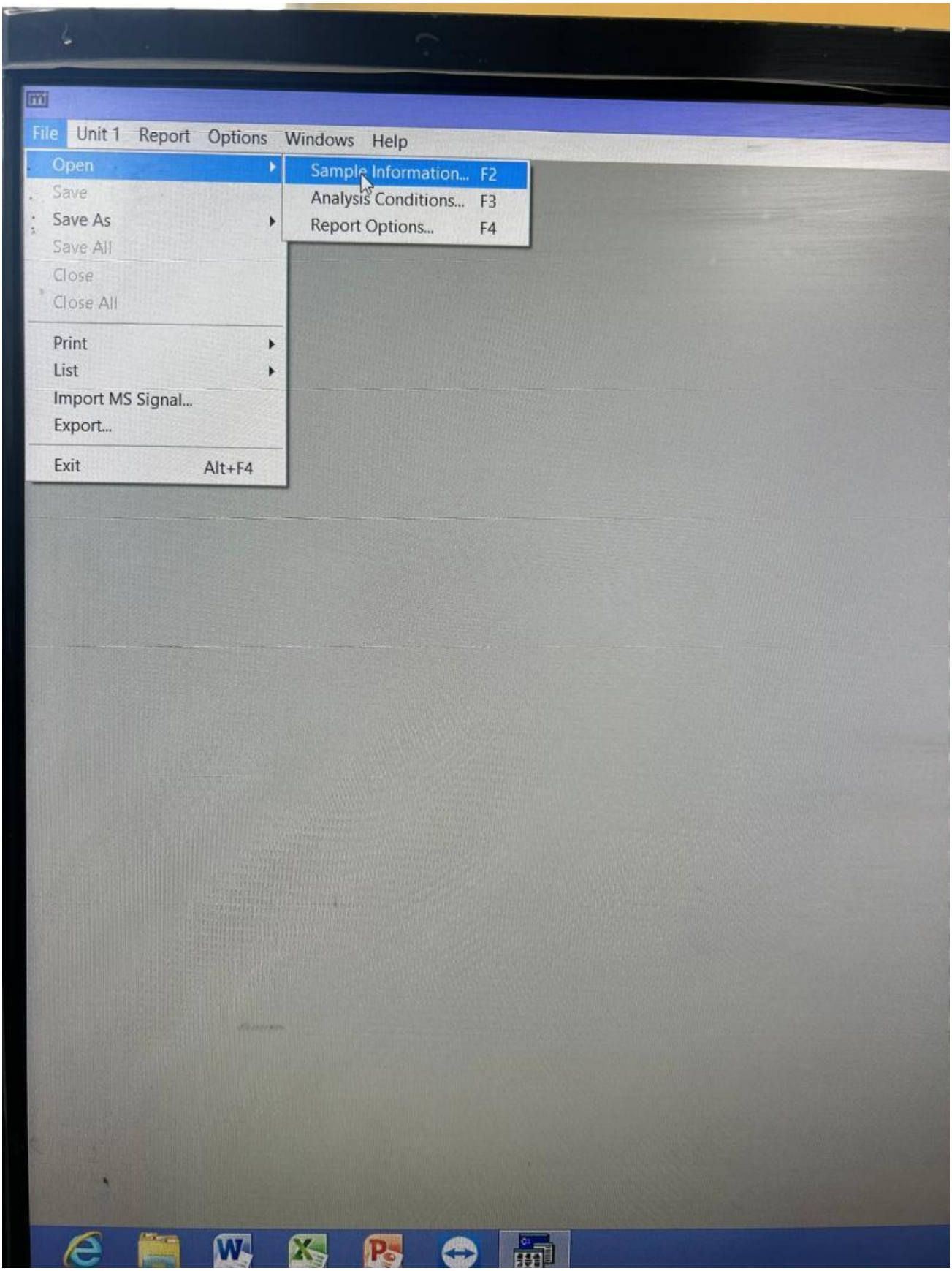
Note: Before turning on the fan make sure to remove the carton beneath the furnace and press the fan button accordingly.

After it colled (less than 35 ^{o}C) you are good to go for the analysis part

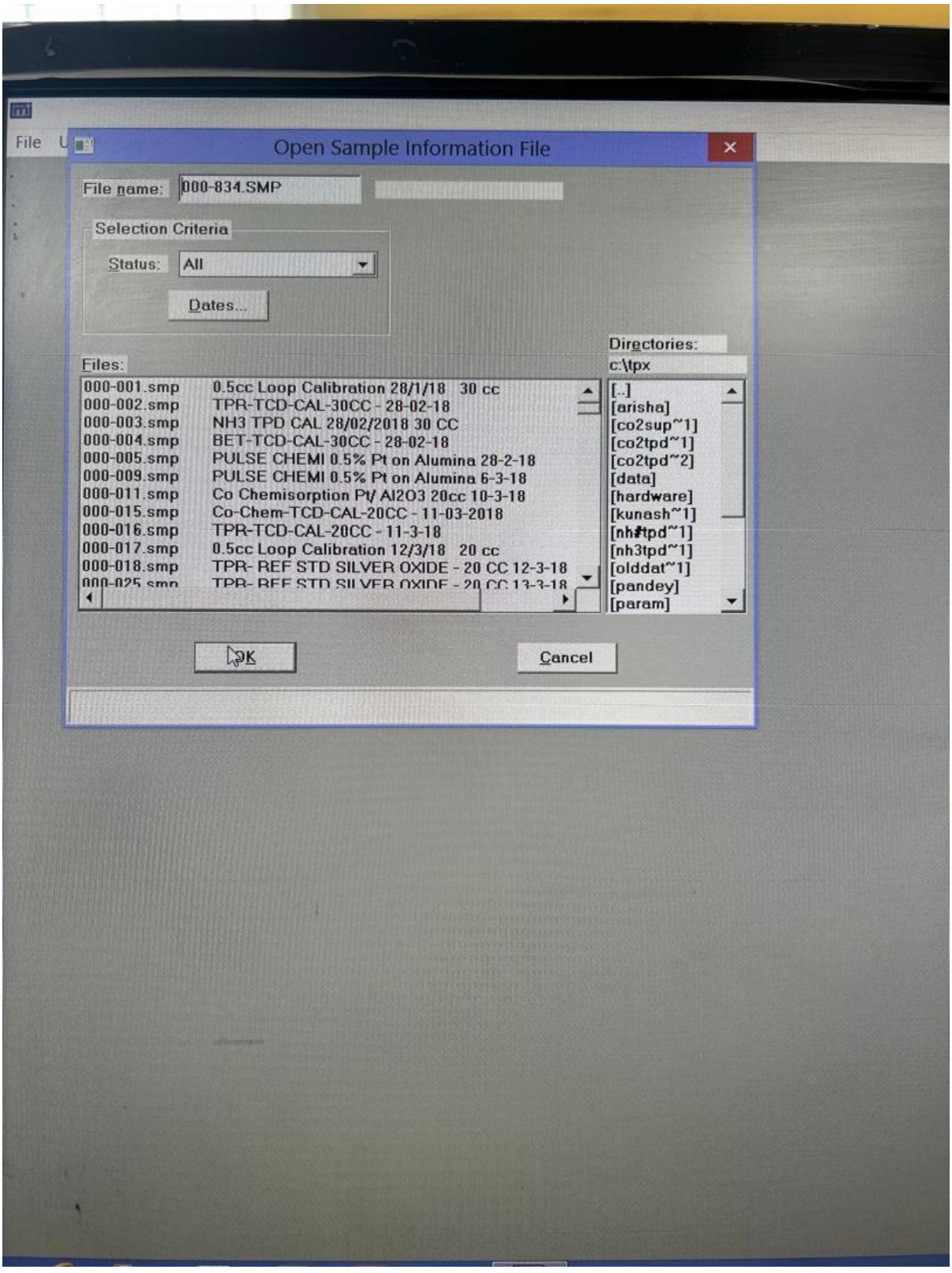
Major Step One: TPR-Analysis

Step 1: Open the software called Chemisoft from the screen

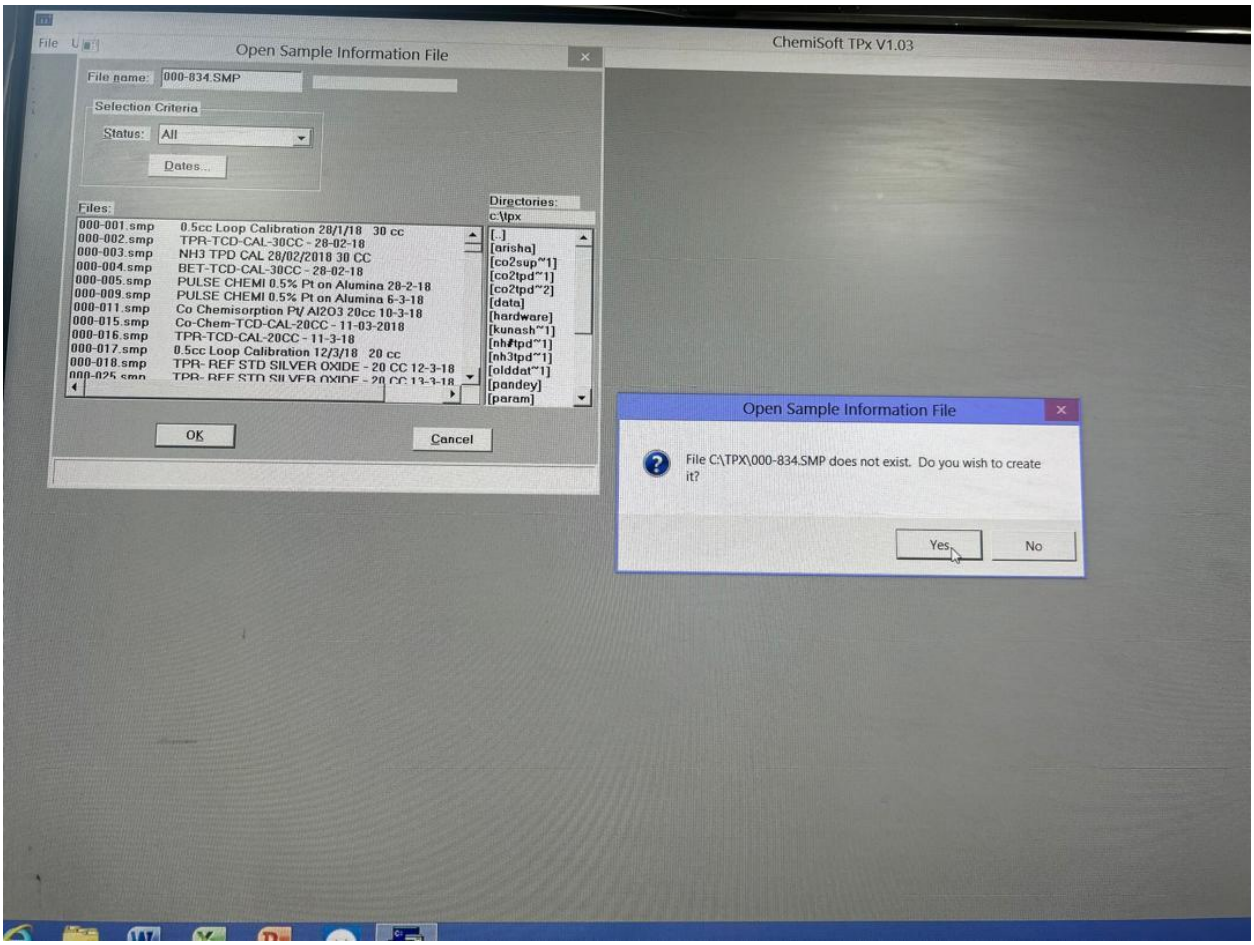
Step 2: Go to the file from the left corner → open → sample information


File
Unit 1
Report
Options
Windows
Help
Open
Save
Save As
Save All
Close
Close All
Print
List
Import MS Signal...
Export...
Exit
Alt+F4
Sample Information... F2
Analysis Conditions... F3
Report Options... F4

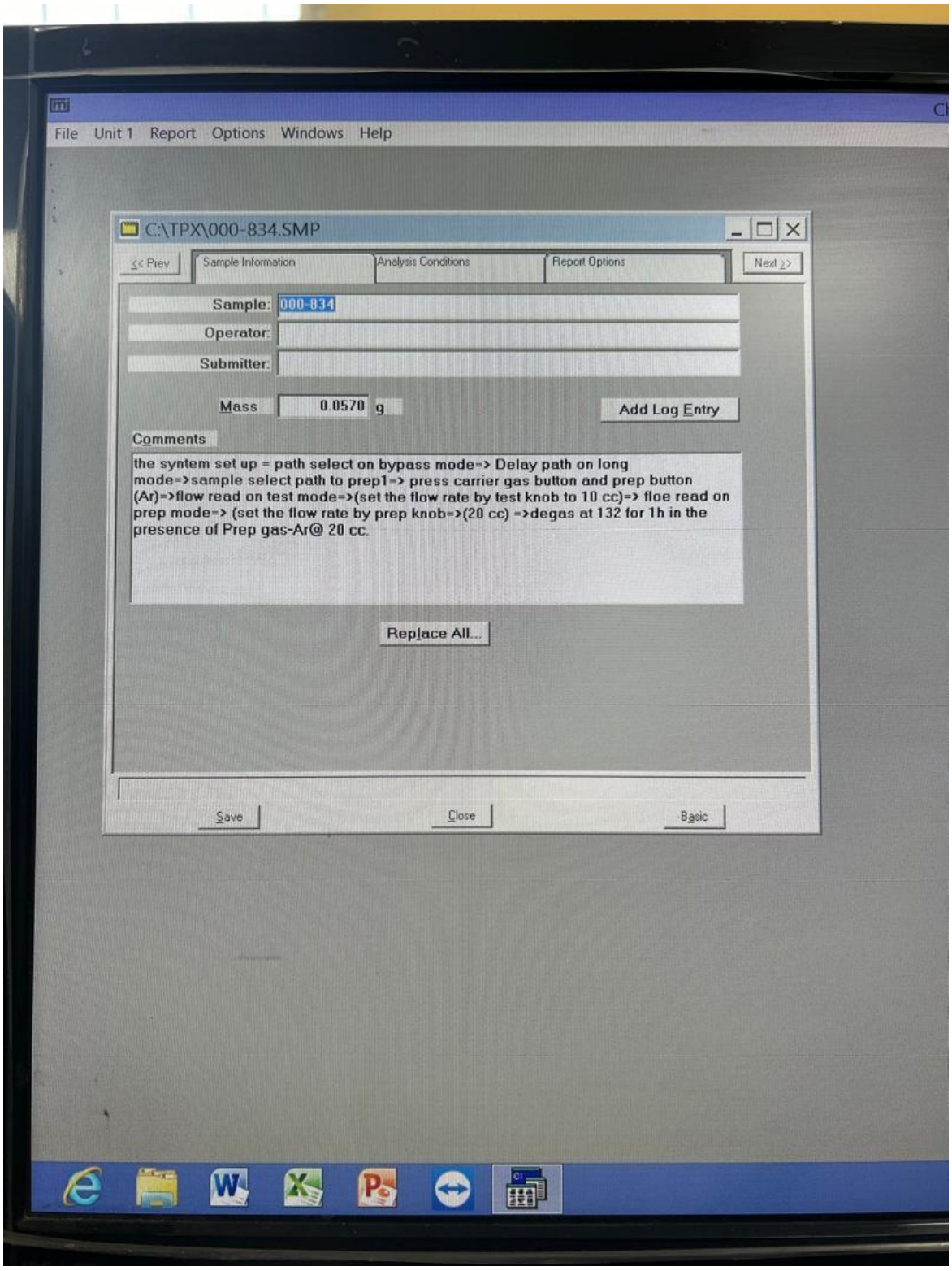
Step 3 : select nothing just click “ok:



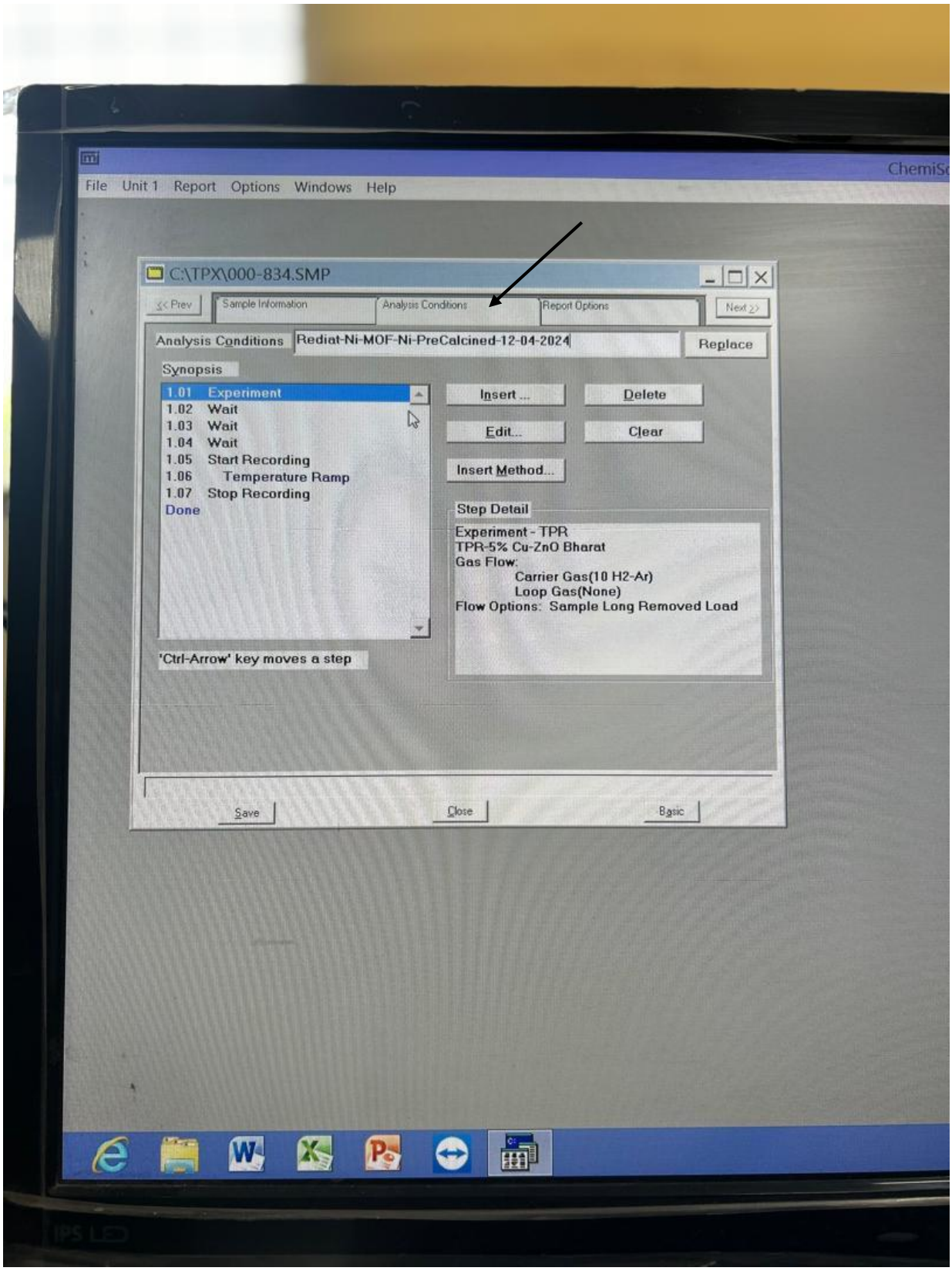
Step 4: A dialog box will come “ Do you wish to create it?” then click “ yes”



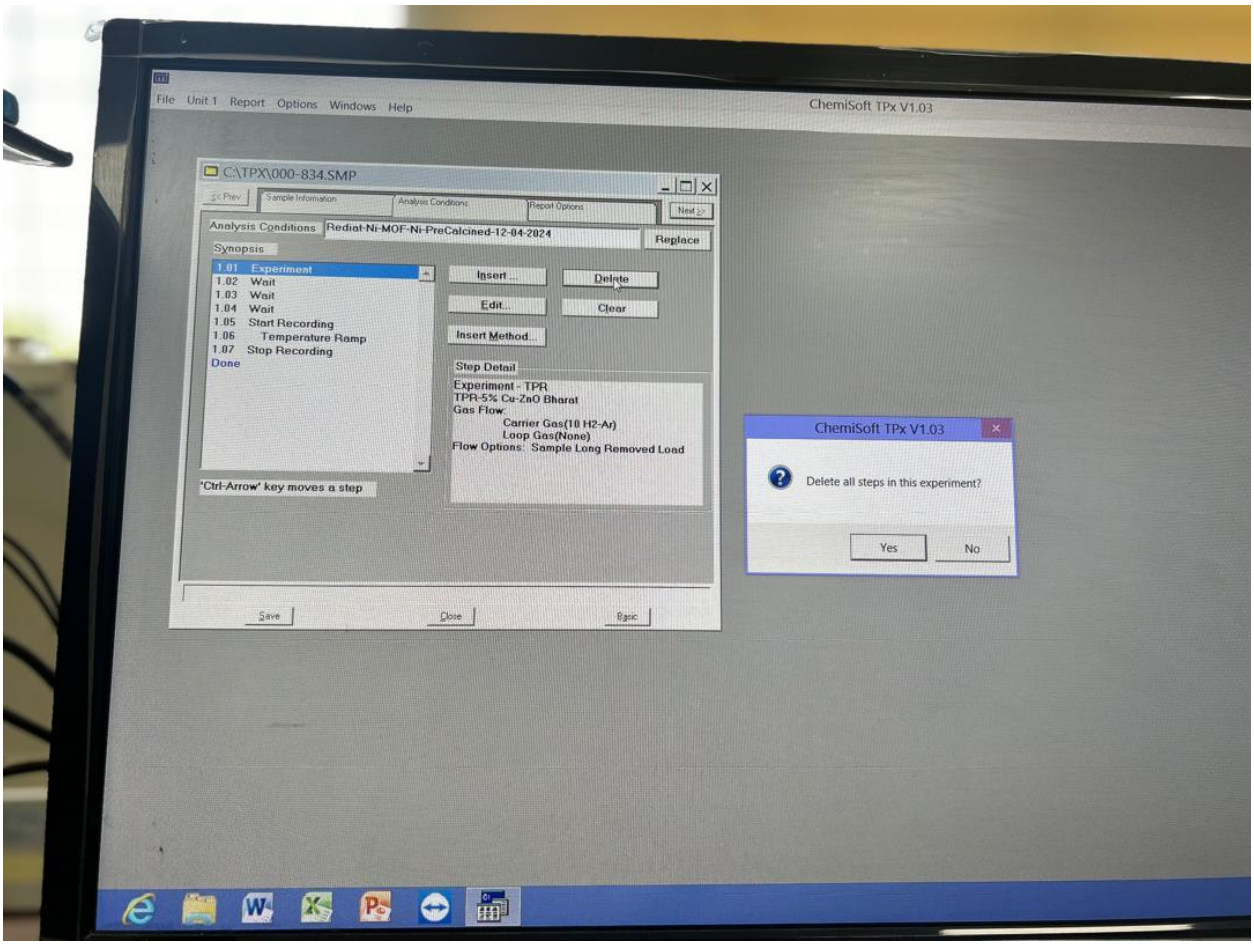
Step 5: Put your sample name in the sample input box copy it for further use and put the same info for the operator and submitter accordingly where the operator will be your name, and the submitter is IITR and put your sample mass.



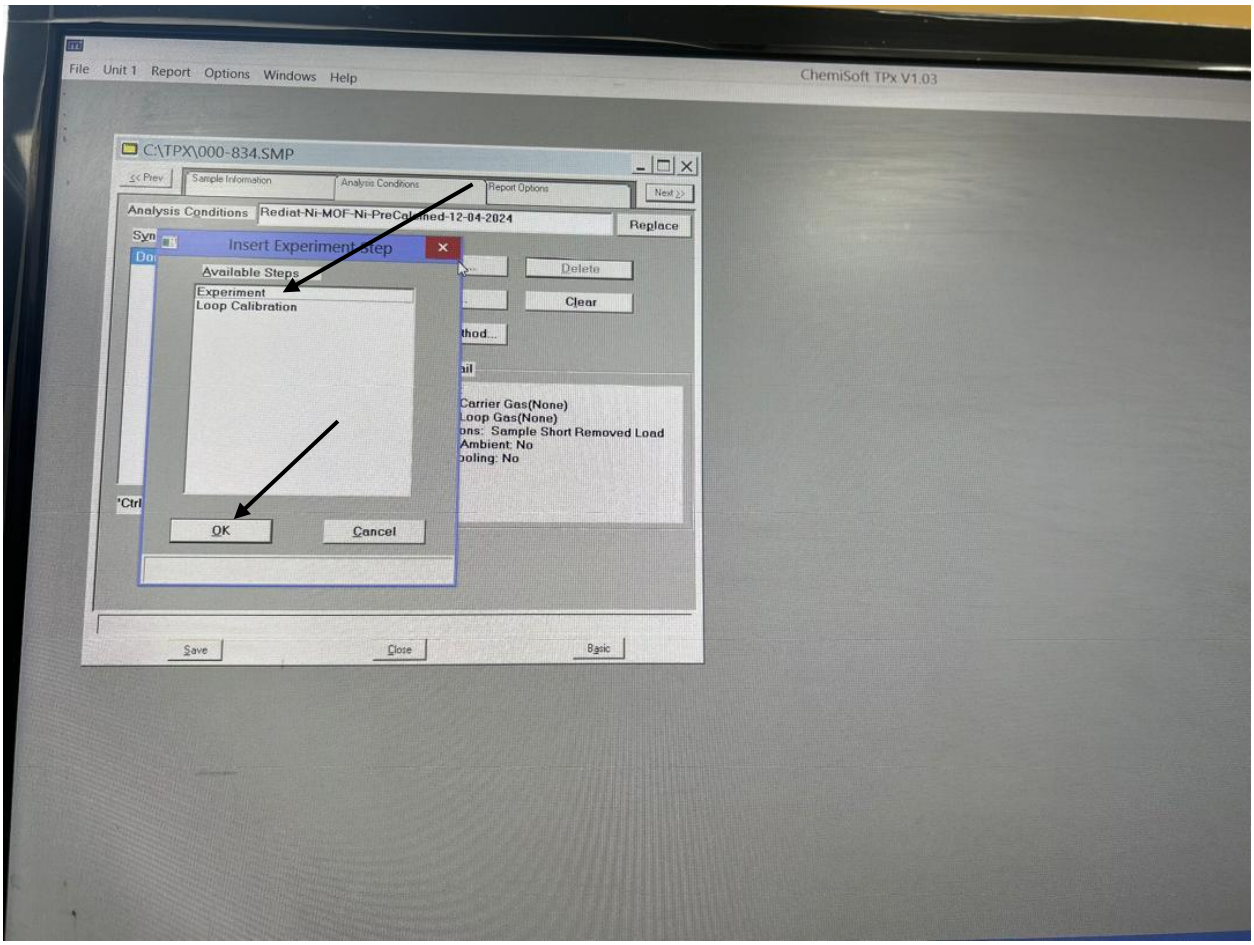
Step 6: Go to the Analysis condition and paste the name of the sample and click delete



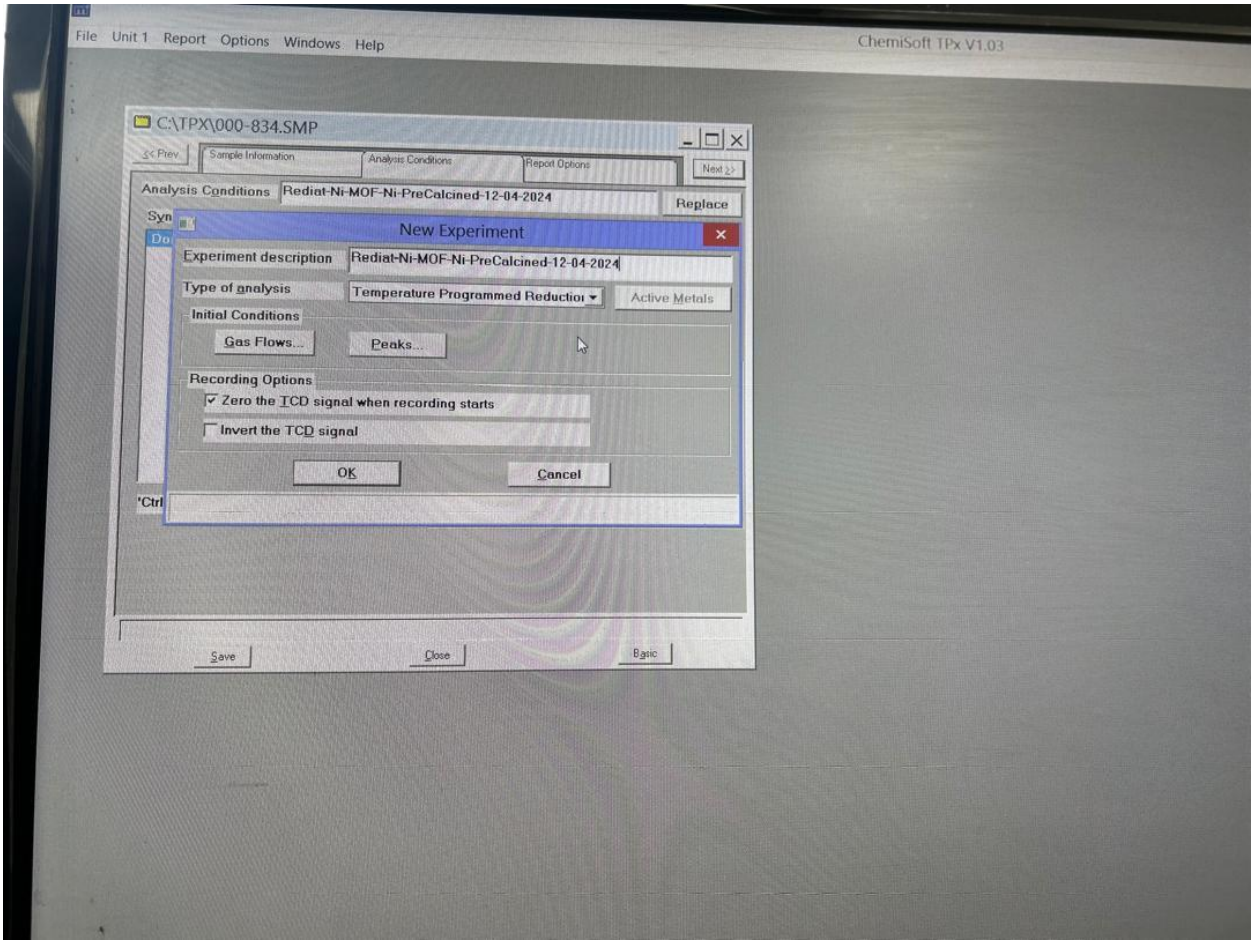
Step 7: Click on yes when the small dialog box for deleting comes which will clear the synopsis section.



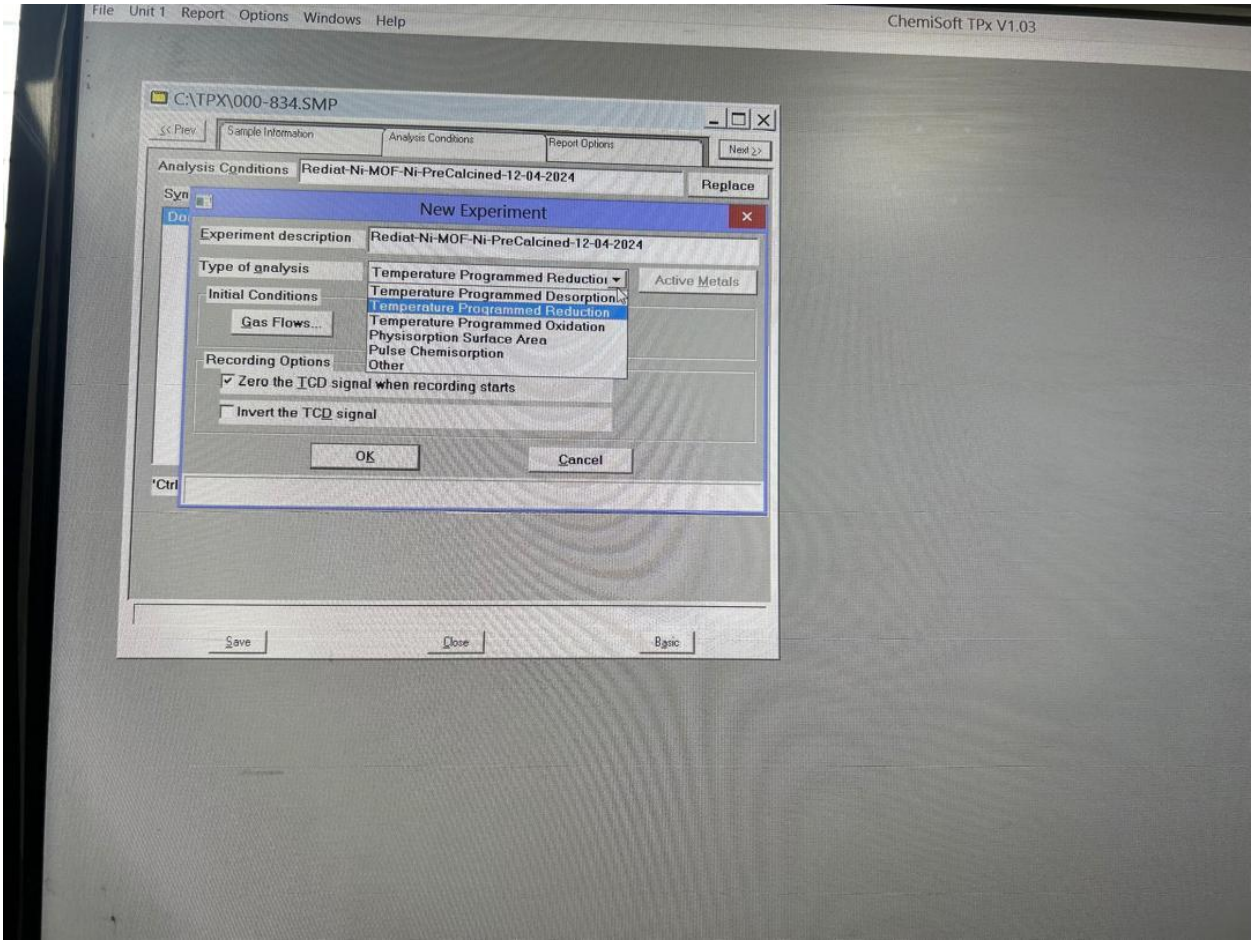
Step 8: Click on Insert Select Experiment and click ok



Step 9: Paste the name in the experiment description input tag



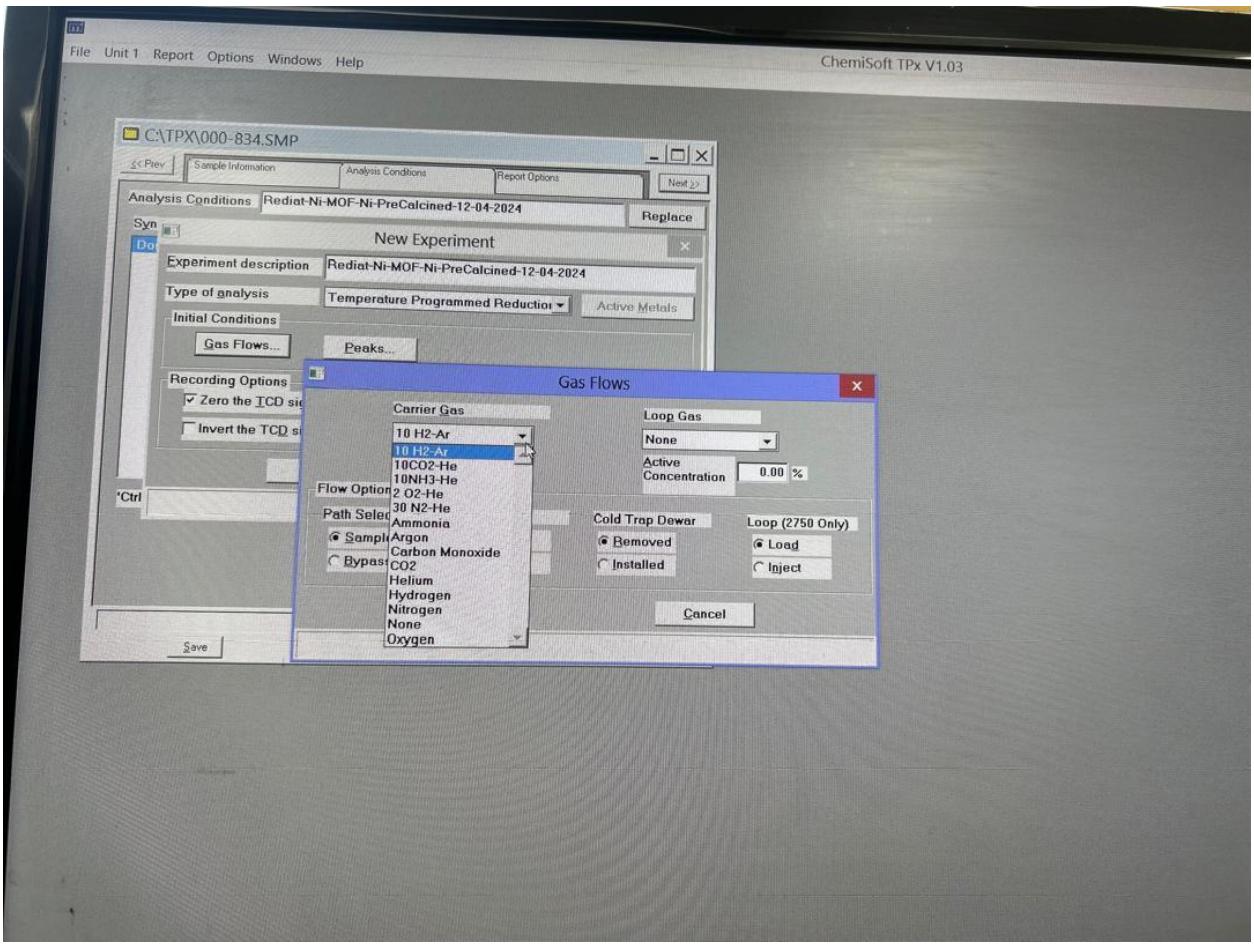
Step 10: Select the type of analysis to be TPR

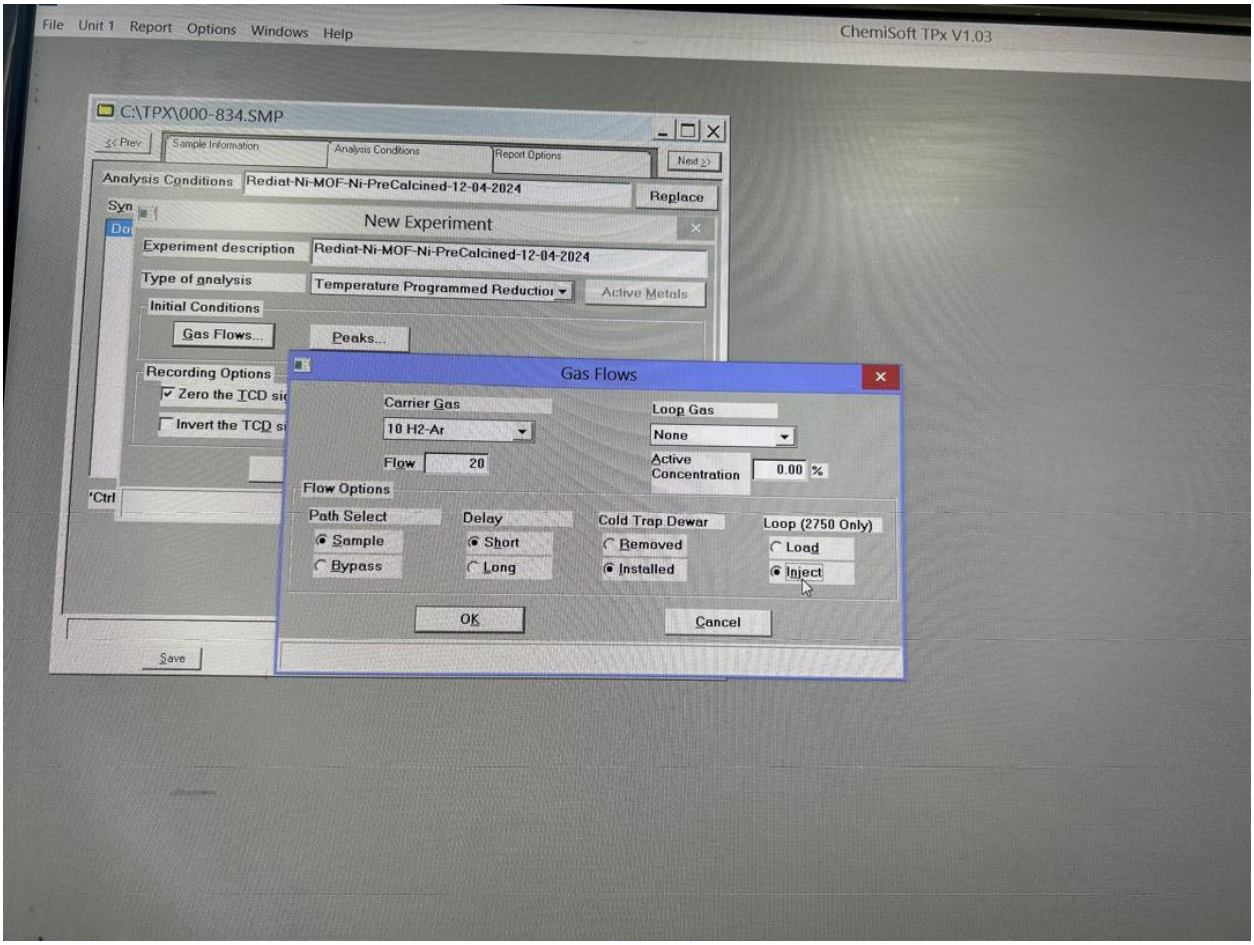


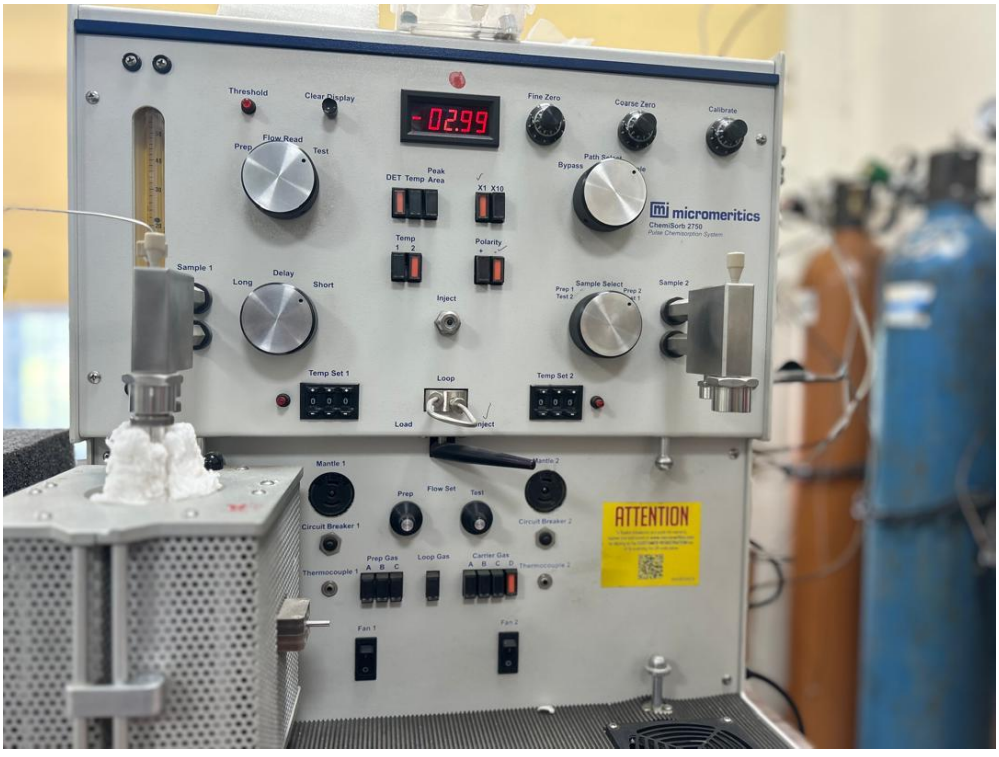
Step 11:click on Gas Flow after selecting the type of analysis on the same dialog box and small box for gas flow will come.and make sure the following :

- Make sure its 10%H_2 balance in Argon for carior gas
- Make sure flow is 20 also look the machine flow meter if it corrosponnds
- Path select should be on “sample”
- Delay should be on "short”
- Cold trap should be on “installed”
- Loop should be on "inject”

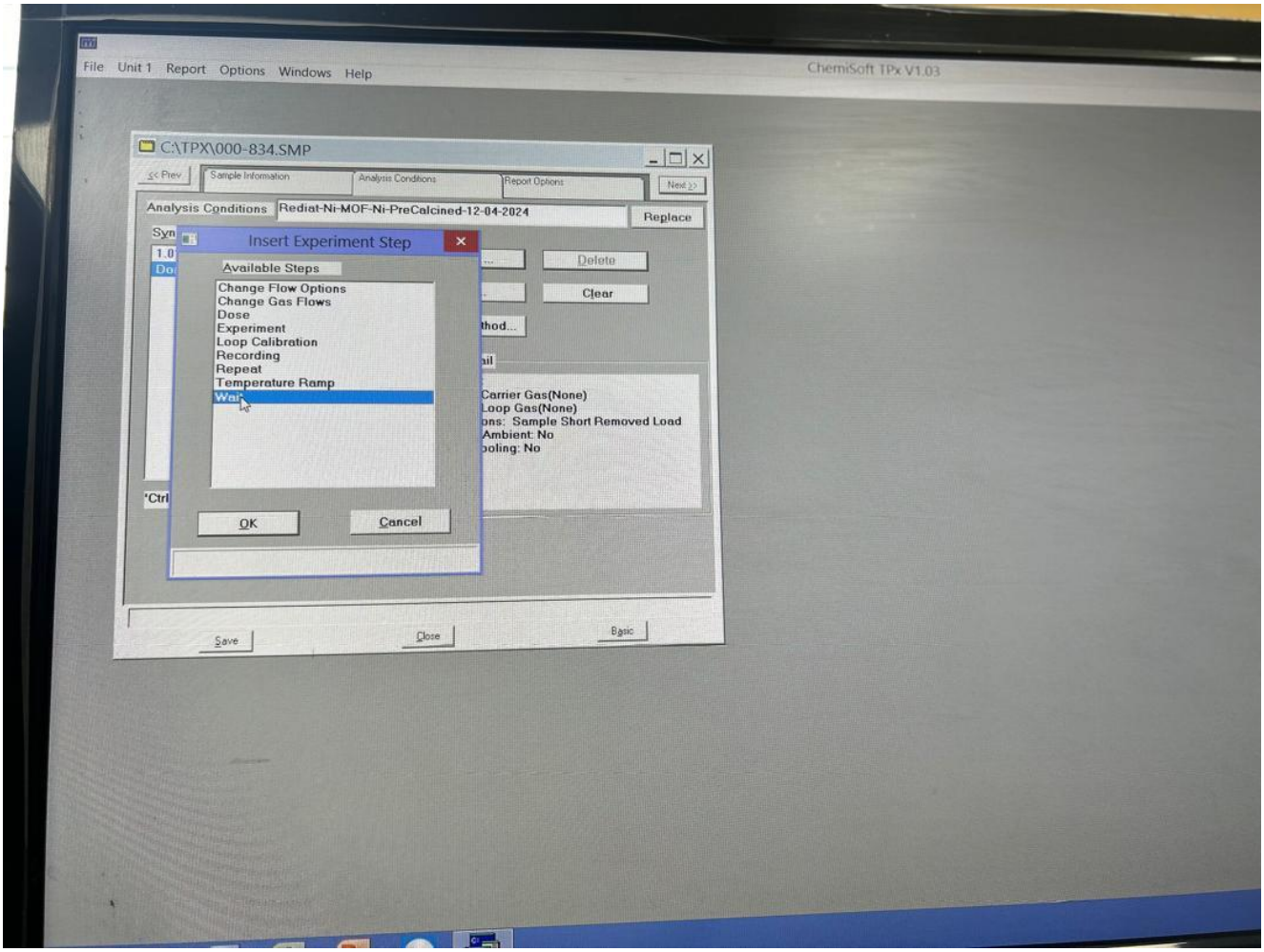
  If the above are all set then click Ok



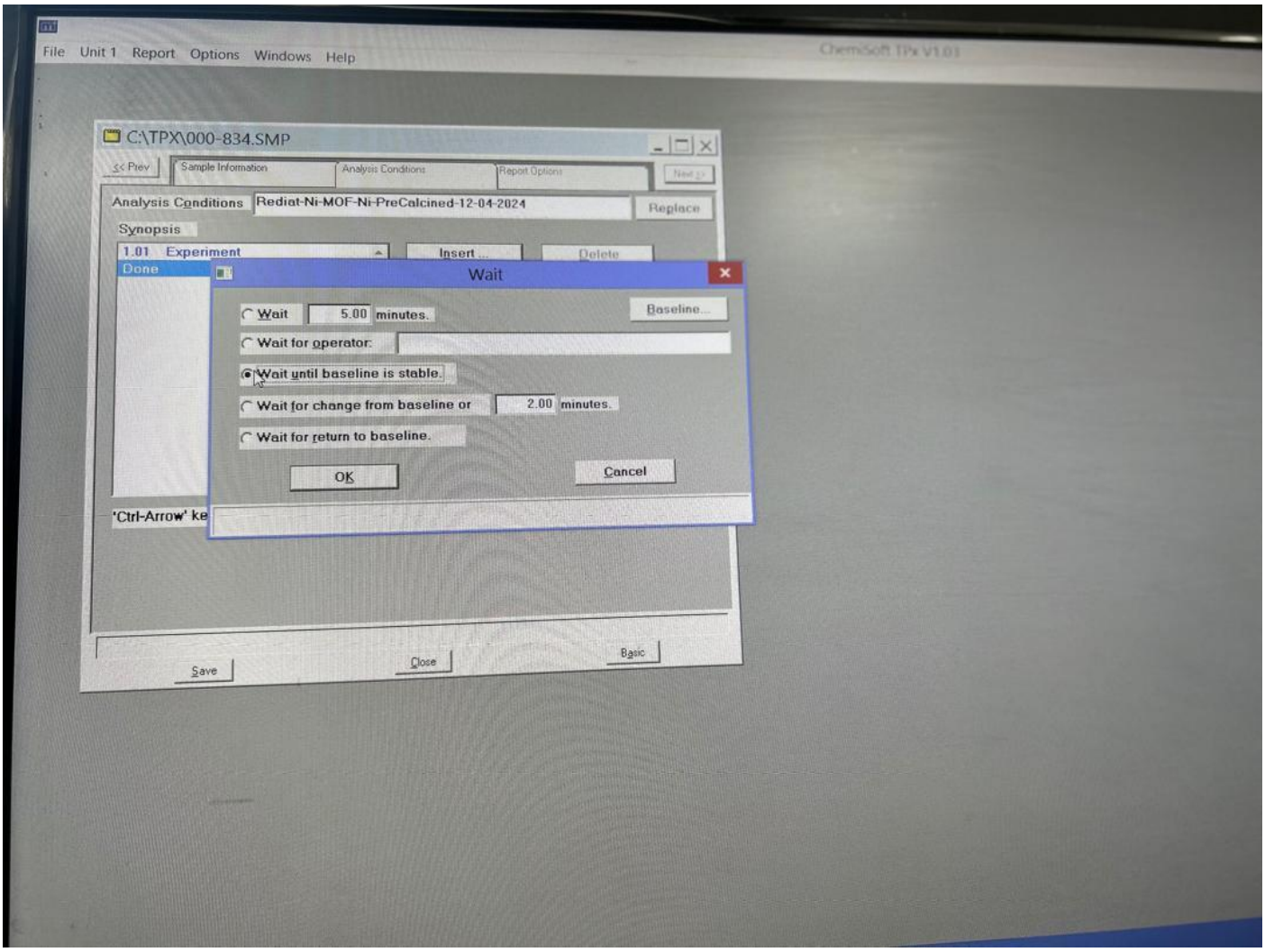

File Unit 1 Report Options Windows Help
ChemiSoft TPx V1.03
C:\TPX\000-834.SMP
Prev
Sample Information
Analysis Conditions
Report Options
Next
Analysis Conditions
Rediat-Ni-MOF-Ni-PreCalcined-12-04-2024
Replace
New Experiment
Experiment description
Rediat-Ni-MOF-Ni-PreCalcined-12-04-2024
Type of analysis
Temperature Programmed Reductior
Active Metals
Initial Conditions
Gas Flows...
Peaks...
Recording Options
Zero the TCD si
Invert the TCD s
Gas Flows
Carrier Gas
10 H2-Ar
Flow
20
Loop Gas
None
Active Concentration
0.00 %
Flow Options
Path Select
Sample
Bypass
Delay
Short
Long
Cold Trap Dewar
Removed
Installed
Loop (2750 Only)
Load
Inject
OK
Cancel
Save


micromeritics
ATTENTION

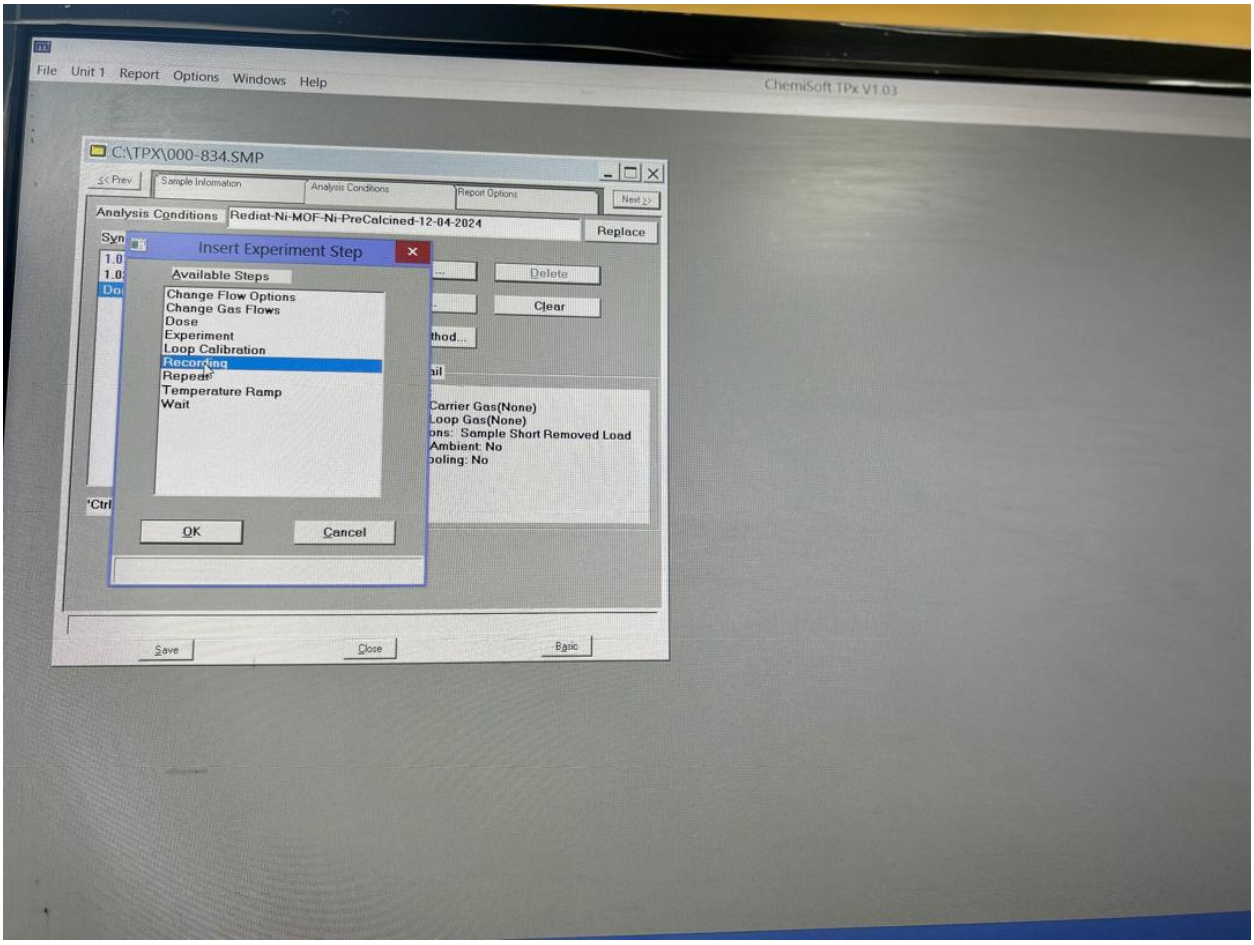
Step 12 : Click on “Insert” again click on “Wait” and click “Ok”

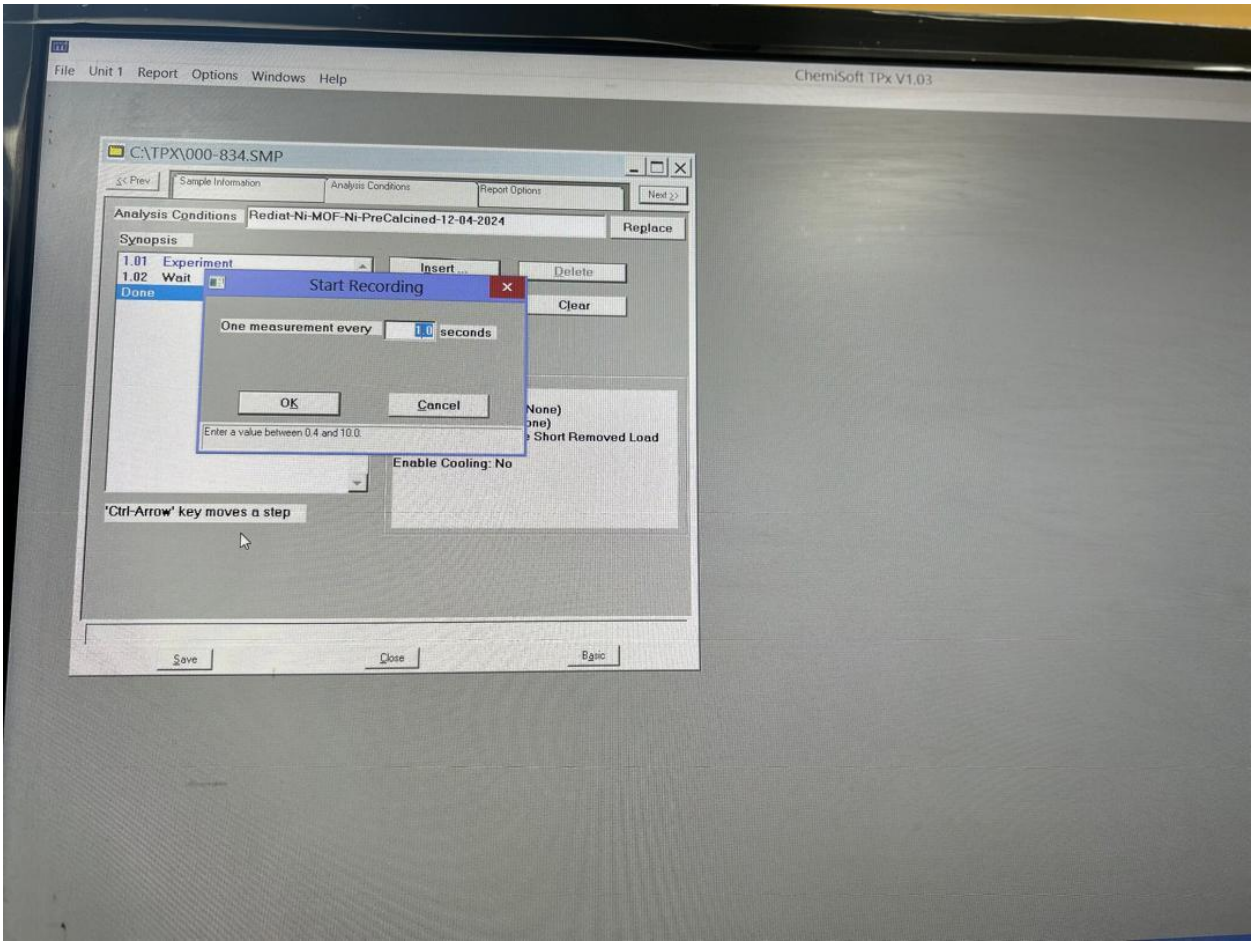


On the coming dialog box select “Until baseline in stable” then press “Ok”

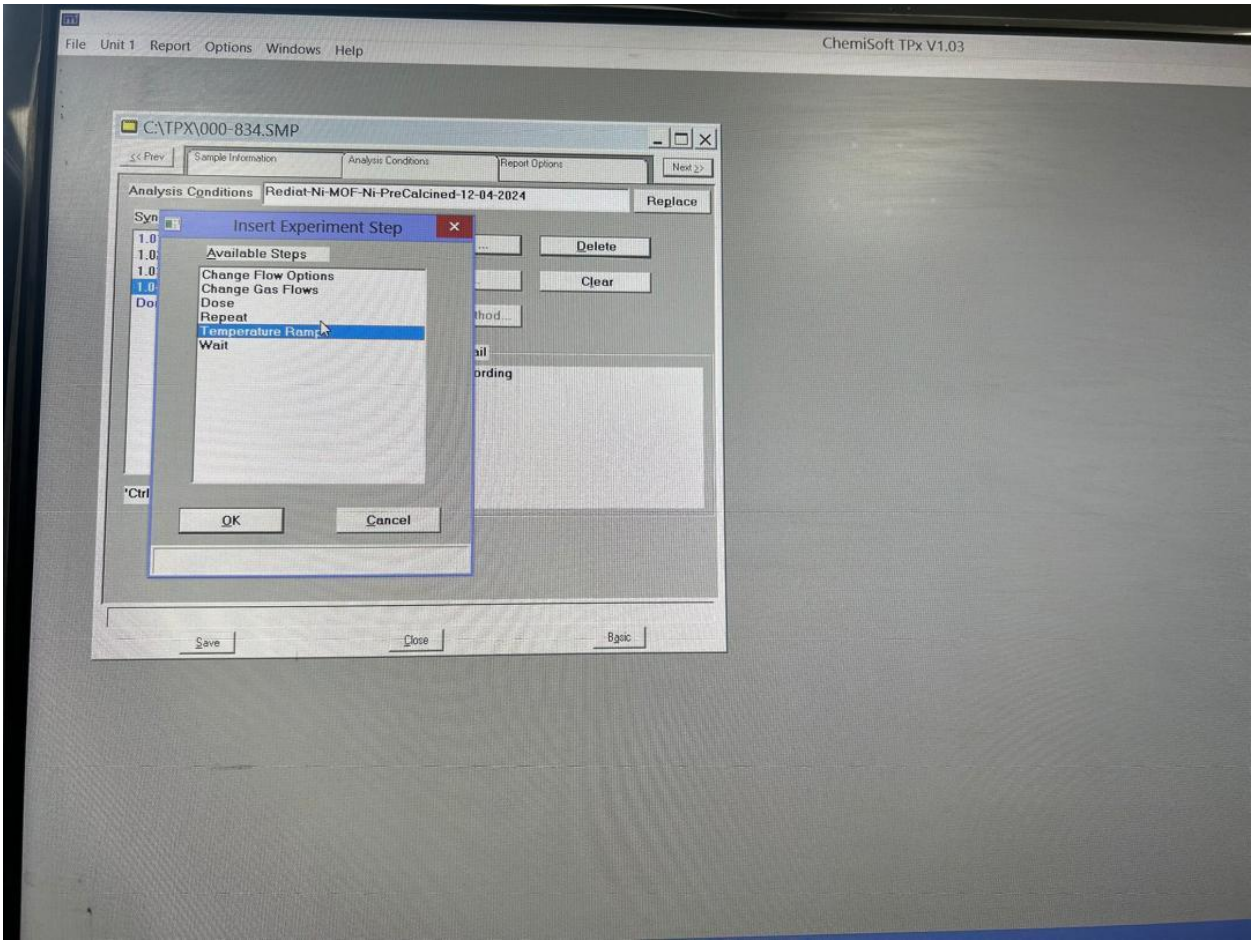


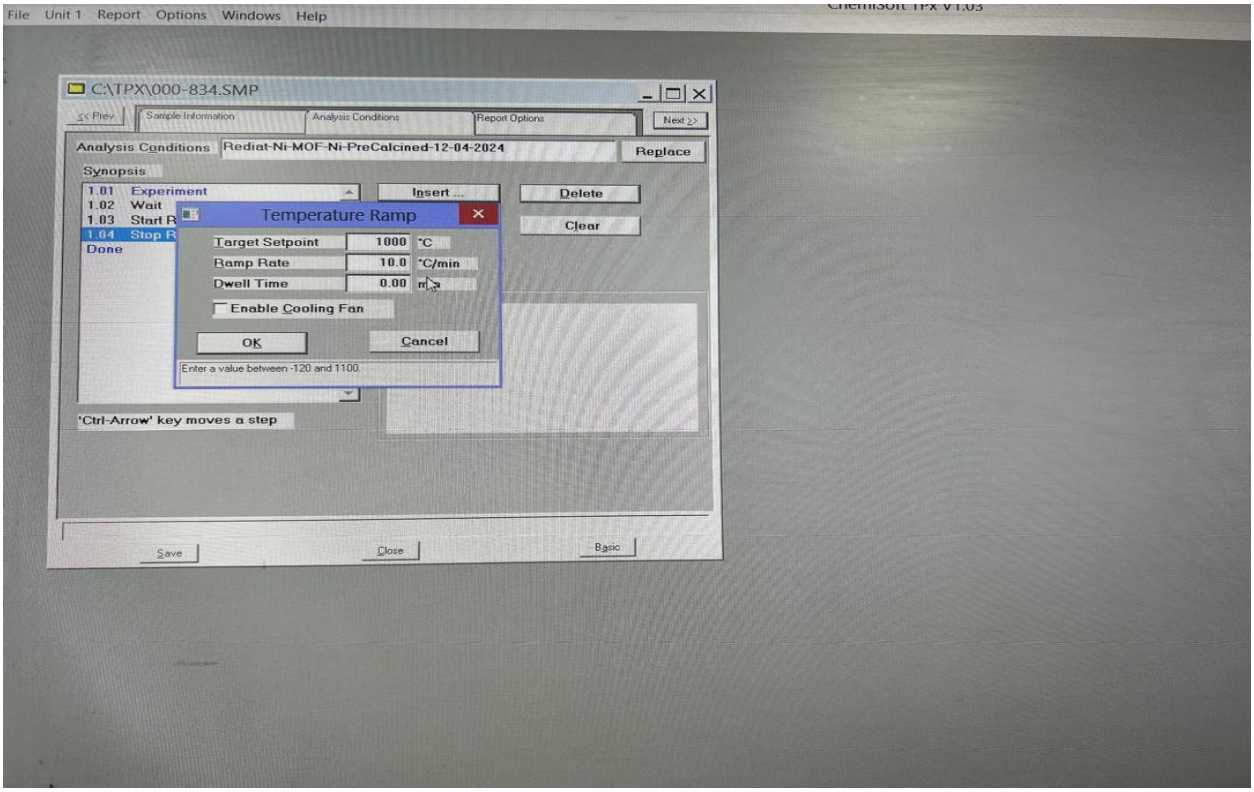
Step 13: Again click on “Insert” select “Recording” then click “OK” another dialog box will open , make sure the “on measurement is on 1.0 sec then click “OK”




File Unit 1 Report Options Windows Help
ChemiSoft TPx V1.03
C:\TPX\000-834.SMP
<< Prev
Sample Information
Analysis Conditions
Report Options
Next >>
Analysis Conditions
Rediat-Ni-MOF-Ni-PreCalcined-12-04-2024
Replace
Synopsis
1.01 Experiment
1.02 Wait
Done
Insert ...
Delete
Clear
Start Recording
One measurement every
1.0
seconds
OK
Cancel
Enter a value between 0.4 and 10.0.
None)
one)
Short Removed Load
Enable Cooling: No
'Ctrl-Arrow' key moves a step
Save
Close
Basic

Step 14: Again click on “Insert” click on “ Temperature ramp” and click “OK” Then another dialog box will open, make sure the temperature is 1000 ^{o}C if not set it 1000 ^{o}C and click “OK”

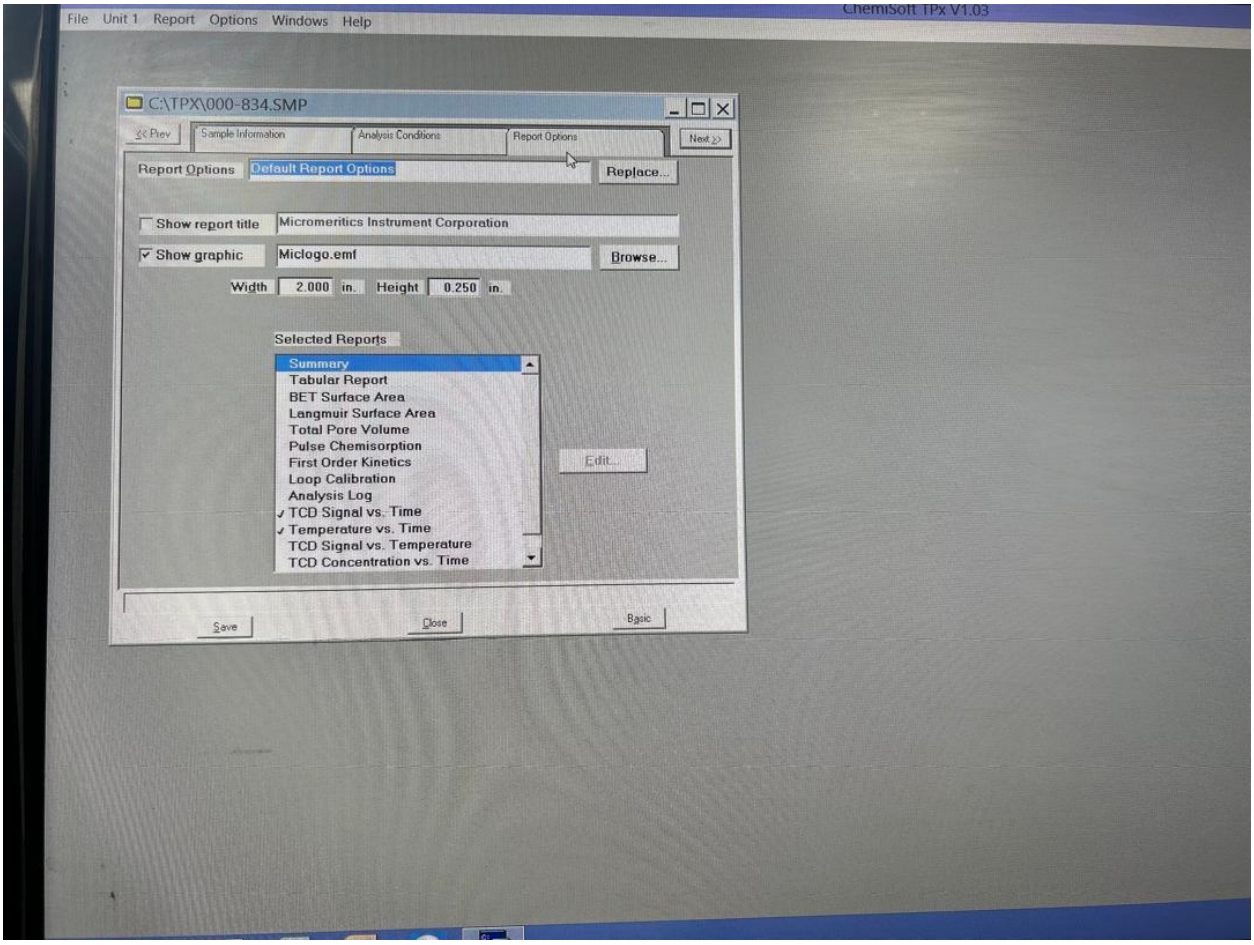


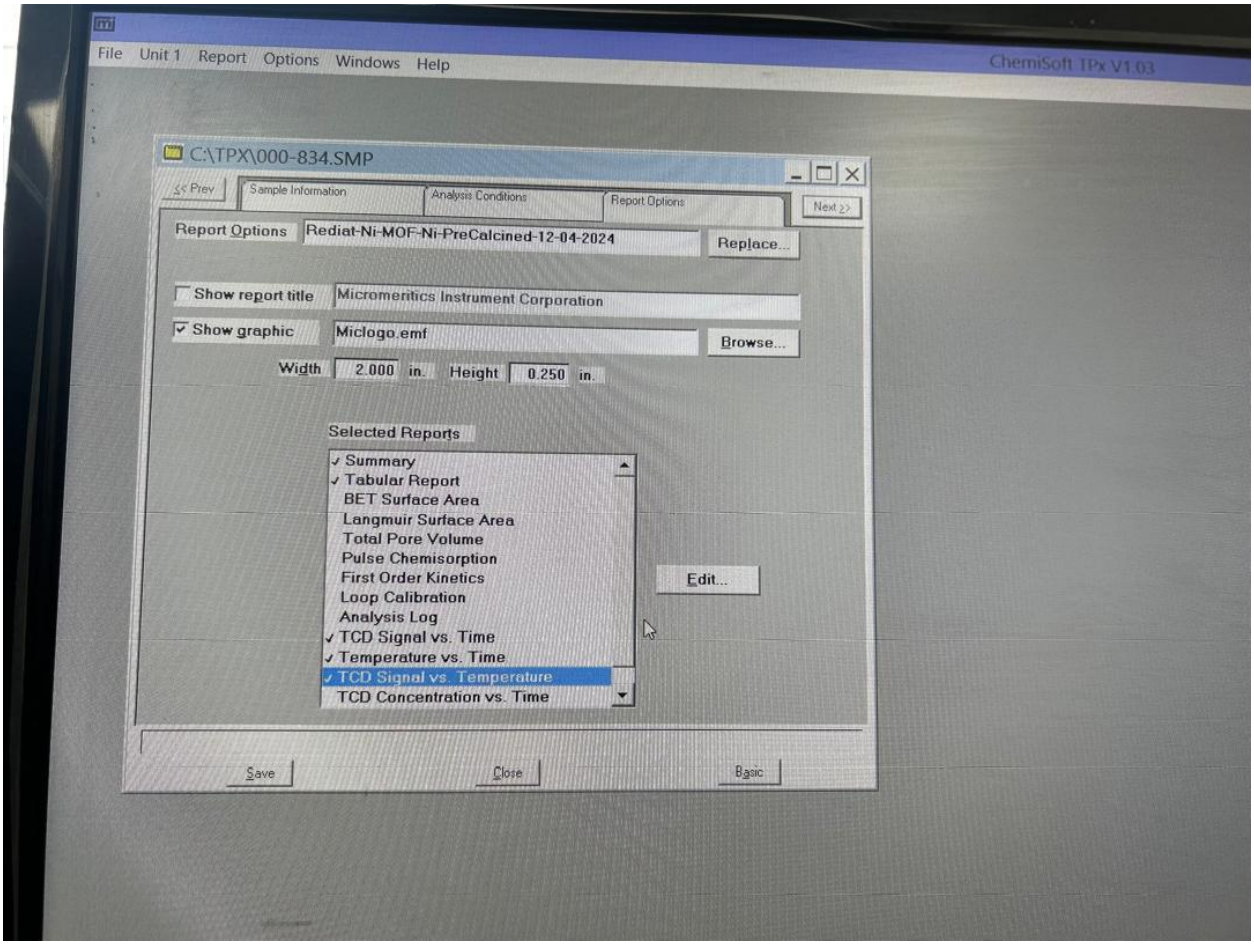

File Unit 1 Report Options Windows Help
C:\TPX\000-834.SMP
<< Prev
Sample Information
Analysis Conditions
Report Options
Next >>
Analysis Conditions
Rediat-Ni-MOF-Ni-PreCalcined-12-04-2024
Replace
Synopsis
1.01 Experiment
1.02 Wait
1.03 Start R
1.04 Stop R
Done
Insert ...
Delete
Clear
Temperature Ramp
Target Setpoint 1000 °C
Ramp Rate 10.0 °C/min
Dwell Time 0.00 m
Enable Cooling Fan
OK
Cancel
Enter a value between -120 and 1100.
'Ctrl-Arrow' key moves a step
Save
Close
Basic

Step 15: Go to report condition

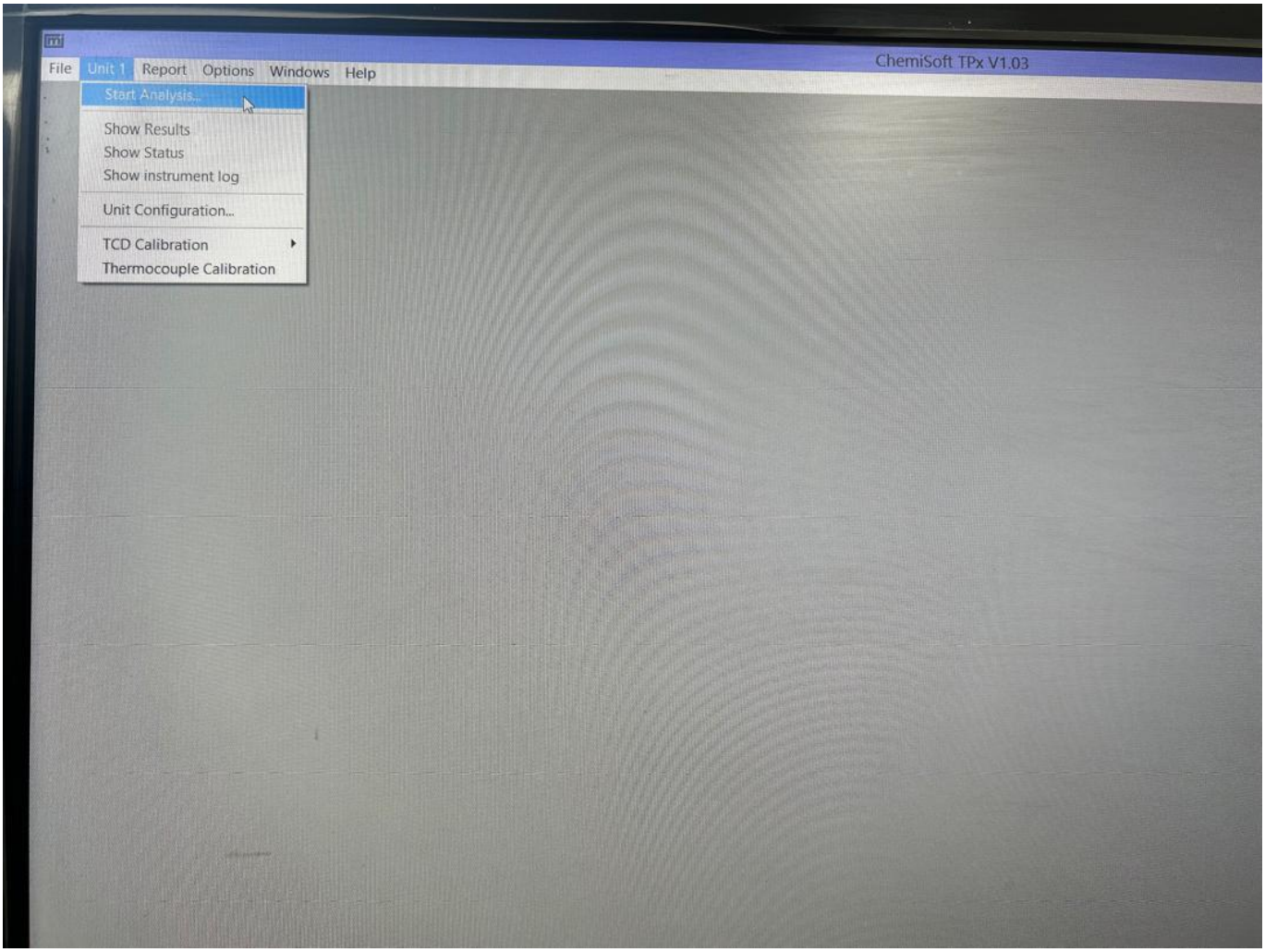
- Paste the sample name on the report option section
- By double-clicking on the following names make sure they are selected and when they are selected a check mark will appear on the left side.
    - Summery
    - Tabular report
    - Already two selection will be made and add the one which says “TCD signal Vs Temperature”

Then click save and click close

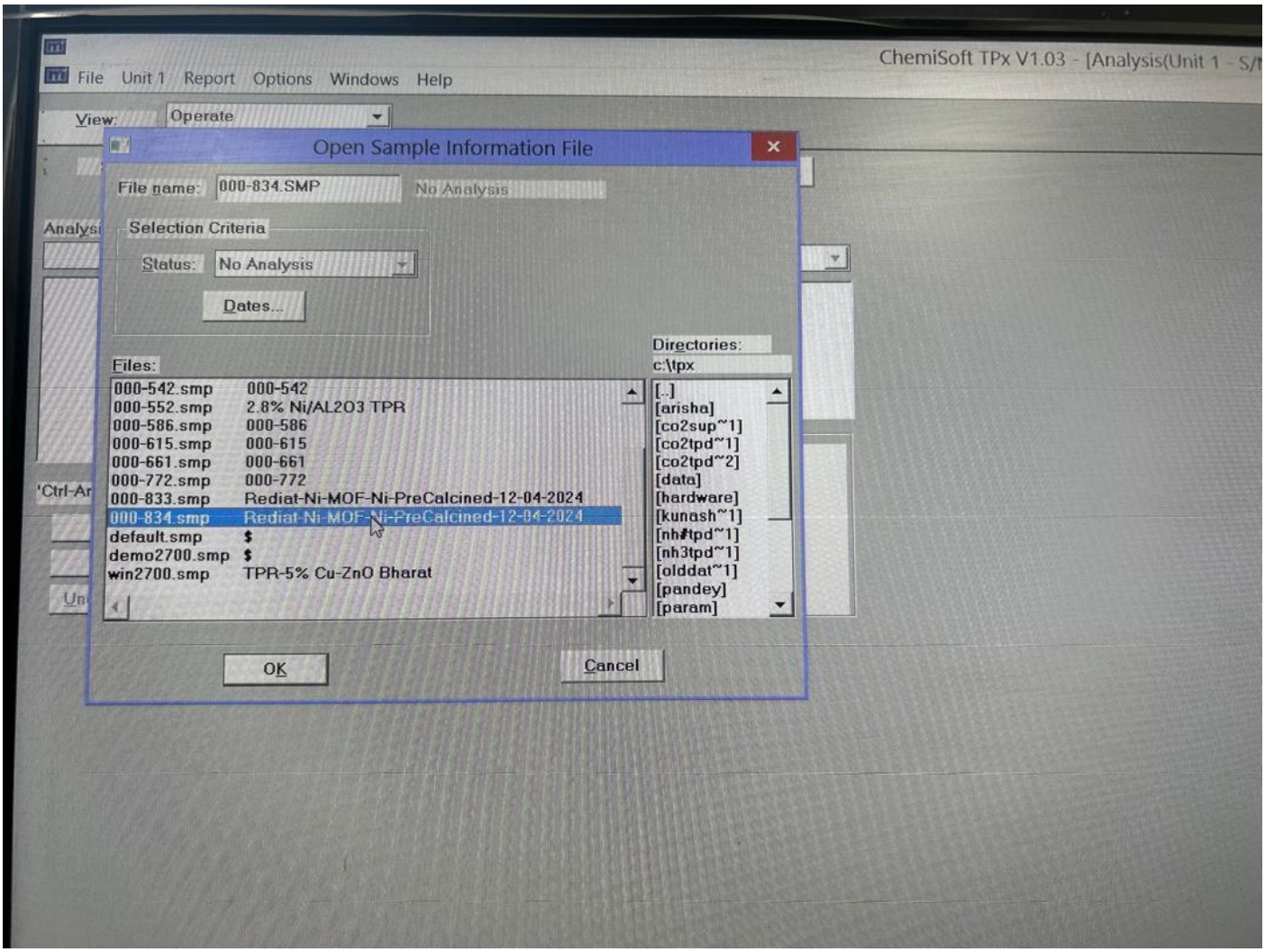



File Unit 1 Report Options Windows Help
ChemiSoft TPx V1.03
C:\TPX\000-834.SMP
<< Prev
Sample Information
Analysis Conditions
Report Options
Next >>
Report Options
Rediat-Ni-MOF-Ni-PreCalcined-12-04-2024
Replace...
Show report title
Micromeritics Instrument Corporation
Show graphic
Miclogo.emf
Browse...
Width 2.000 in.
Height 0.250 in.
Selected Reports
✓ Summary
✓ Tabular Report
BET Surface Area
Langmuir Surface Area
Total Pore Volume
Pulse Chemisorption
First Order Kinetics
Loop Calibration
Analysis Log
✓ TCD Signal vs. Time
✓ Temperature vs. Time
✓ TCD Signal vs. Temperature
TCD Concentration vs. Time
Edit...
Save
Close
Basic

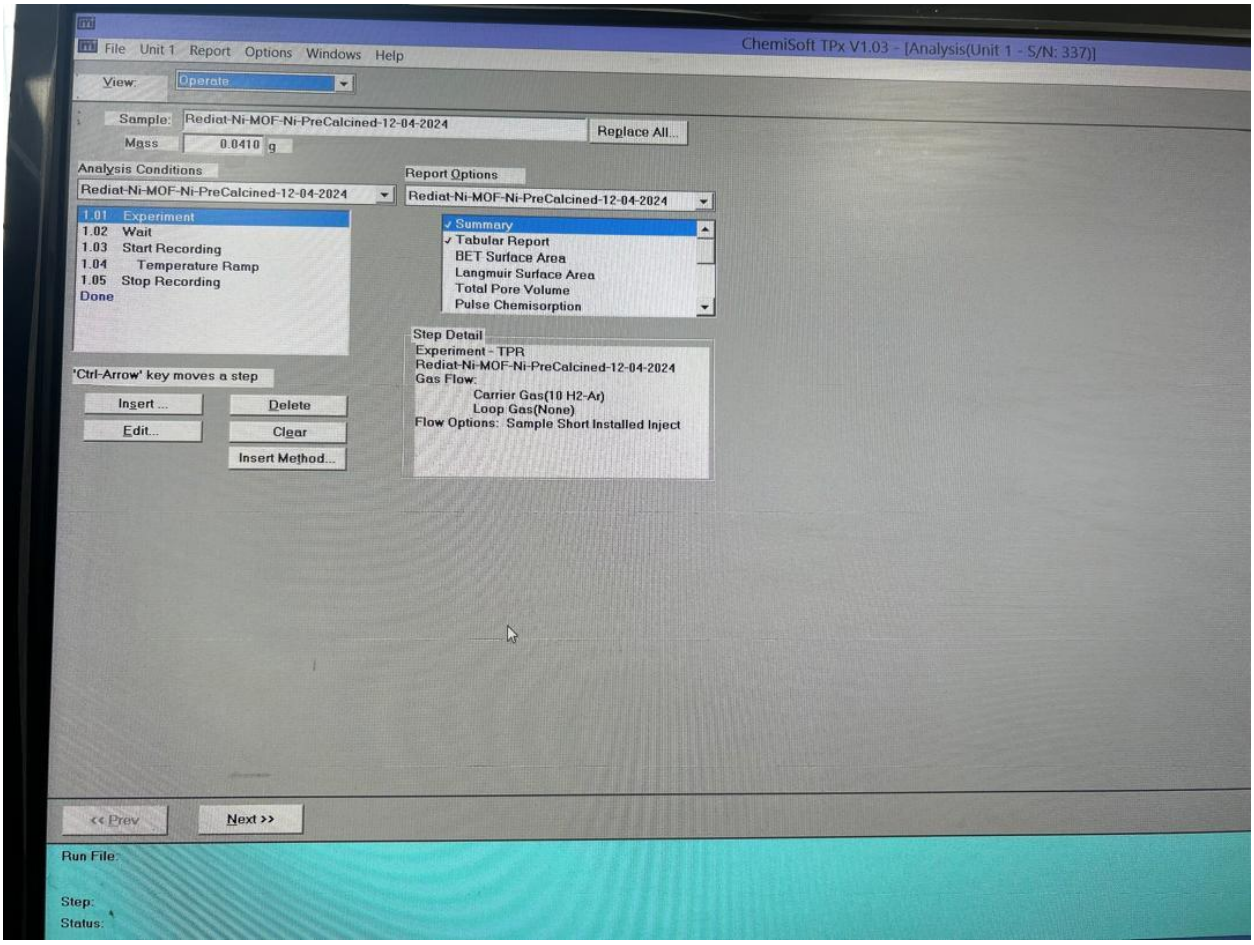
Step 16 : click on the “Unit 1” and click on “Start Analysis”



and then on the showing dialog box select your program and click “OK”



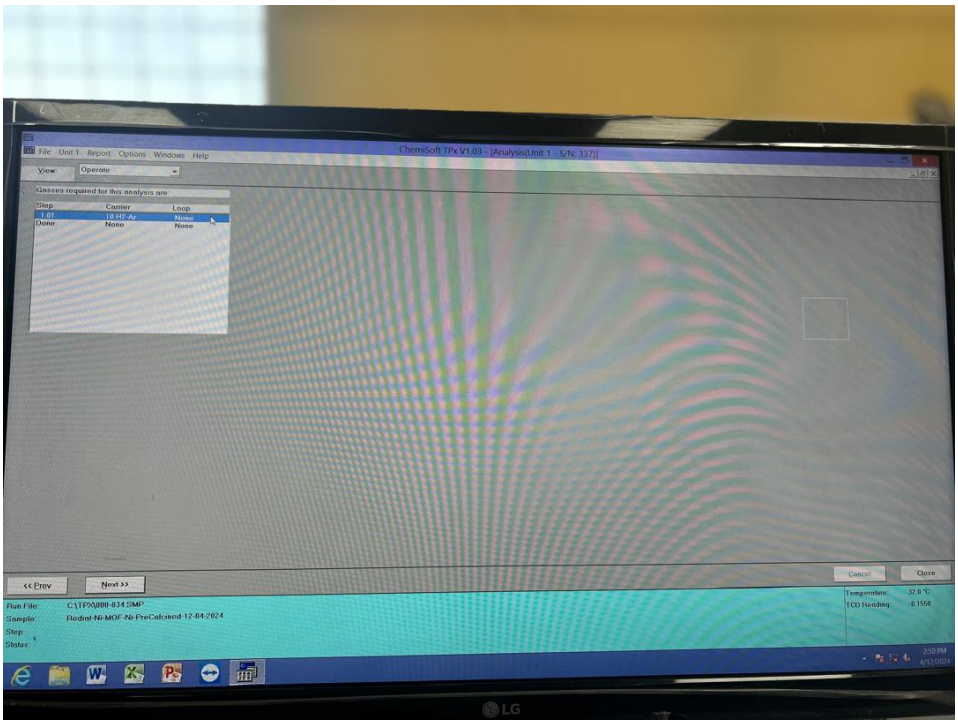
Step 17: After clicking “ok” from the previous dialog box, now all the things that you have filled will be shown so make sure everything is accordingly put or set.



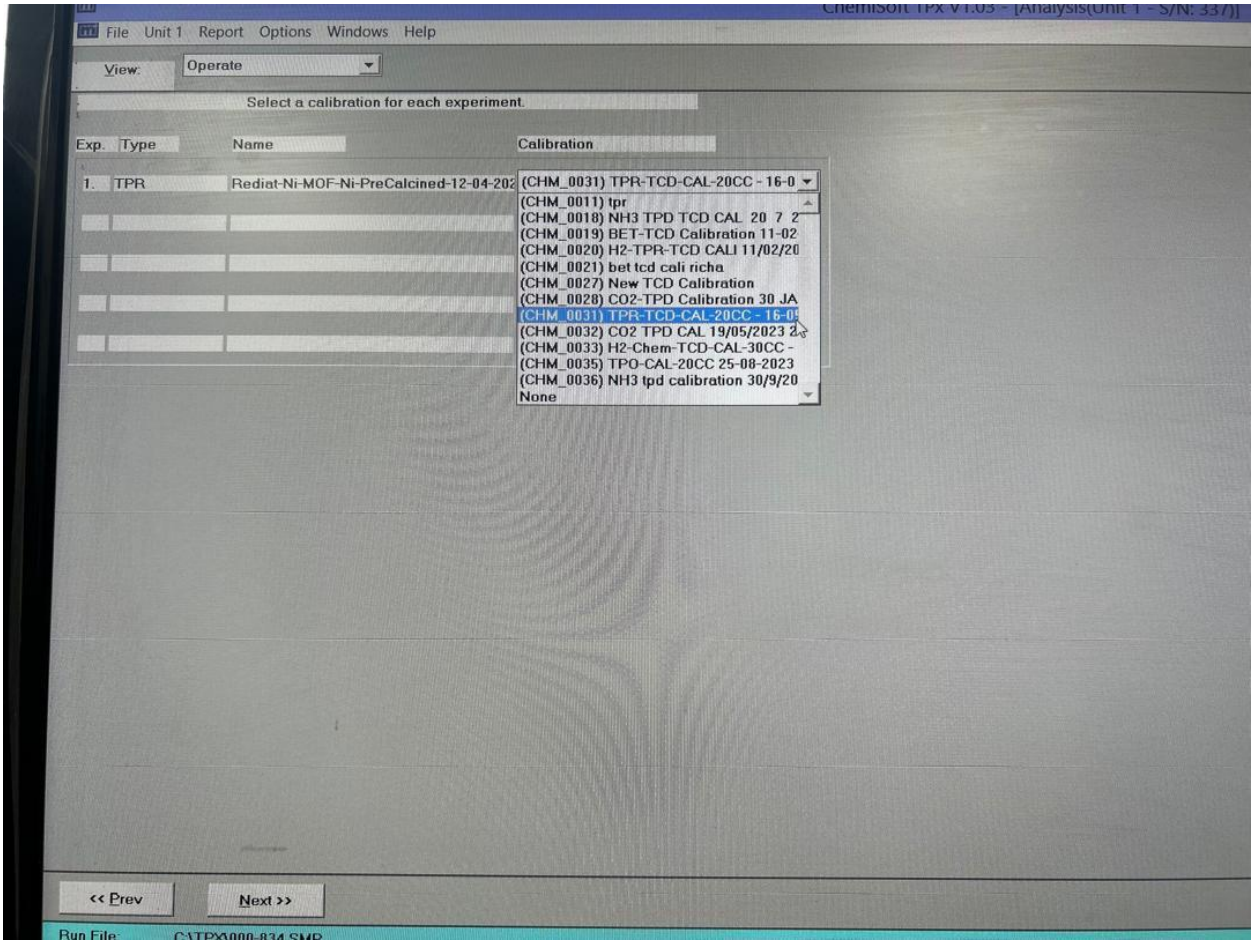
Step 18: Prepare a cold trap place it in the machine and cover it

- For speeding up purposes, change gas to H_2 balance Ar first
- While preparing the cold trap, make sure you wear the proper glove
- Make sure the metal container is at list 2/3 and is filled with IPA
- Slowly add the liquid N_2 along with continuous stirring and make sure no ice balls are made which will possibly break the U-shaped tube.

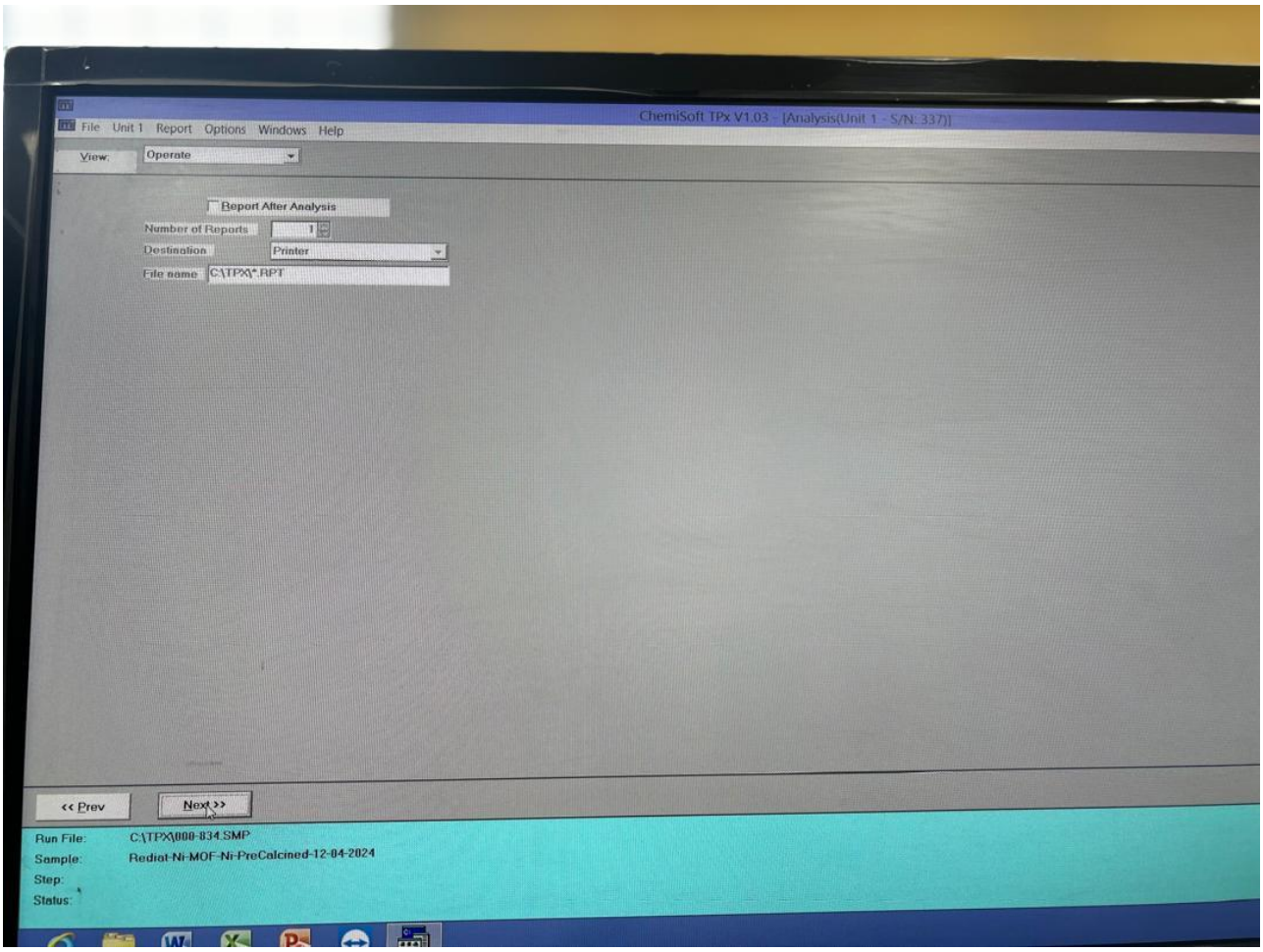
Step 19 : then click Next on the screen select $10H_2$-Ar and then click “Next”



Step 20: Under the calibration drop select the TPR calibration method shown in the picture then click "Next"



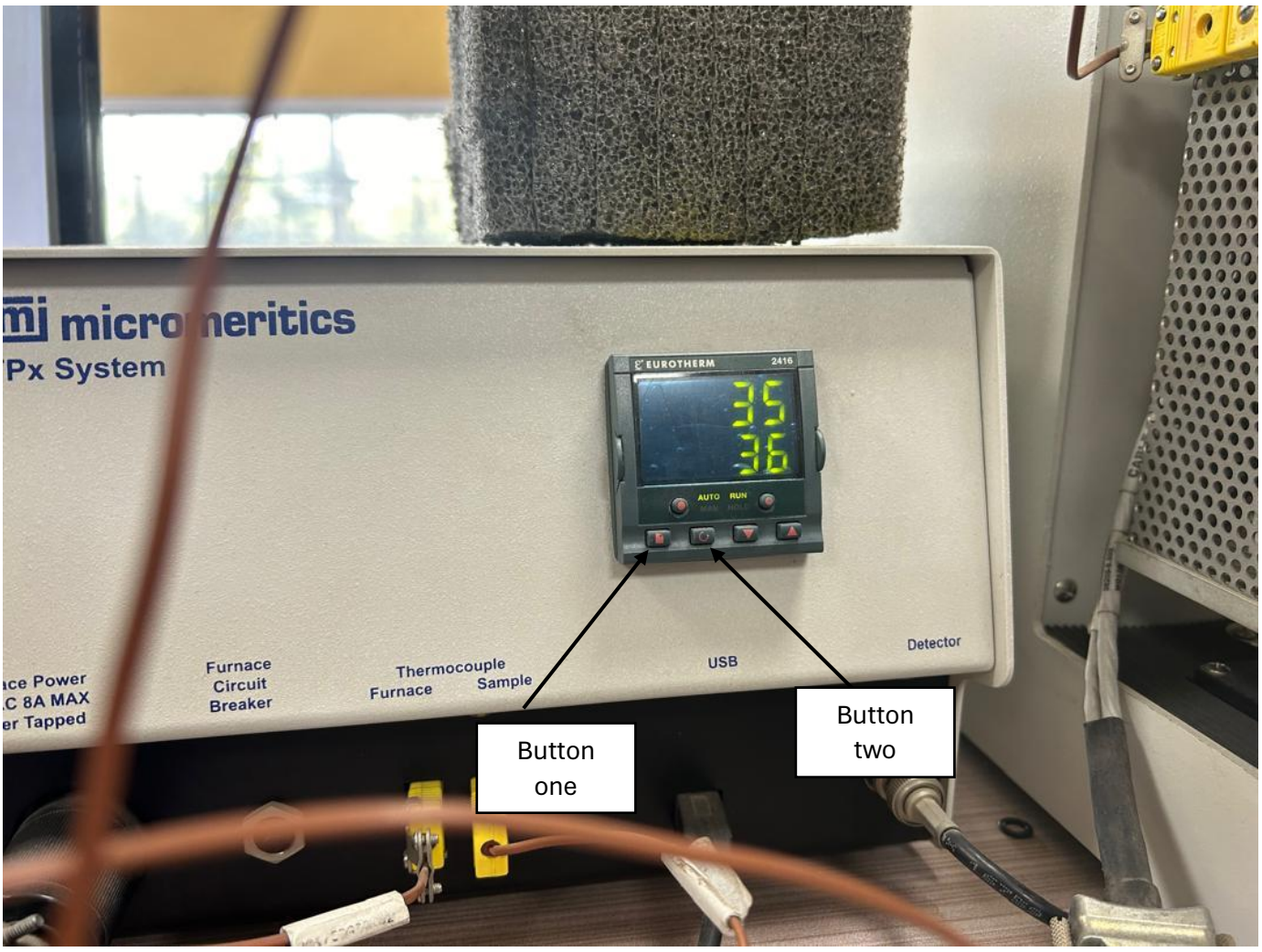
Again “Next”

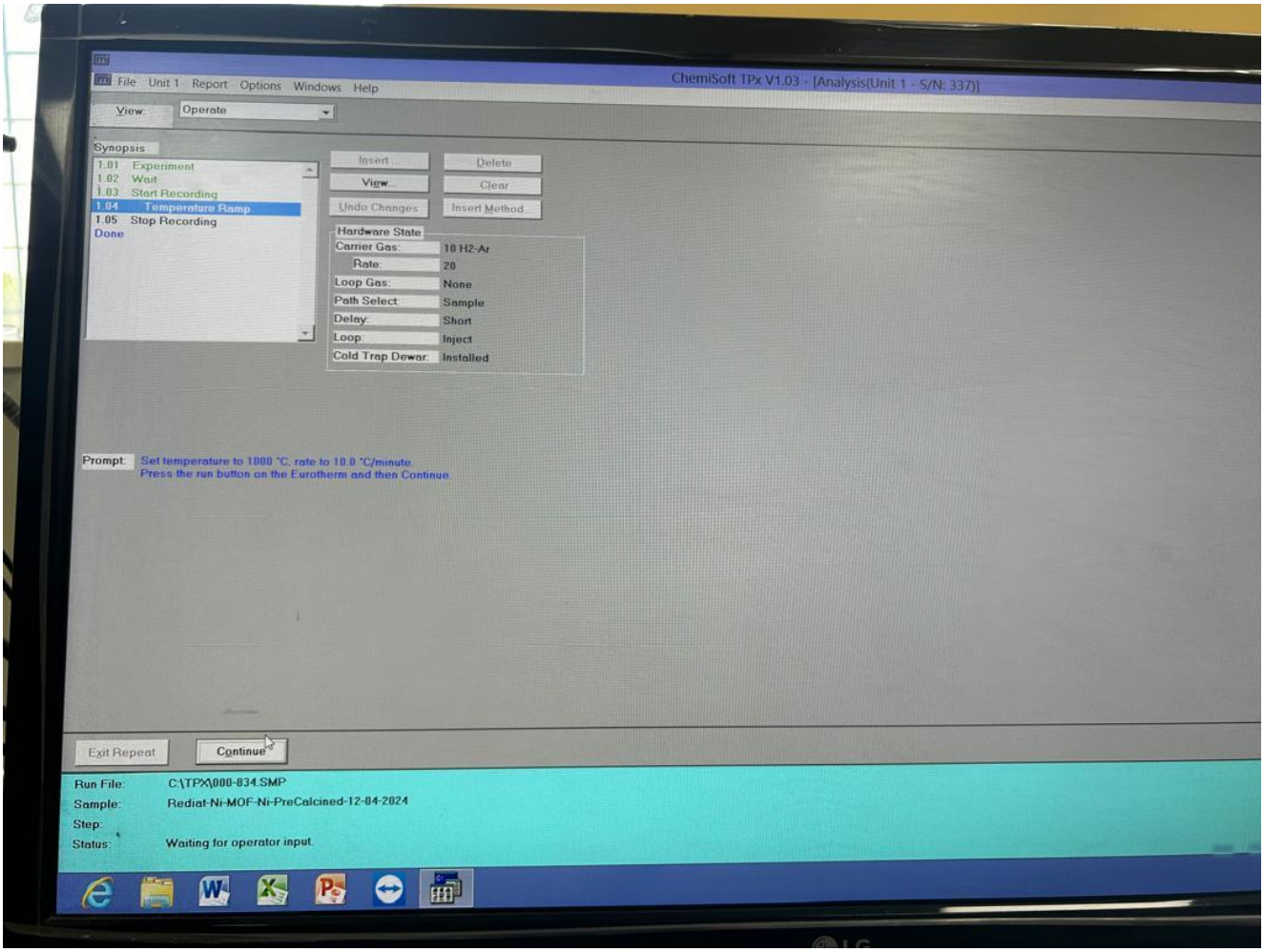


Step 21: then click “Next” and “Continue” accordingly by seeing the prompt message till you reach the message about the temperature setting 1000 ^{o}C

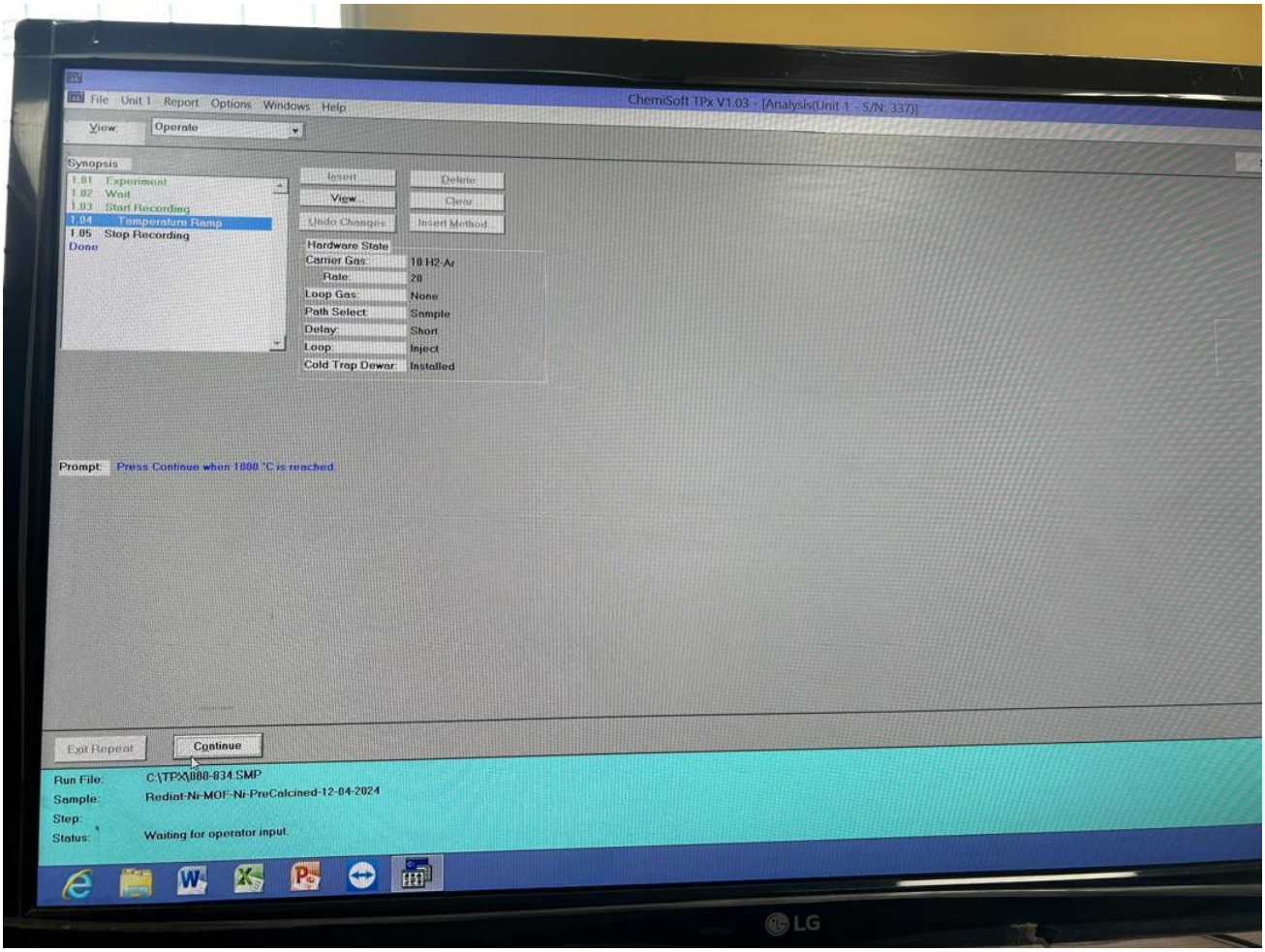
Step 22: When the message says “set the temperature 1000 ºC rate to 10.0 ºC/min. press the run button on the Eurotherm and then continue” so press the run button on the Eurotherm and continue **but** before that check if the set temperature is 1000 ºC by using this link for the video : https://drive.google.com/file/d/1FjbMahdLZQ6j51CvnWHhta0nQAlY0Vw_/view?usp=drive_link or use the following step

Press the first button from the left on the four buttons together in the Eurotherm then you will see “PROGRAM LIST” → press the next button from the button that you have pressed before till you see TGT(target) and check if its 1000 if not make it 1000 by using the increase button which is the forth one → after setting and checking, press again the second button from the left till you find TYPE END then press the first button till it shows the set and target temperature display.

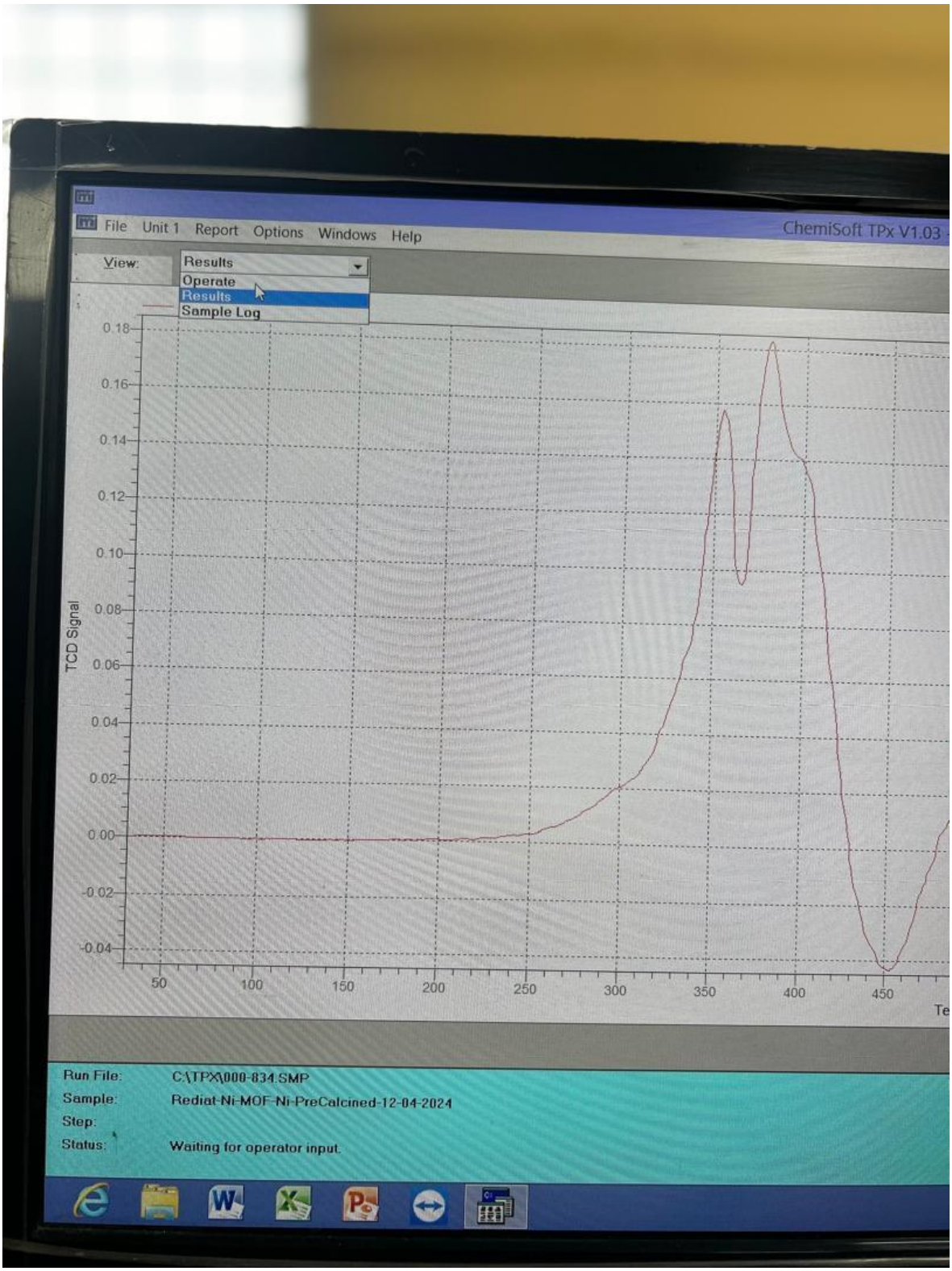



File Unit 1 Report Options Windows Help
ChemiSoft TPx V1.03 - [Analysis(Unit 1 - S/N: 337)]
View: Operate
Synopsis
1.01 Experiment
1.02 Wait
1.03 Start Recording
1.04 Temperature Ramp
1.05 Stop Recording
Done
Insert...
Delete
View...
Clear
Undo Changes
Insert Method...
Hardware State
Carrier Gas: 10 H2-Ar
Rate: 20
Loop Gas: None
Path Select: Sample
Delay: Short
Loop: Inject
Cold Trap Dewar: Installed
Prompt: Set temperature to 1000 °C, rate to 10.0 °C/minute.
Press the run button on the Eurotherm and then Continue.
Exit Repeat
Continue
Run File: C:\TPX\000-834.SMP
Sample: Rediat-Ni-MOF-Ni-PreCalcined-12-04-2024
Step:
Status: Waiting for operator input.

Step 23 : when the temperature reaches(approximately after 1:30hr) 1000 °C then press continue and next button till you see the close button showing for closing, for faster cooling long press the button which you used to run the erothrem till you see 33 display on the machine if you are using helium cylinder then you can open other gases like N2 and press the button accordingly on the machine for cooling and close the helium cylinder.after machine is cooled you meaning if the temperature in the eurotherm is < 100 °C you can close the eurotherm. When closing make sure you close the main switch of the eurotherm and the main machine first then close the cylinders and after that close the main switch.



Step 24: to see the analysis graph, you can click on the drop-down and select “Results”



Step 25: extracting the result

Click on Report on top → Start Report find your name of program → click Ok → click OK→ Click on "Save Us" on the right side → change the file name accordingly as you want and choose the file type to be .xls so that you can get it in a format compatible for drawing on origin → save it on place you want to save.

ADDITIONAL RESOURCE :
https://drive.google.com/file/d/1bKUDbSrk3Ktmefhqmp_MMfcJQOKxGf-B/view?usp=sharing