# Netzsch TGA Training Notebook

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# Before you begin…

- ❑ Receive a user name and temporary password for Faces scheduling
- ❑ Identify your ENGR username and Password from Systems
  - ❑ If you don't have an ENGR account, send me the following:
    - ❑ Full name
    - ❑ Principal Investigator (PI)
    - ❑ UCR Net ID
    - ❑ Email
- ❑ Coordinate a time with the lab manager for training
- ❑ Schedule a 2 hour block on Faces for your training

# Netzsch TGA Operation

I. Preparation

II. Start

III. Setup & Control

IV. New Method (no baseline measurement)

V. Opening Method

VI. ASC Manager

VII. Running Experiments

VIII. Results

IX. Cleanup

X. Red Flags & Mistakes

XI. Troubleshooting

XII. Baseline Corrections

XIII. OIT Experiments – coming soon

# I. Preparation – 1/2

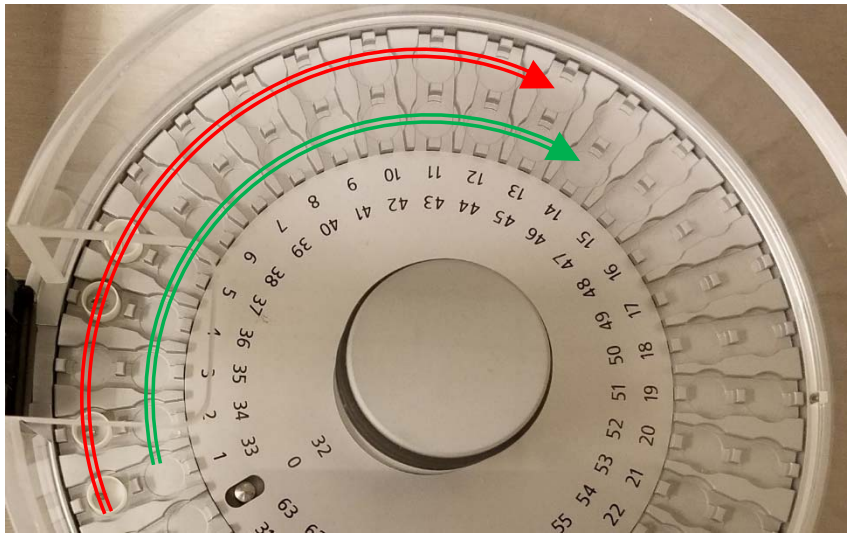
1. Prior to running a TGA test, it is important to perform the following checks to plan for an efficient run and avoid damage to TGA
   I. Acceptable ***Calibrated Crucibles***
      a) Alumina ($Al_2O_3$) Crucibles – 6.8 mm diameter, 4 mm height, 85 µL
   II. Acceptable ***Calibrated Gas environments***
      a) 100% Nitrogen = Purge 2: N2 20 mL/min + Protection: N2 20 mL/min
      b) Air (80% Nitrogen + 20% Oxygen) = Purge 1: Air 20 mL/min + Protection: N2 20 mL/min
   III. Acceptable ***Calibrated Heating Rates***
      a) Nitrogen: 5 K/min, 10 K/min, 20 K/min, 30 K/min <see Lab Manager for different rates>
      b) Air: 2.5 K/min, 20 K/min <see Lab Manager for different rates>

2. Sign in on the ***Sign-In Sheet***

3. Prepare your ***EMPTY Crucibles*** first
   BUT DO NOT PLACE SAMPLES INTO CRUCIBLES YET!

# I. Preparation – 2/2

1. Prepare empty Alumina crucible
2. Blow dry with the provided air gun
3. Identify which position(s) you want your samples to have in ASC tray (Position 0 -> 31, 32 -> 63)



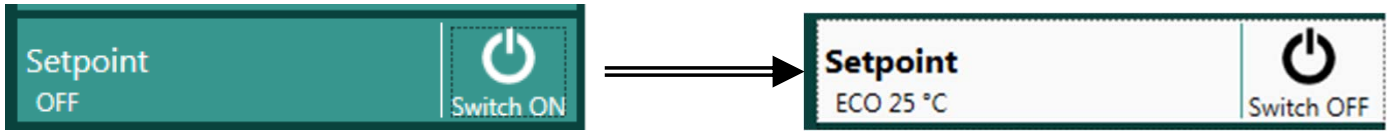
4. With the **FURNACE CLOSED**, you may blow away any dust on the gripper and ASC tray using the provided air gun

# II. Start – 1/2


1. Click ***SmartMode Measurement*** to start TGA measurement program

2. If asked if you want to start ***Setpoint*** now, confirm and click ***Yes***


3. It's **IMPORTANT** to check that the ***Setpoint*** is always ***ON*** when not actively running a test (***Setpoint*** protects TGA with Nitrogen!)




4. Confirm that either ***IDLE 25 °C*** or ***ECO 25 °C*** is shown under ***Setpoint***

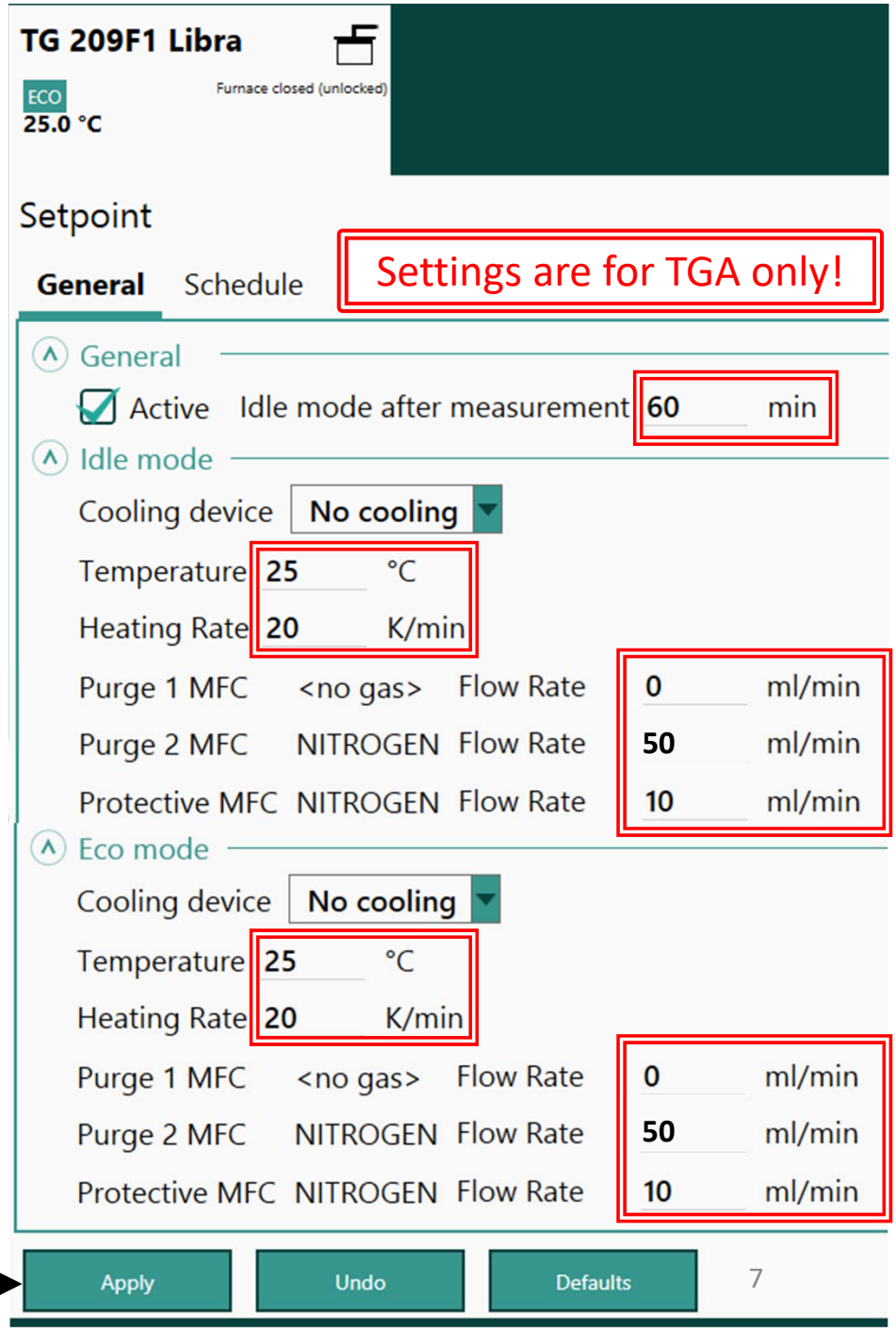
# II. Start – 2/2

5. Click on ***Setpoint***


6. Confirm that the following settings for ***Idle mode*** and ***Eco mode*** match what is shown on the right

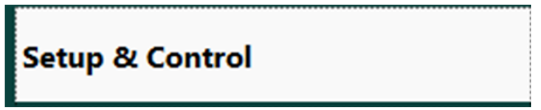
7. If they values are the same, proceed to **III. Setup & Control**

8. If the values are different, proceed to change them back to what is shown on the right and click ***Apply***

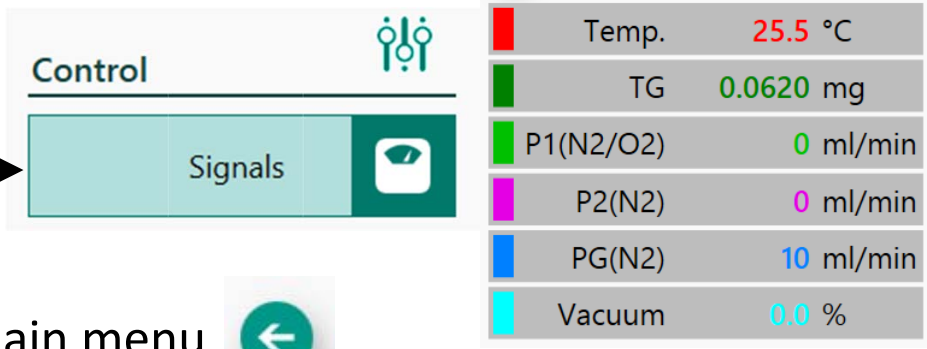


# III. Setup & Control – 1/3

1. Click on ***Setup & Control***

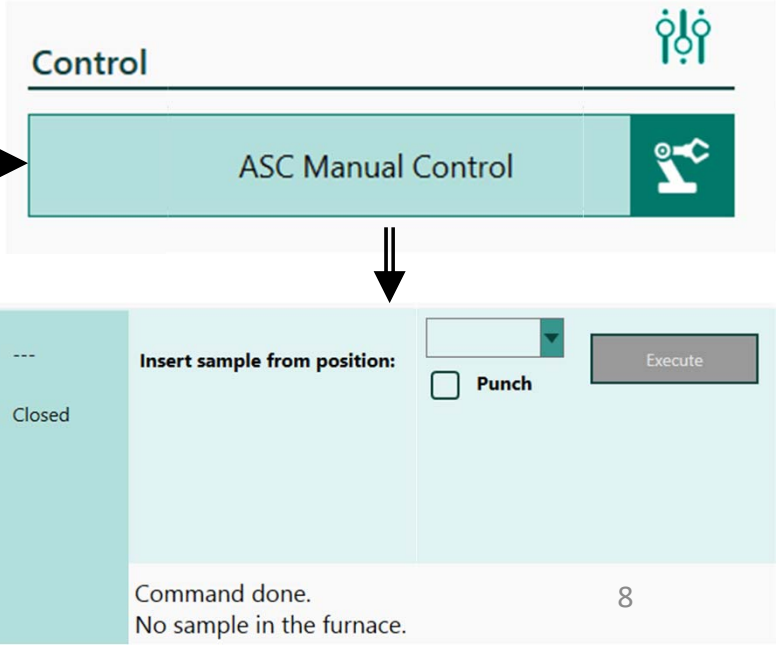



2. Click on ***Signals*** to activate the pop-up window showing the Temperature and Gas Flow signals




3. Click on the ***Back*** button to return to main menu

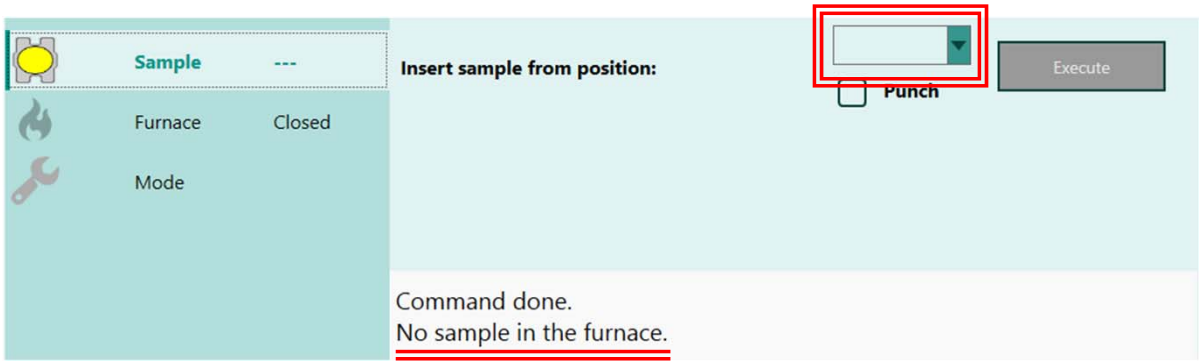
4. Click on ***ASC Manual Control*** to bring up the menu to control the ***Auto Sample Controller***



# III. Setup & Control – 2/3

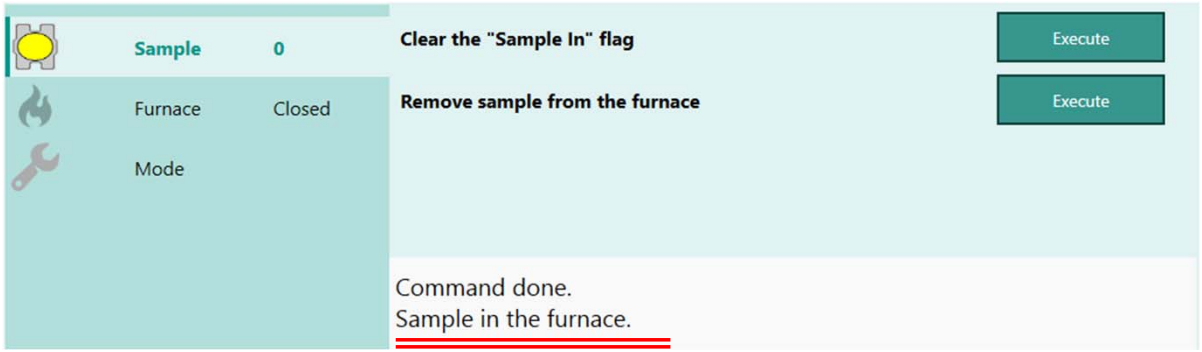
5. Review the different commands available below

Condition: **No sample in the furnace**




- ***Insert sample from position***: Select sample from position in dropdown to insert into furnace

Condition: **Sample in the furnace**

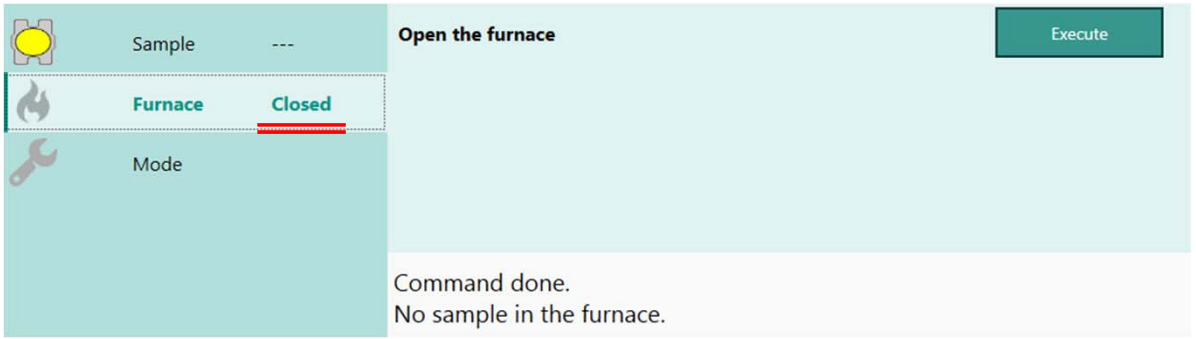



- ***Clear the "Sample In" flag:*** **ONLY** use this if there is **NO** sample in furnace, to correct this "error"
- ***Remove sample from the furnace***: Use this to remove the current sample from the furnace and place back into original sample position

# III. Setup & Control – 3/3

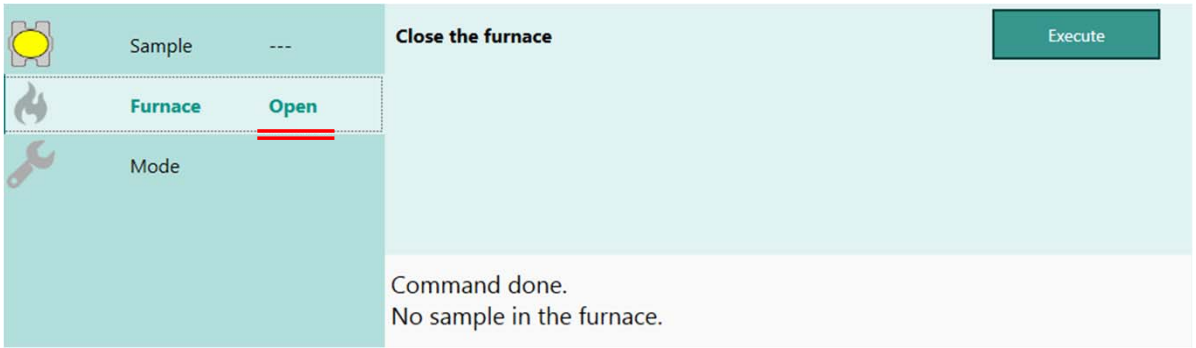
6. Review the different commands available below

Condition: **Furnace is closed**




- ***Open the furnace***: Click to open the furnace

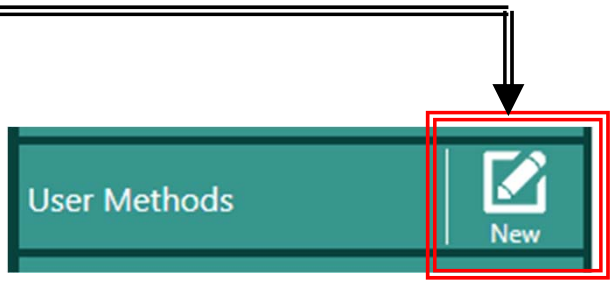
Condition: **Furnace is open**



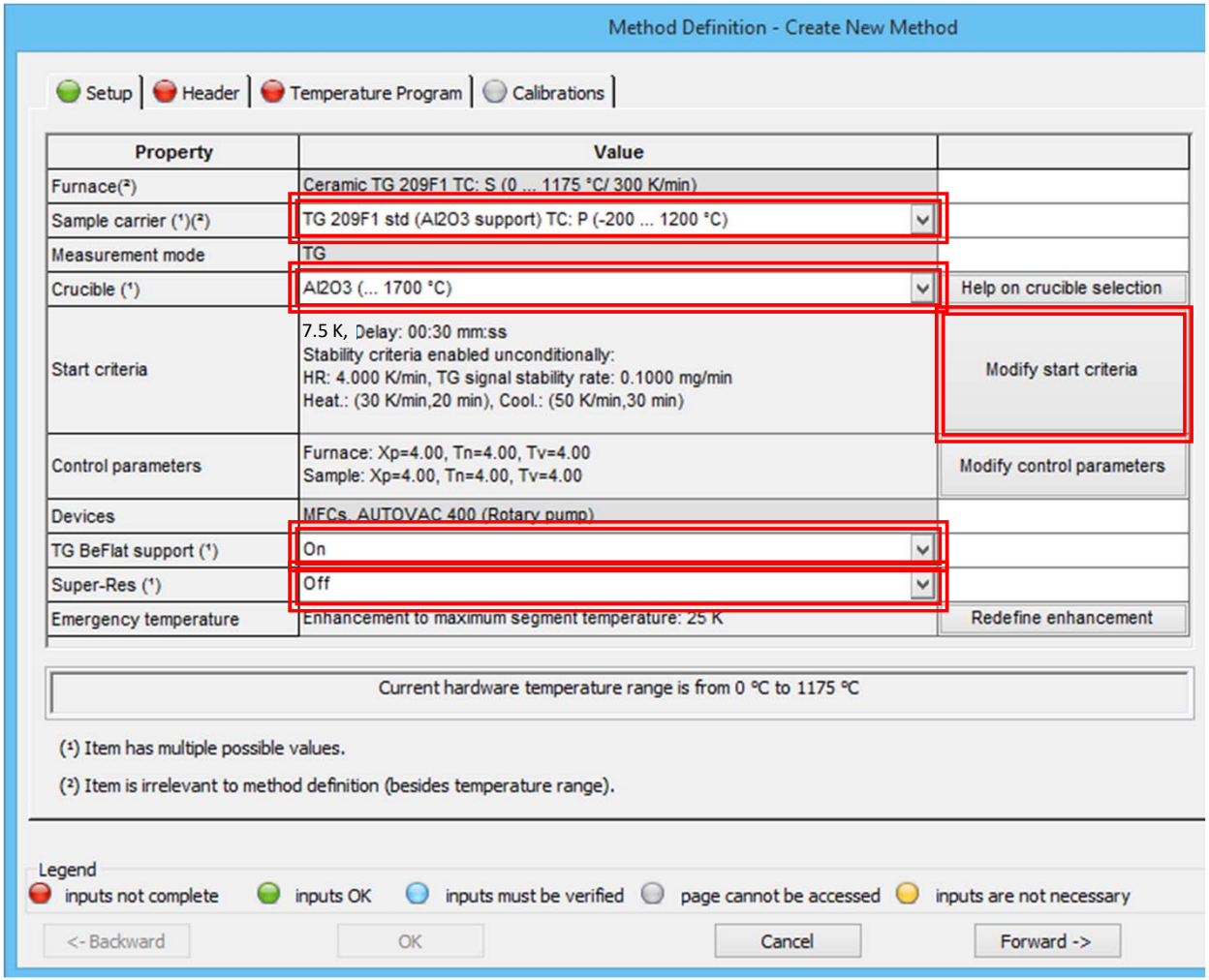

- ***Close the furnace:*** Click to close the furnace

# IV. New Method – 1/9

1. Click on ***New*** next to ***User Methods***




2. Confirm settings below are accurate or acceptable:




3. Sample carrier = ***TG 209F1 std (Al2O3 support)***
   ***TC: P (-200…1200 °C)***

4. Crucible = ***Al2O3 (… 1700 °C)***

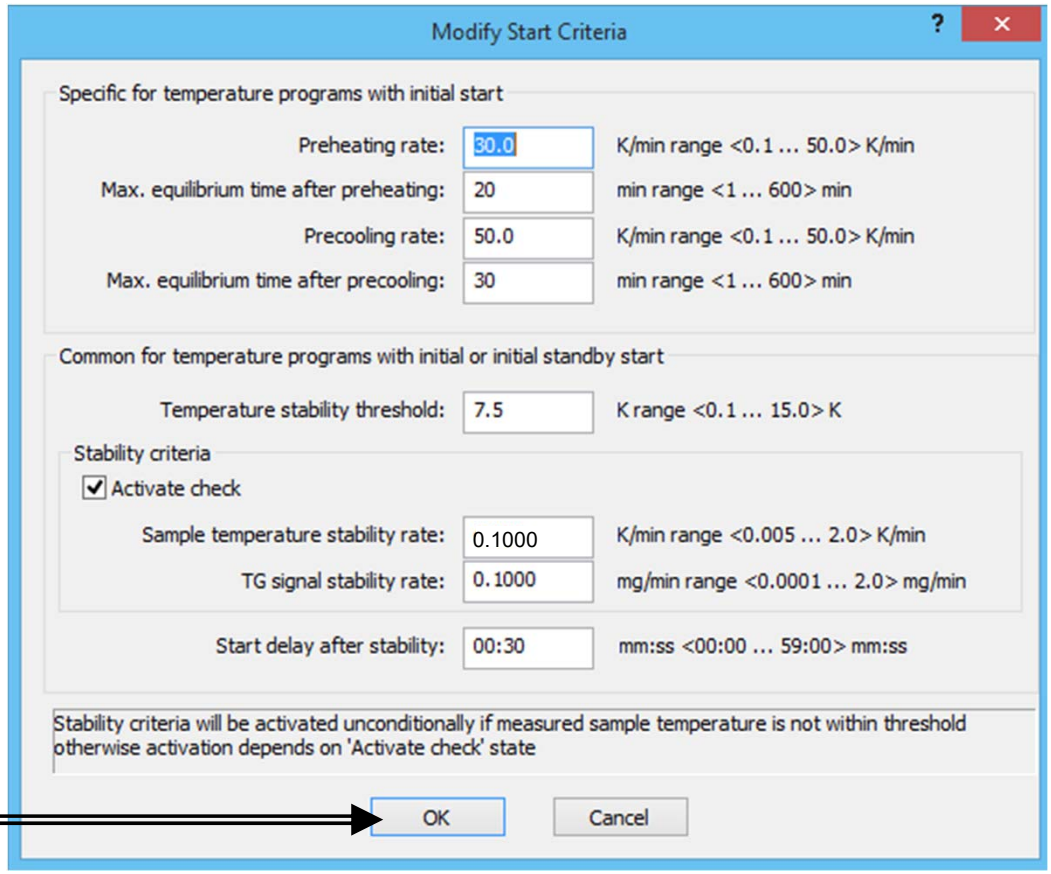
5. TG BeFlat support =
   - ***ON*** if baseline correction is to be handled by Netzsch software
   - ***OFF*** if baseline correction will be determined from an additional "baseline run"

6. Super-Res = ***OFF***

7. Click ***Modify start criteria***

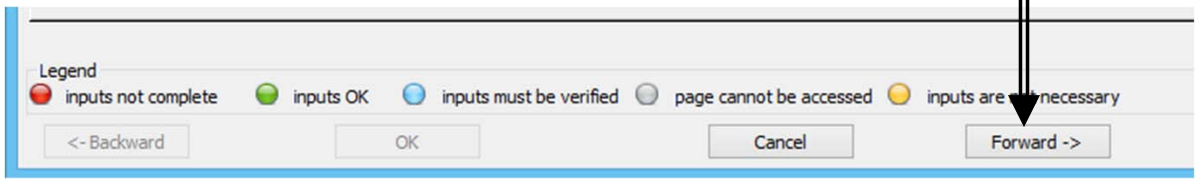
# IV. New Method – 2/9

8. Confirm the following ***Start Criteria*** default settings are appropriate:
   - **Preheating rate** = **30 K/min**
   - **Max. equilibrium time after preheating** = **20 min**
   - **Precooling rate** = **50 K/min**
   - **Max. equilibrium time after precooling** = **30 min**
   - **Temperature stability threshold** = **7.5 K**
   - **Activate check** is **checked off**
   - **Sample temperature stability rate** = **0.1 K/min**
   - **TG signal stability rate** = **0.10 mg/min**
   - **Start delay after stability** = **30 sec**



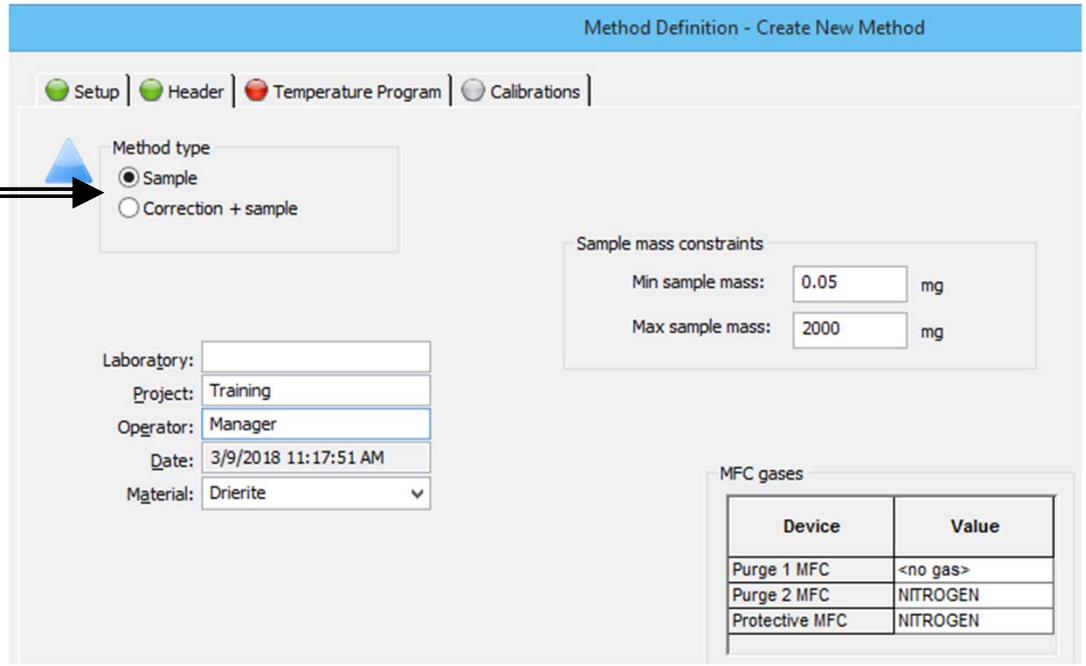

9. Click ***OK*** to accept settings

10. Click ***Forward ->*** to advance

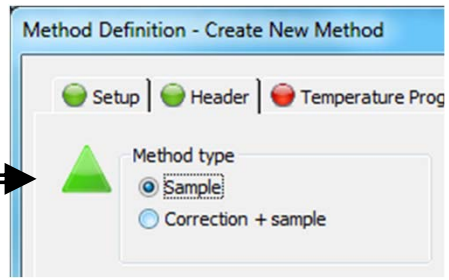


# IV. New Method – 3/9

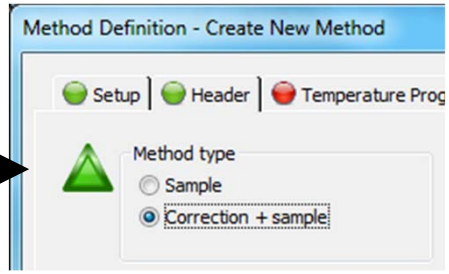
11. Select the desired ***Method*** type:



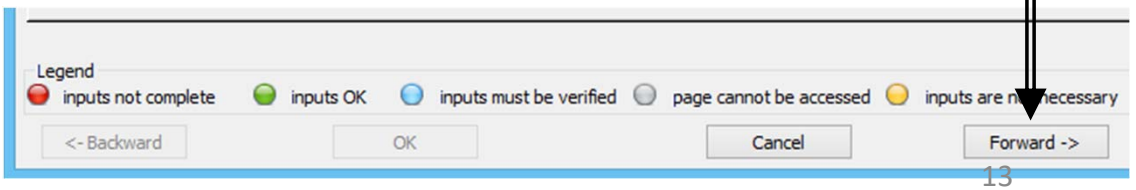

***Sample*** = Single experiment with a sample inside crucible (Default)




***Correction + sample*** = Baseline correction experiments with empty crucible **BEFORE** single experiment with sample inside crucible – recommend if mass change results are critical




12. Click ***Forward ->*** to advance



# IV. New Method – 4/9

13. It's IMPORTANT to check off the following first:

***STC** = **Sample Temp. Controller***

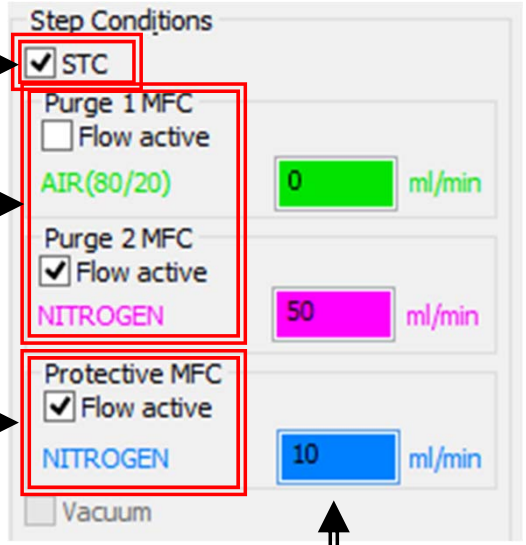
***Protective MFC** = **Nitrogen over electronics***

**+**

***Nitrogen over sample** = **Purge 2 MFC***

or

***Air over sample** = **Purge 1 MFC***




14. Enter in desired gas flowrates:

(Default) Purge 2 (or 1) Gas: **50 mL/min $N_2$ (or Air)**

(Default) Protective: **10 mL/min $N_2$**

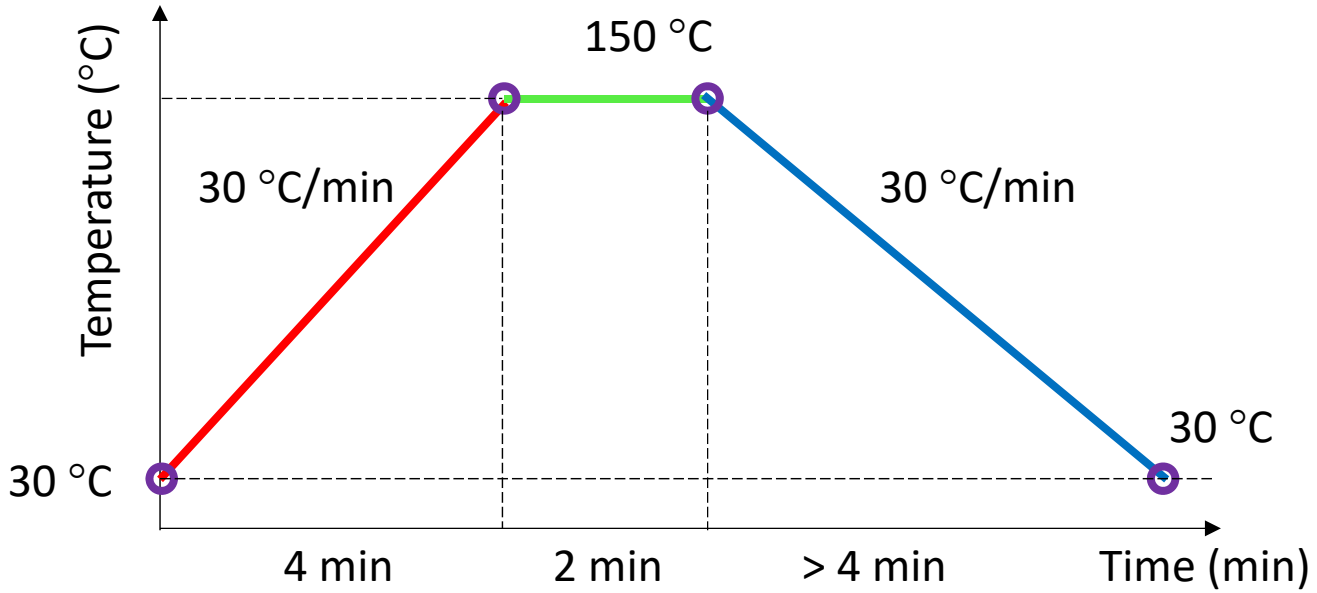
Protective must always ≥ 10 mL/min $N_2$

# IV. New Method – 5/9

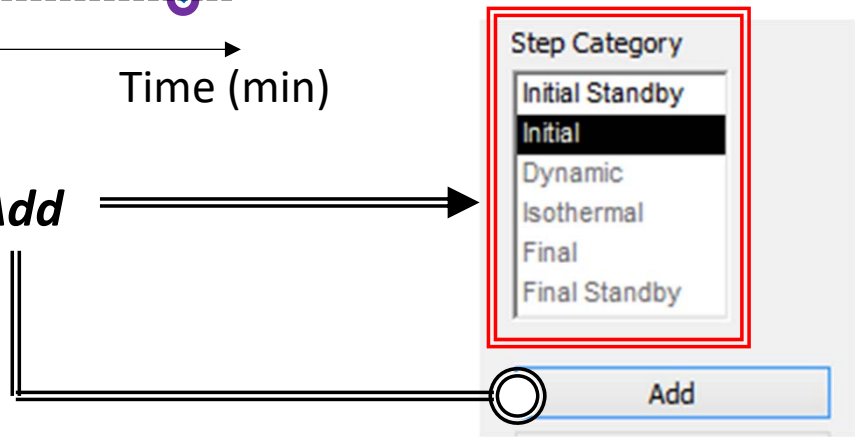
15. Build ***Temperature Program*** with desired ***Step Categories***

For NEW USERS AND TRAINING PURPOSES ONLY!

Go ahead and create the new method below with the following temperature profile

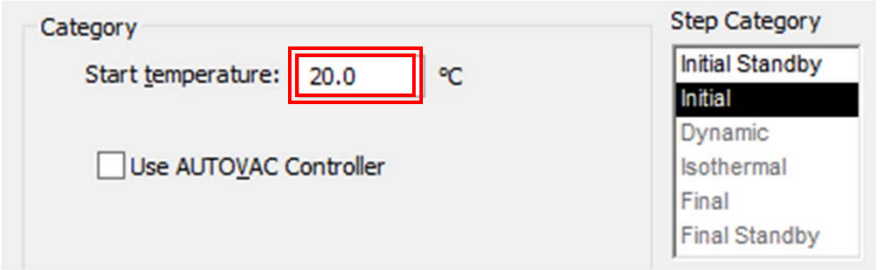



16. Select desired ***Step Categories*** and click ***Add***



# IV. New Method – 6/9

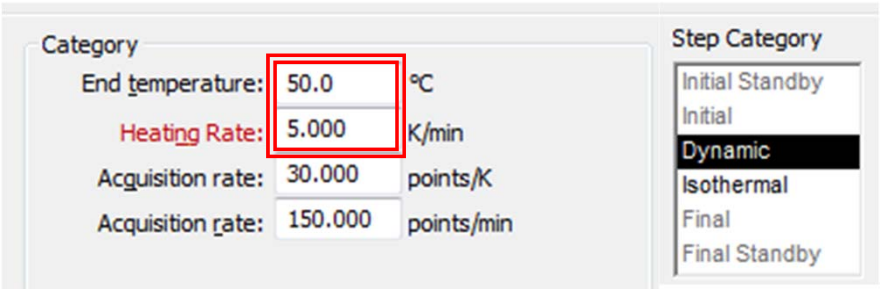
17. For ***Initial*** step (starting temp)
    a) Input Start temperature
    Recommended temp = ***25 – 30 ℃***
    b) Check Use AUTOVAC Controller if you want to remove residual oxygen from chamber

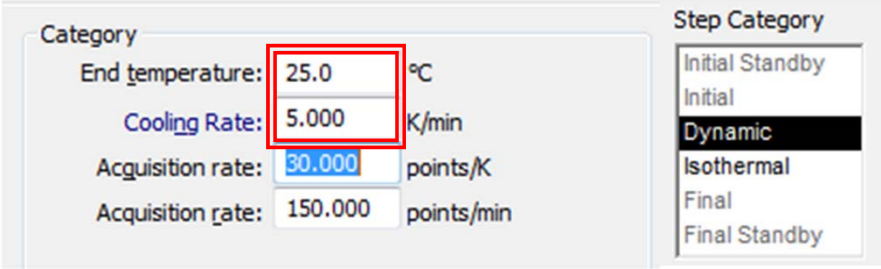



18. For ***Dynamic*** step (changing temp.)
    a) Input End temperature
    b) Input Heating Rate or Cooling Rate

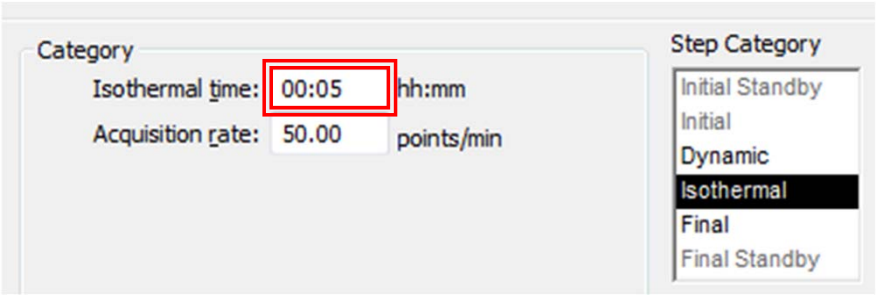
    Note: Heating Rate MUST be equal to calibrated rates

    a) Input Acquisition rate
    (default values will be automatically inserted)



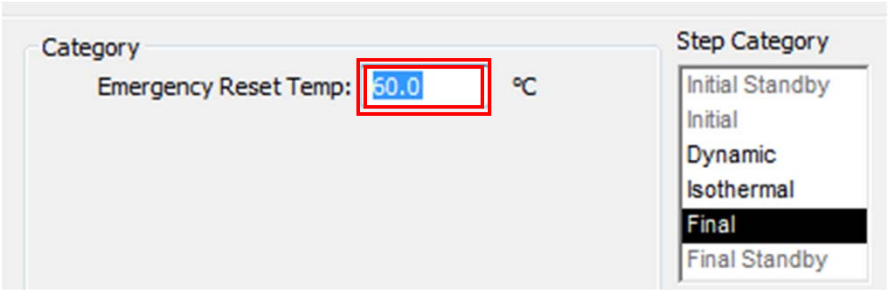




19. For ***Isothermal*** step (constant temp.)
    a) Input Isothermal time
    b) Input Acquisition rate
    (default values will be automatically inserted)



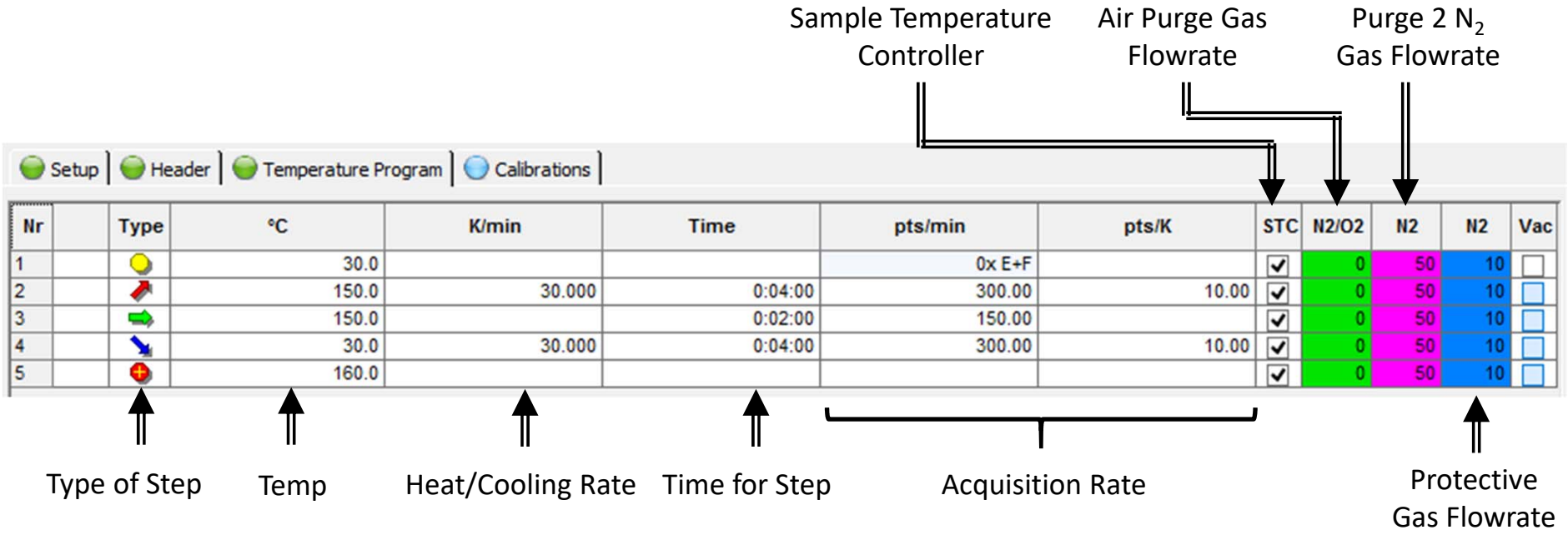
# IV. New Method – 7/9

20. Add ***Final*** step (Auto filled)
    a) Input Emergency Reset Temp (default values will be automatically inserted)



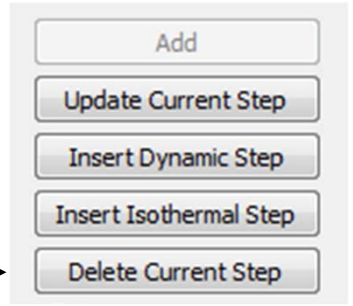

NOTE: DO NOT ENTER 60, JUST LET IT AUTO FILL!

21. Your desired ***Temperature Program*** details can be reviewed in table



# IV. New Method – 8/9

22. Modify individual steps with ***Update Current Step*** or add new steps by ***Insert Dynamic Step***, ***Insert Isothermal Step***, or remove unwanted steps with ***Delete Current Step***




23. Click ***Forward ->*** to advance

24. Select ***Will be used*** for ***Temperature calibration***

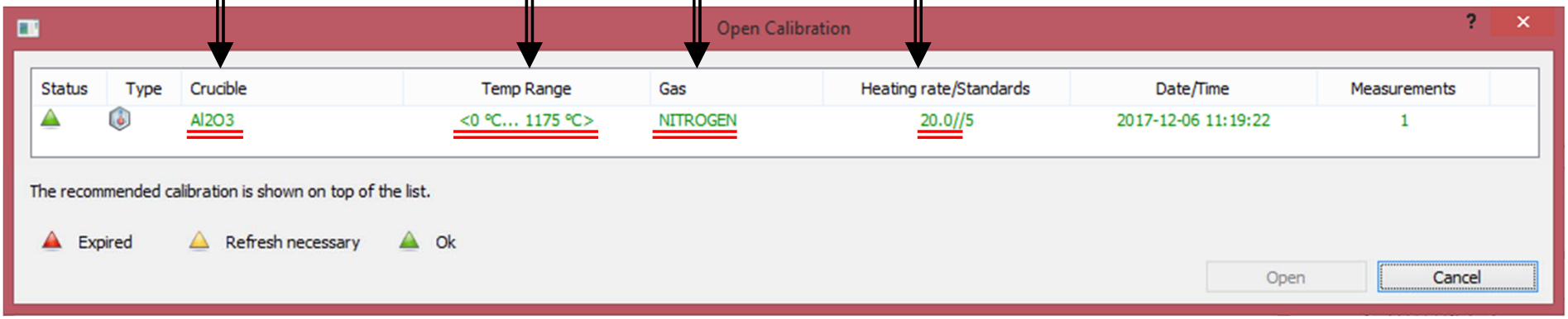

25. Select correct ***Calibration File***, checking **EVERY** condition is correct:

***1) Crucible: Al2O3***

***2) Temp Range: 0 ℃ …1175 °C***

***3) Gas: NITROGEN or AIR***

***4) Heating rate: 5, 10, or 20 K/min***



# IV. New Method – 9/9

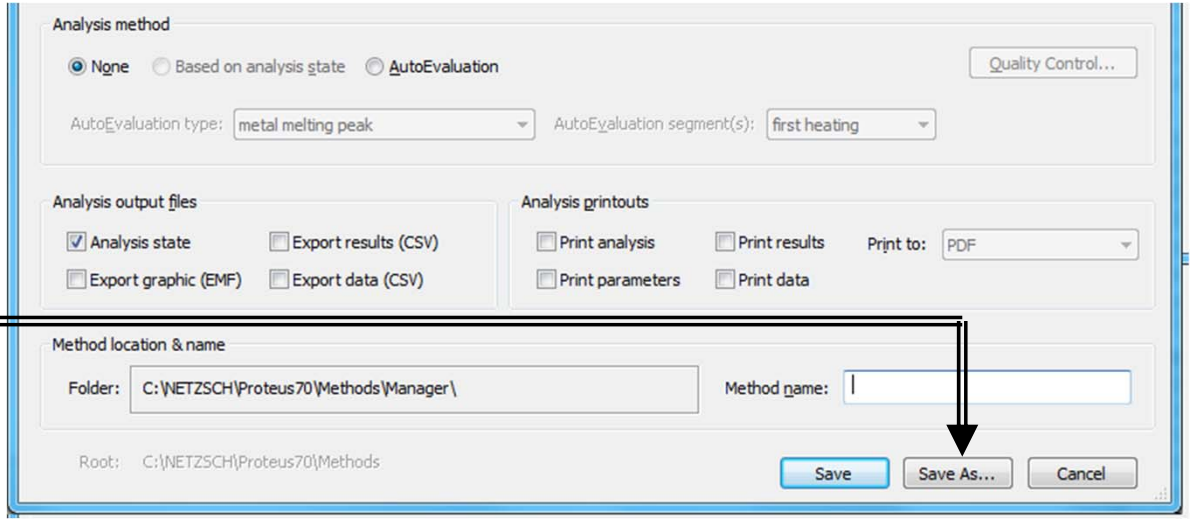
26. Click ***Forward ->*** to advance

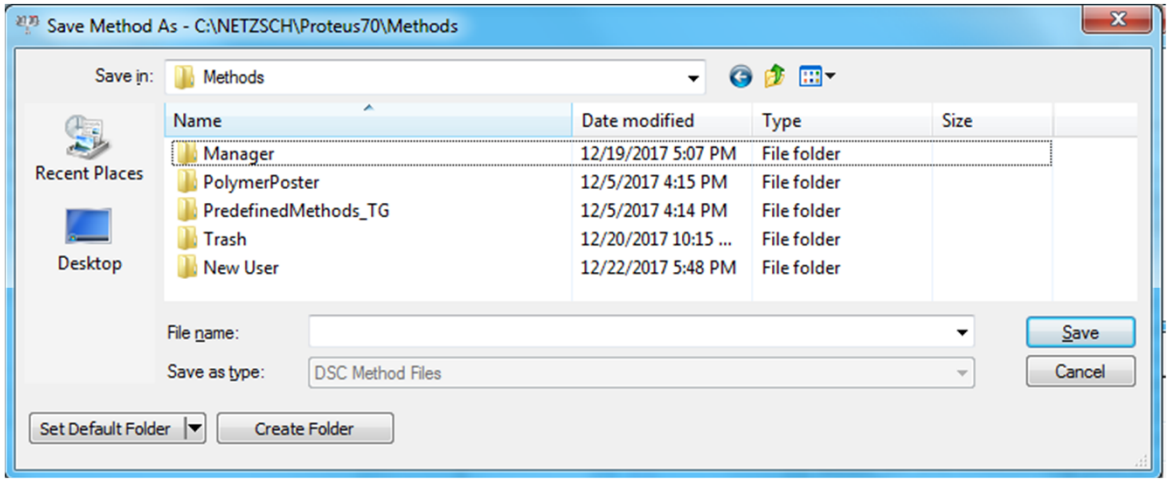
27. Click ***Save As…*** to save Method into desired folder under ***Methods***

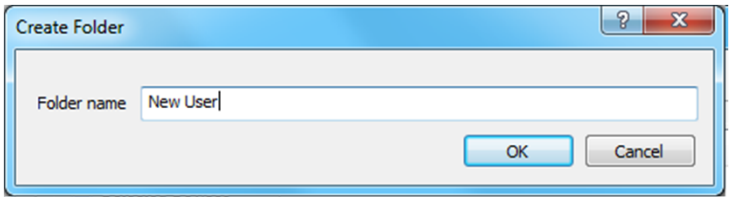
28. Choose or ***Create Folder*** with your ***PI's name*** if you are a new user

29. Create a ***New Folder*** with your user name if you are a new user

30. Click ***Save***



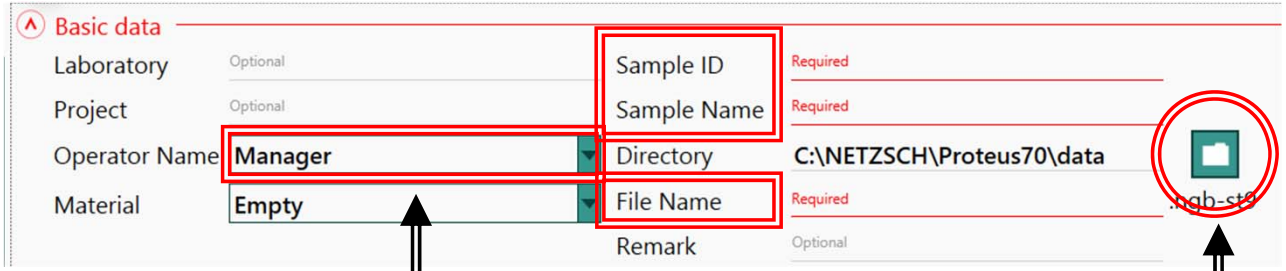
# V. Opening Method – 1/3

1. Click ***User Methods*** if desired method already exists


2. Select desired method under Methods Folder:
   C:\Netzsch\Proteus70\Methods\"***PI NAME"\"YOUR FOLDER***"

3. Enter ***Required Information*** such as:

   ***Sample ID***
   ***Sample Name***
   ***File Name***

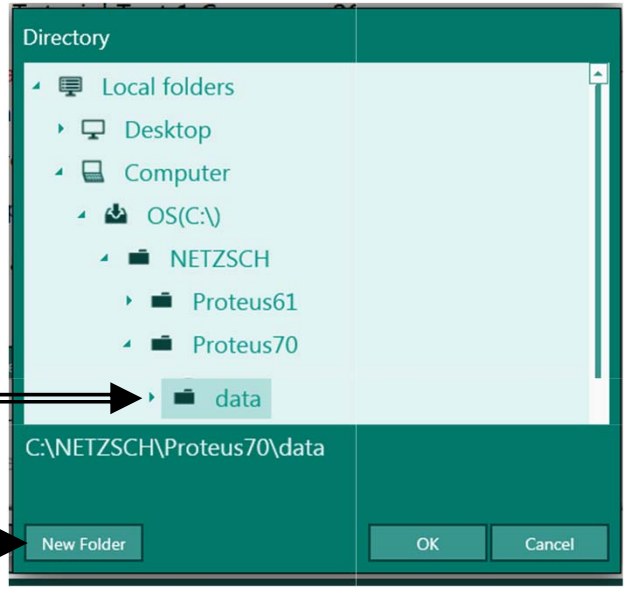



4. Enter ***Operator Name*** with your ***User Name*** for reference

5. Click on ***Folder Icon*** to store data in your designated folder (IMPORTANT)

6. Choose your ***PI Name*** and ***User Data Folder*** by clicking on ***"data"***

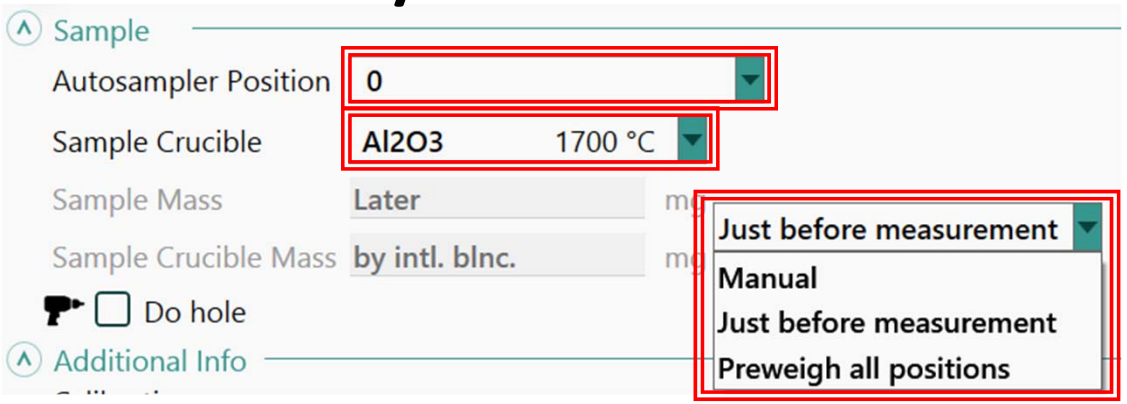
   or create ***New Folder*** and enter ***PI Name*** and ***Username***



# V. Opening Method – 2/3

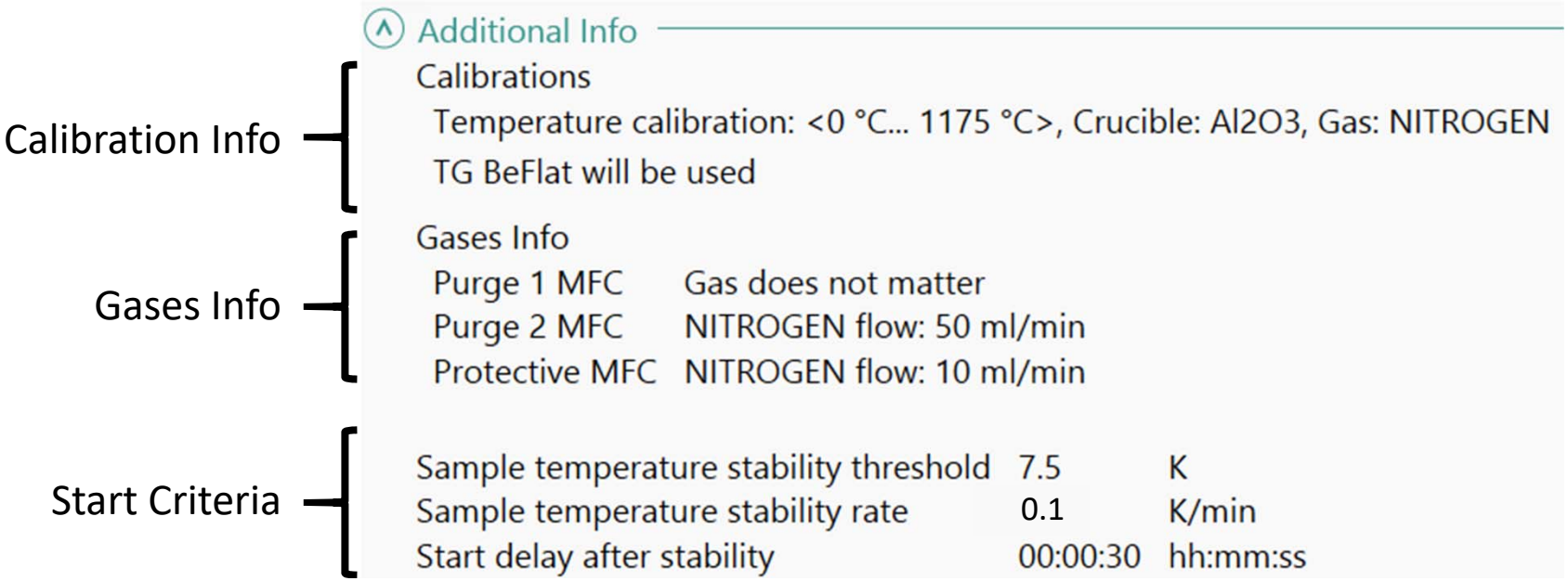
7. Select ***Autosampler Position*** for your sample

8. Select type of ***Sample Crucible:*** (Default = ***Al2O3 1700 ℃***)



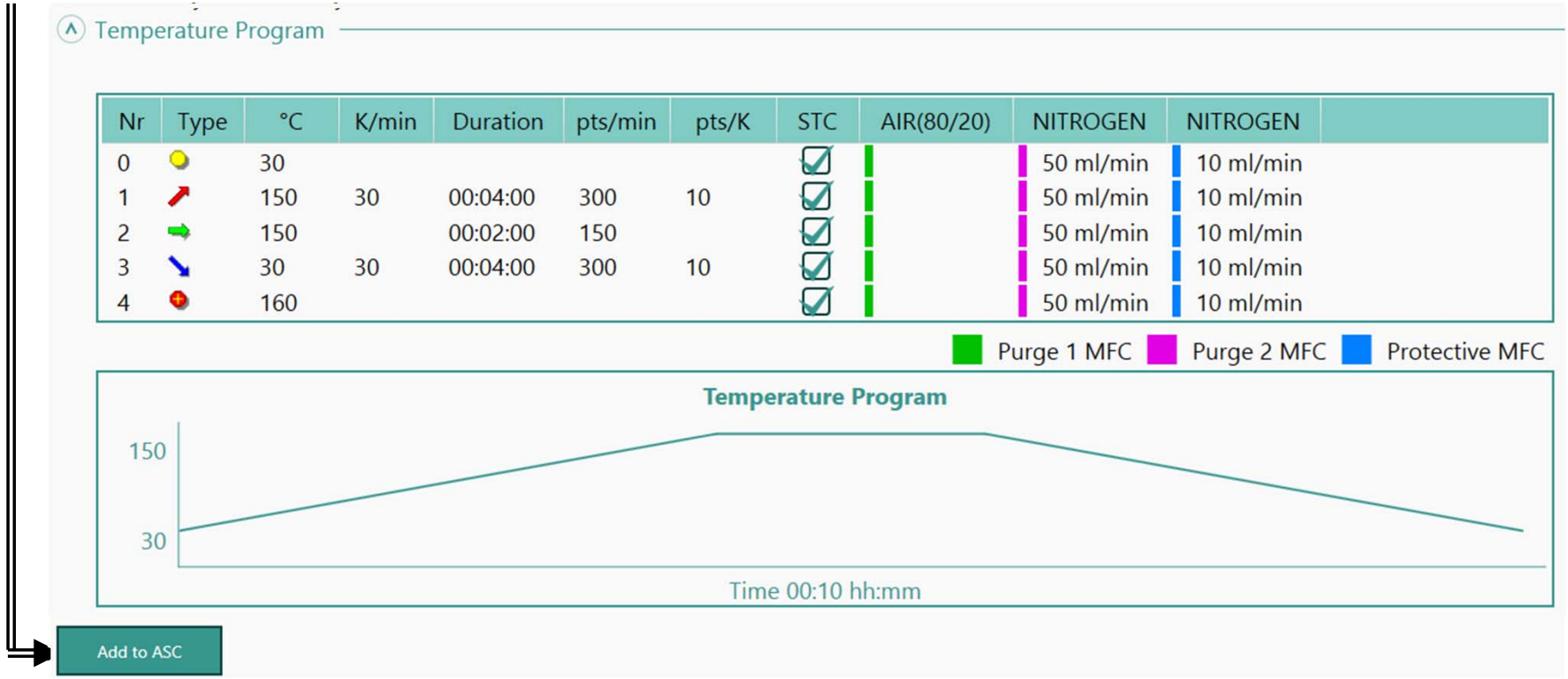

9. Select when your ***Sample Mass*** is weighed: ***Just before measurement*** (default)

10. Review that the following are correct for your desired ***Method***:



# V. Opening Method – 3/3

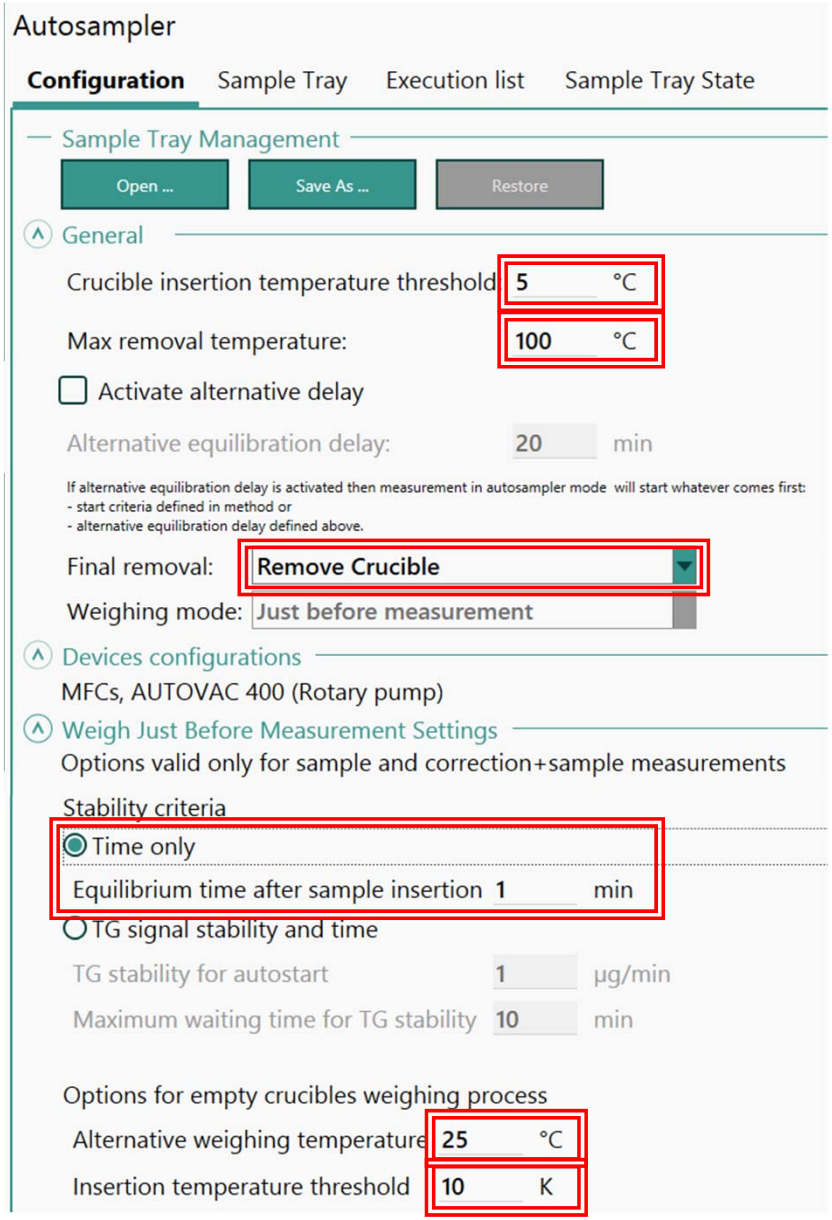
11. Review that the ***Temperature Program*** is correct for your ***Method***:
12. If everything is correct, proceed to Add Method to the ASC queue by clicking ***Add to ASC*** at the bottom



# VI. ASC Manager – 1/2

1. Click on ***ASC Manager*** and ***Switch ON*** if not already ON

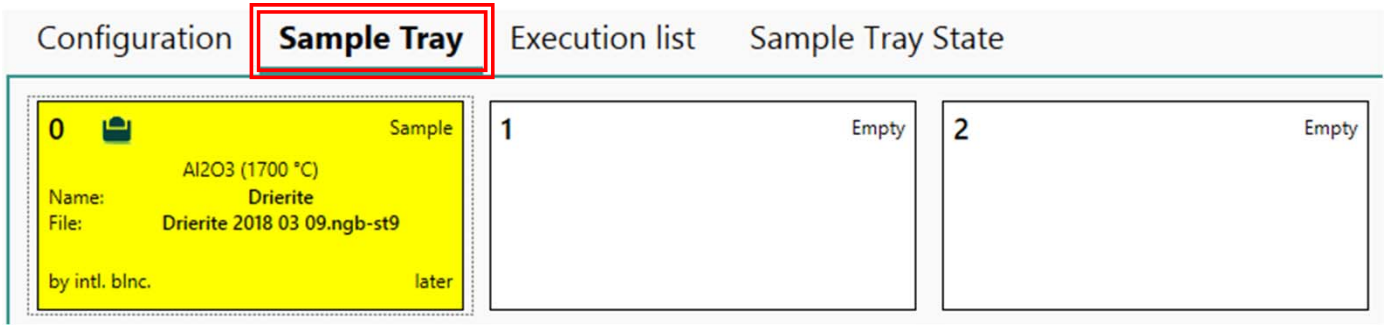

2. Confirm ***Crucible insertion temperature threshold*** (Default = ***5 °C***)
3. Confirm ***Max removal temperature*** of crucible (Default = ***100 °C***)
4. Pick ***Final removal*** action for your last sample: (Default = ***Remove Crucible***)
5. Pick Stability criteria before experiment starts: (Default: ***Time only; 1 min***)
6. Confirm ***Alternative weighing temperatures*** (Default = ***25 °C***)
7. Confirm ***Insertion temperature threshold*** (Default = ***10 °K***)



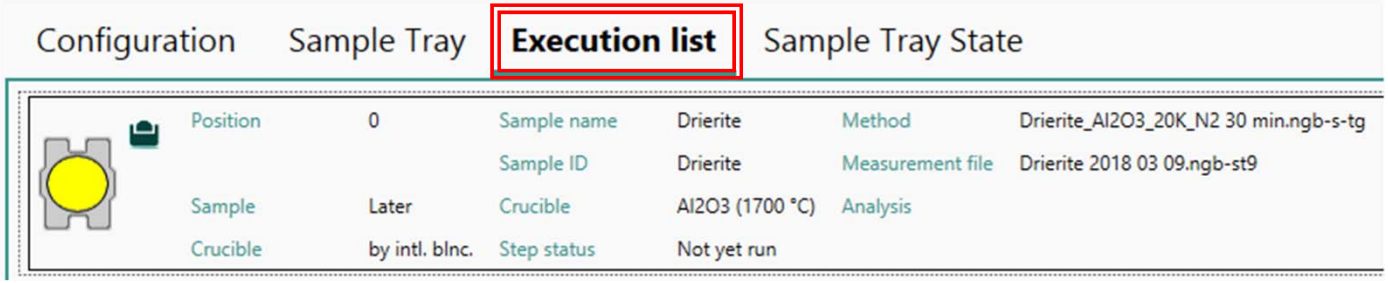
# VI. ASC Manager – 2/2

8. Click on ***Sample Tray*** tab under ***ASC Manager*** to review the Positions and status

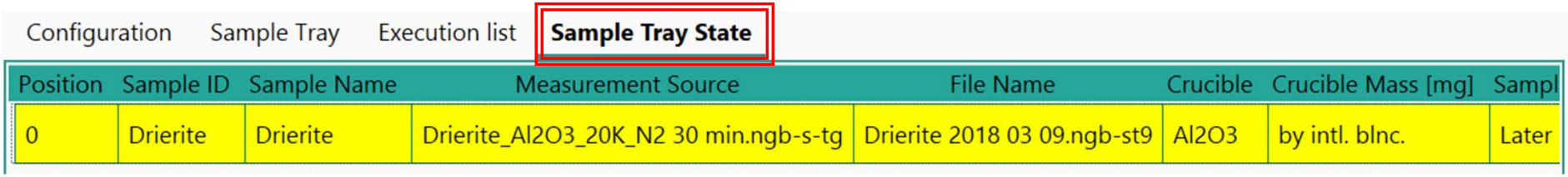
| | |
|---|---|
| | Defined |
| | Done |
| | Done (analysis failed) |
| | Failed |
| | Measurement Active |
| | Reference |




9. Click on ***Execution list*** tab to review the order of experiments scheduled

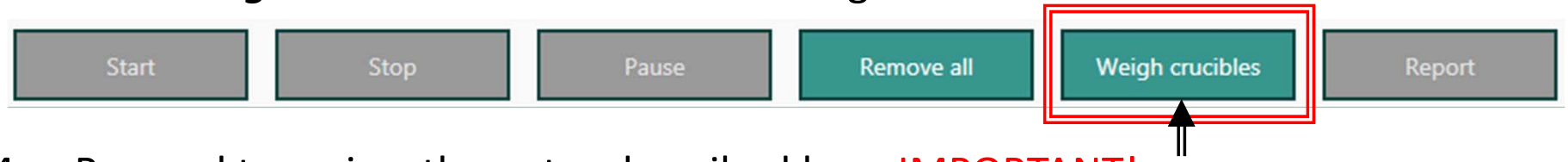



10. Click ***Sample Tray State*** to review details of experiments scheduled

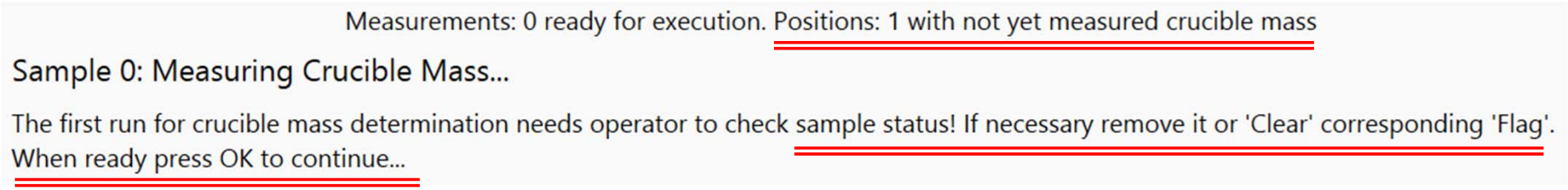


# VII. Running Experiments – 1/5

1. Review ***ASC Manager*** settings and confirm all is correct
2. Ensure that ALL crucibles to be run **ARE EMPTY!**
3. Click ***Weigh crucibles*** to have the ASC weigh ALL EMPTY CRUCIBLES NOW

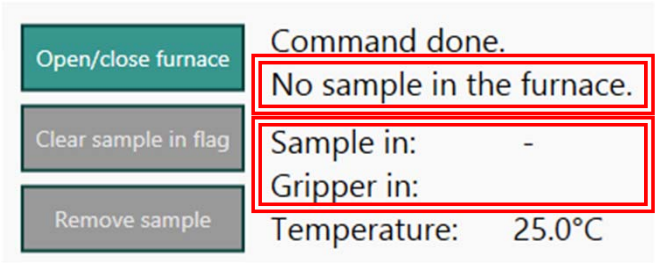



4. Proceed to review the notes described here IMPORTANT!




5. Check if the conditions of the furnace are correct BEFORE proceeding

E.g. “Not correct sample in the furnace. Remove it first”




Things to consider:

- Is the furnace open or closed?
- Are the crucibles ALL empty?
- Is there a sample inside furnace?

# VII. Running Experiments – 2/5

6. Correct **ALL** issues before proceeding using the various ***buttons*** on the left


- Click to “open” or “close” furnace to check what is inside…
- Click **ONLY IF** there is **NO** sample in the furnace
- Click to “remove” the current sample

7. After all flags and conditions are corrected, click ***OK*** to begin experiments

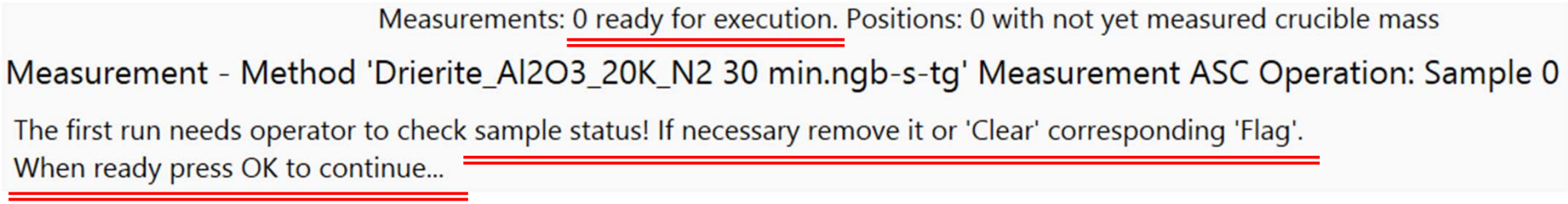

8. Follow any instructions or prompts that appear

9. A prompt will appear when ***Empty Crucible Weighing*** is completed, click ***OK***

# VII. Running Experiments – 3/5

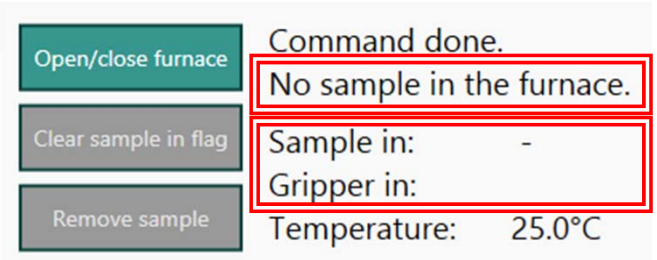
10. Proceed to FILL IN YOUR SAMPLES into the EMPTY CRUCIBLES now
11. Click ***Start*** at bottom of screen when ready to begin experiments


12. Proceed to review the notes described here IMPORTANT!




13. Check if the conditions of the furnace are correct BEFORE proceeding

E.g. “Not correct sample in the furnace. Remove it first”




Things to consider:

- Is the furnace open or closed?
- Are the samples insides the crucibles now?
- Is there a sample inside furnace?

# VII. Running Experiments – 4/5

14. Correct **ALL** issues before proceeding using the various ***buttons*** on the left

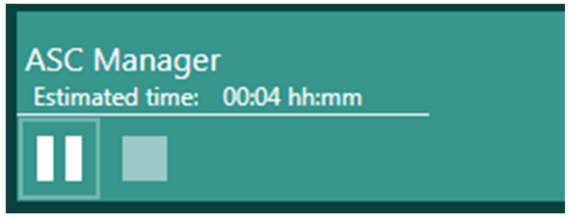

- Click to “open” or “close” furnace to check what is inside…
- Click **ONLY IF** there is **NO** sample in the furnace
- Click to “remove” the current sample

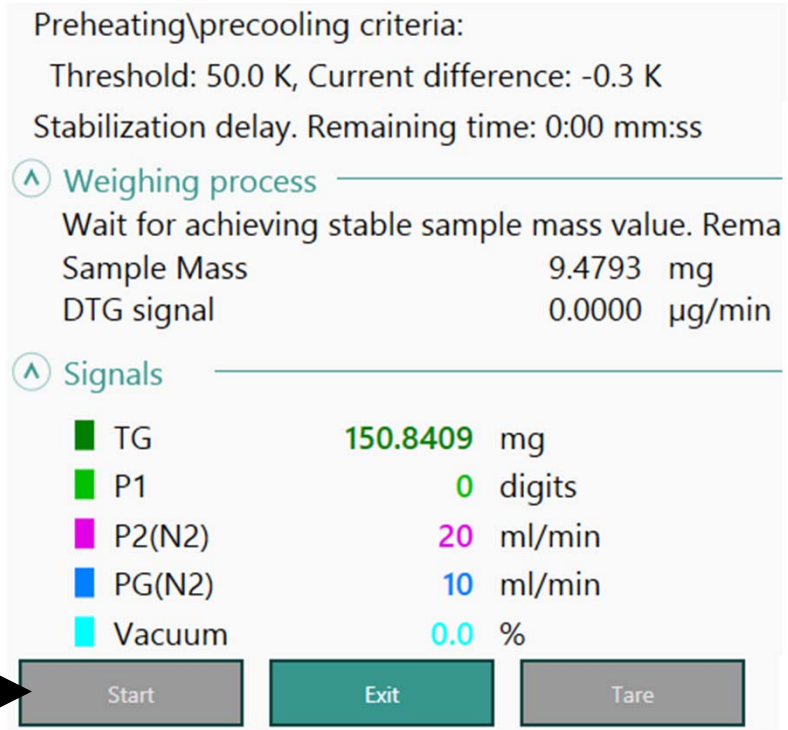
15. After all flags and conditions are corrected, click ***OK*** to begin experiments

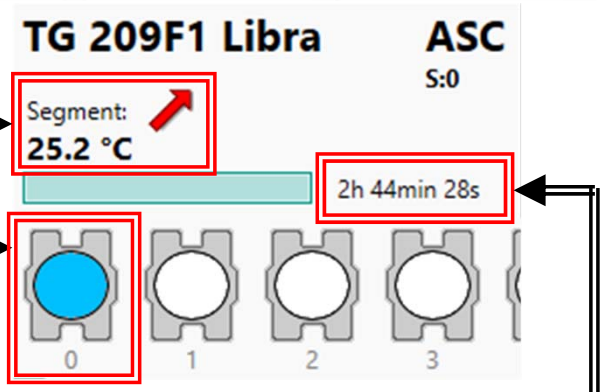
16. Follow any instructions or prompts that appear

# VII. Running Experiments – 5/5

17. TGA will now begin to preheat/precool to target Initial temperature
18. Program will automatically ***Start*** when ***Start Criteria*** conditions have been met
19. The ***Estimated time*** for all experiments are shown here
20. The ***current temperature*** and ***segment action*** is shown here
21. Active measurement can be shown here
22. Remaining ***segment time*** and ***completion %*** is shown here

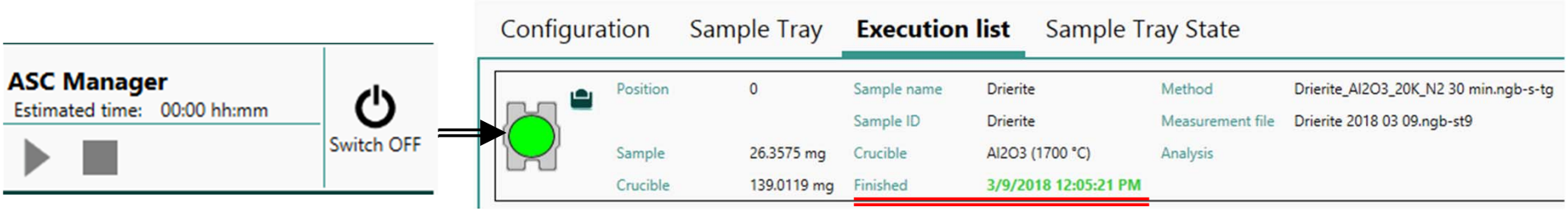


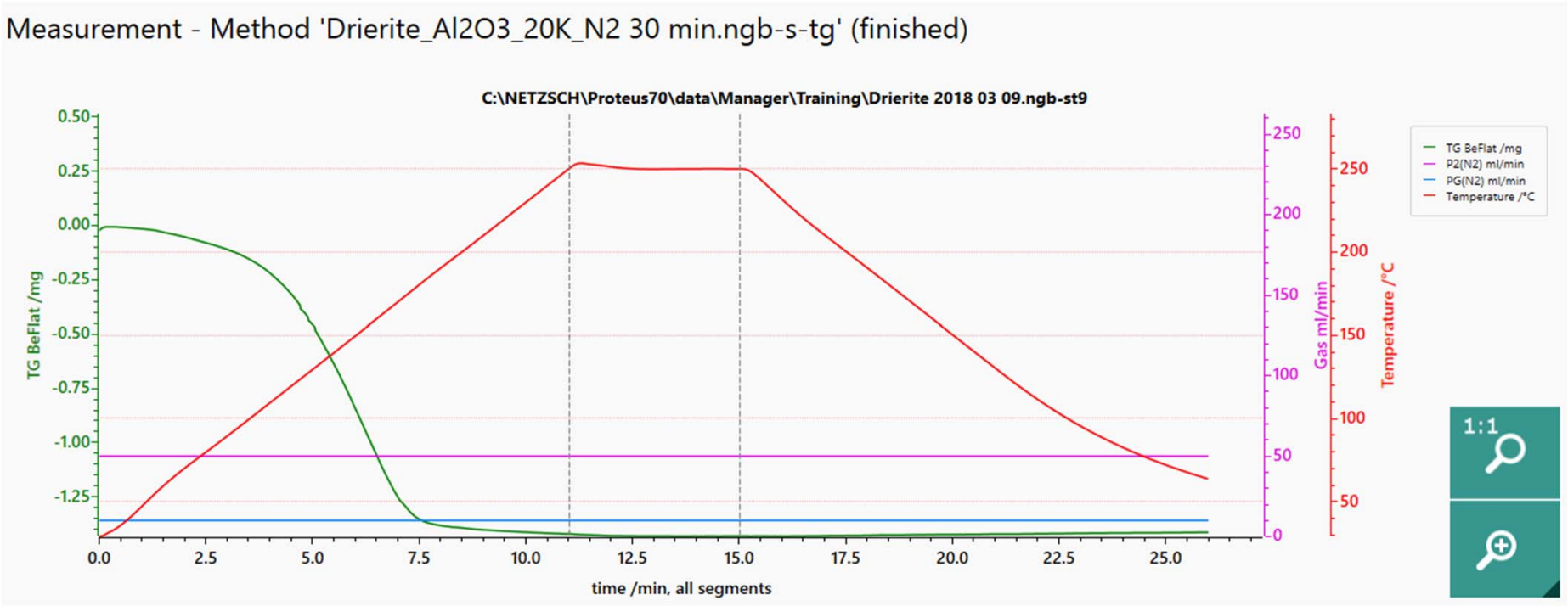



# VIII. Results – 1/1

1. Click ***ASC Manager***, and completed experiments will be updated with Green color code and completion date and time




2. Click ***Measurement***, to show the results

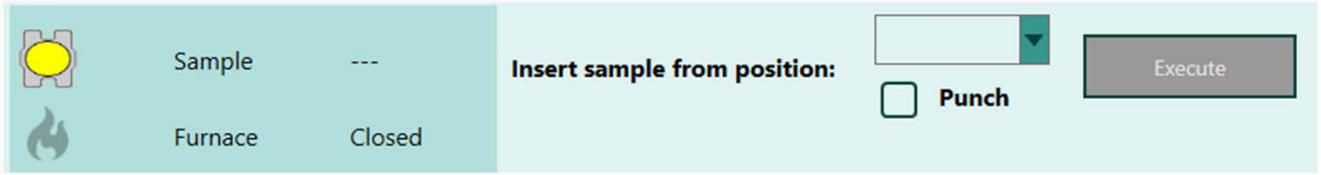
# IX. Clean Up – 1/1

1. After experiment(s), TGA should cool itself down to 25°C via ***Idle Mode*** or ***Eco Mode*** at top of the screen
2. If ***Idle Mode*** or ***Eco Mode*** is not activated, proceed to turn back on ***Setpoint*** first by clicking ***Apply***
3. Click on ***Setup & Control***
4. Click on ***ASC Manual Control***
5. Check that the following are true, else correct:

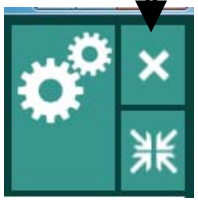
- ***No Sample***
- ***Furnace is Closed***



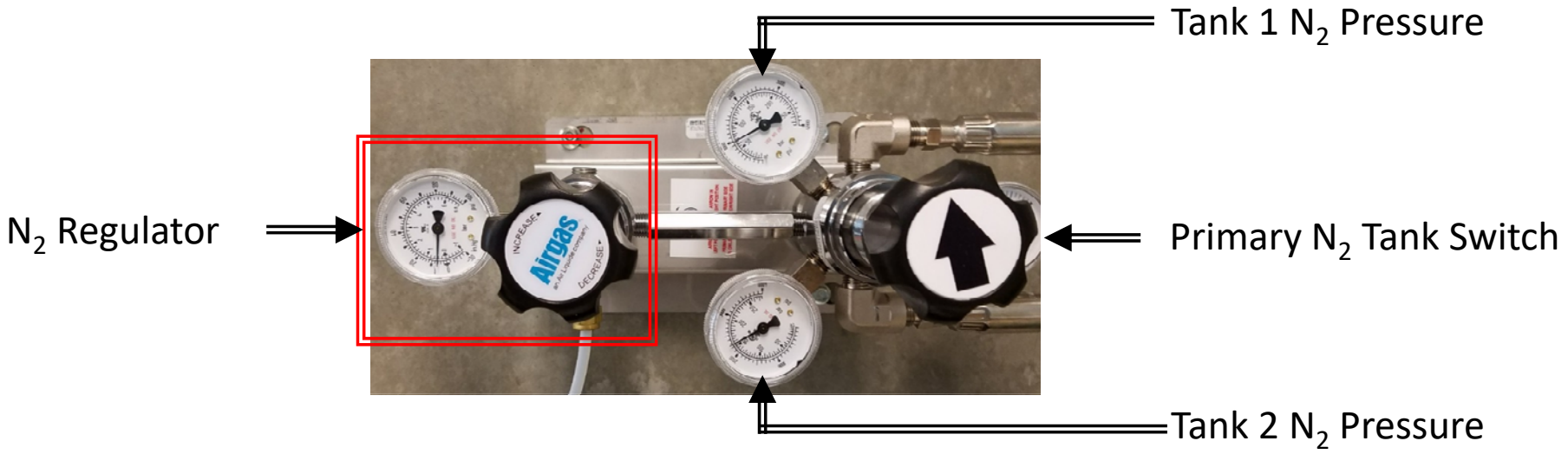





6. When TGA temperature reaches ***25 ± 5 ℃***, click the ***X*** to close the software
7. Confirm that you wish to ***keep the Setpoint ON*** after you close software
8. ***Log out*** of your ENGR account
9. Clean up the lab bench and place all items back in their respective drawers
10. Sign out on the ***Sign-In Sheet*** before leaving

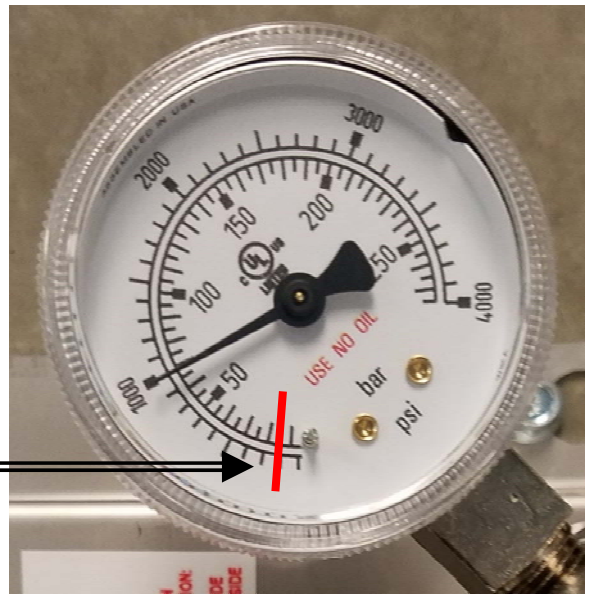


# X. Red Flags & Mistakes – 1/3



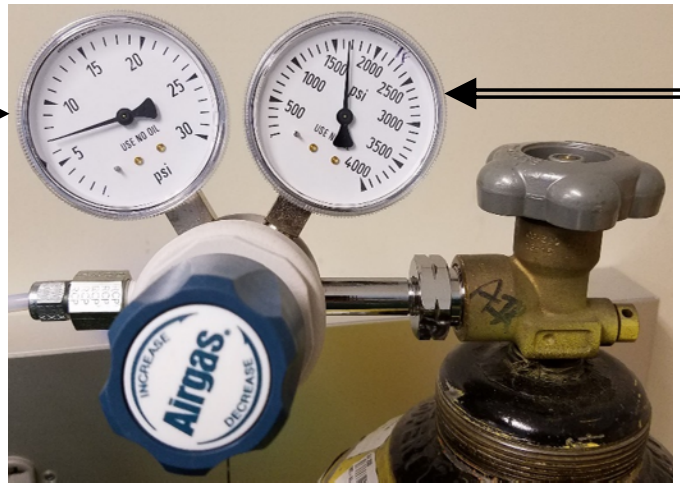

1. DO NOT ADJUST THE REGULATOR AS THIS MAY DAMAGE MASS FLOW CONTROLLERS $$$

2. Check if Tank 1 or 2 $N_2$ pressure is at least **200 psi**, else contact Lab Manager to replace tank



# XI. Red Flags & Mistakes – 2/3

Regulated Air Pressure ≈ 7 psi



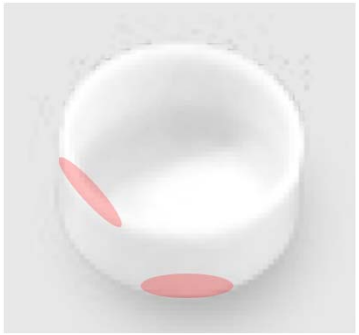

Air Tank Pressure

3. DO NOT ADJUST THE REGULATOR AS THIS MAY DAMAGE MASS FLOW CONTROLLERS $$$

4. Check if Air Tank pressure is at least **200 psi**, else contact Lab Manager to replace tank

# X. Red Flags & Mistakes – 3/3

5. Remove any trace of sample on outside and underneath crucible, as it will contaminate the TGA thermocouple $$$



TGA thermocouple

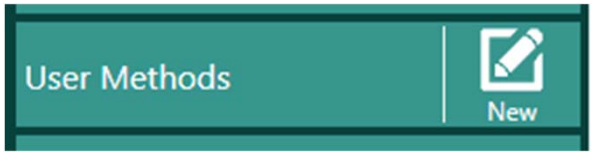
6. Avoid over-filling the crucible in case the sample boils and bubbles over contaminating TGA thermocouple $$$

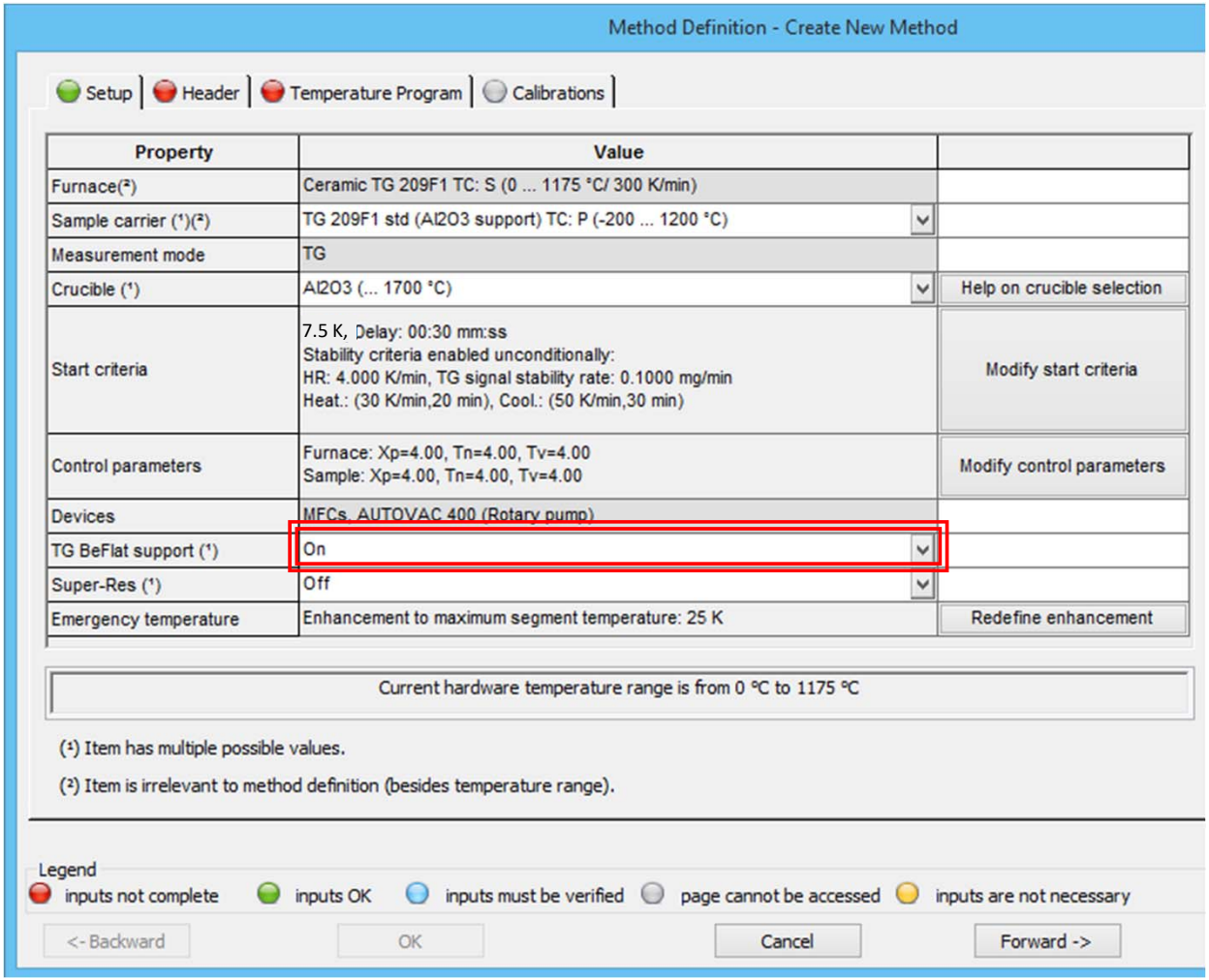
7. If ASC sample changer makes a noise while switching samples, report to Lab Manager immediately!

# XI. Baseline Corrections – 1/6

The following are modifications to perform a baseline correction

1. Click on New next to User Methods



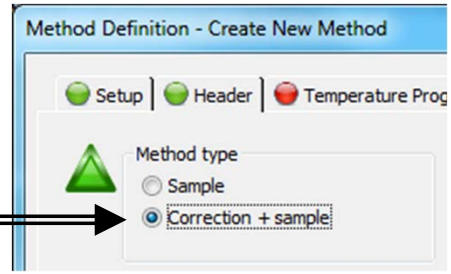

2. Everything else is kept the same as before except…

3. TG BeFlat support =
   - ***OFF*** if baseline correction will be determined from an additional “baseline run”

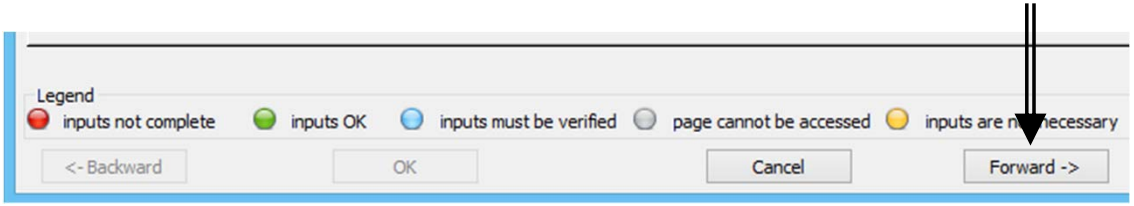
4. Click ***Forward ->*** to advance

# XI. Baseline Corrections – 2/6

5. Select the ***Method*** type = ***Correction + sample***




6. Click ***Forward ->*** to advance




7. Complete the same ***Temperature Program*** as before…

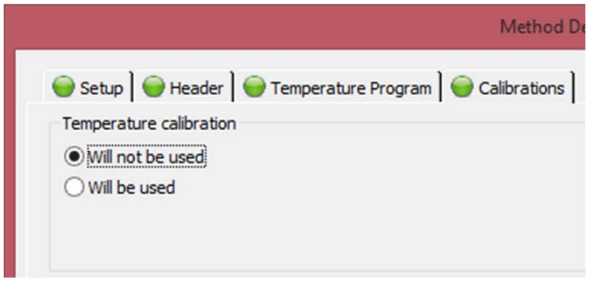
Setup | Header | Temperature Program | Calibrations

| Nr | | Type | °C | K/min | Time | pts/min | pts/K | STC | N2 | N2 | Vac |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | | 30.0 | | | 0x E+F | | ☑ | 50 | 10 | ☐ |
| 2 | | | 250.0 | 20.000 | 0:11:00 | 300.00 | 15.00 | ☑ | 50 | 10 | |
| 3 | | | 250.0 | | 0:04:00 | 75.00 | | ☑ | 50 | 10 | |
| 4 | | | 30.0 | 20.000 | 0:11:00 | 300.00 | 15.00 | ☑ | 50 | 10 | |
| 5 | | | 275.0 | | | | | ☑ | 50 | 10 | |

8. Click ***Forward ->*** to advance

9. Select the same ***Temperature calibration*** as before…

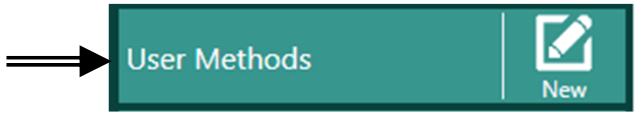
10. Click ***Forward ->*** to advance




11. Proceed to ***Save*** the file as before…

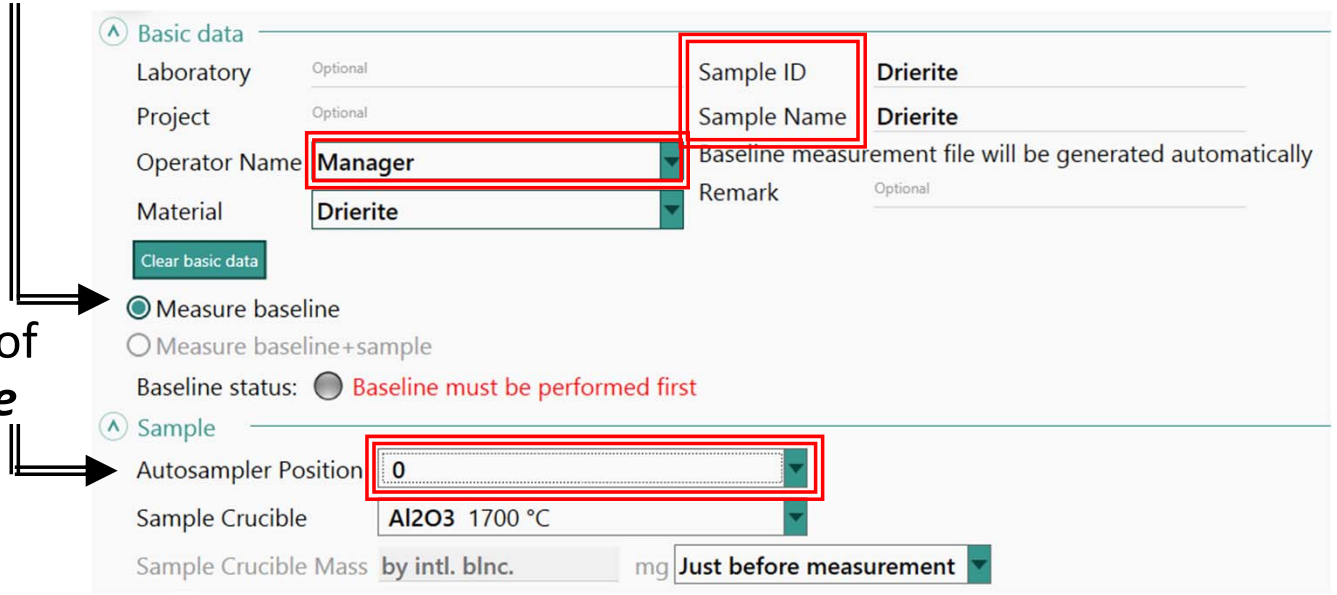
# XI. Baseline Corrections – 3/6

12. Click ***User Methods*** and select your ***Correction + sample*** method

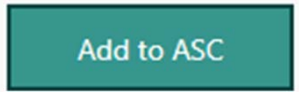



13. You will have to perform a baseline measurement first (unless you have already ran it)

14. Select the position of your ***Empty Crucible***

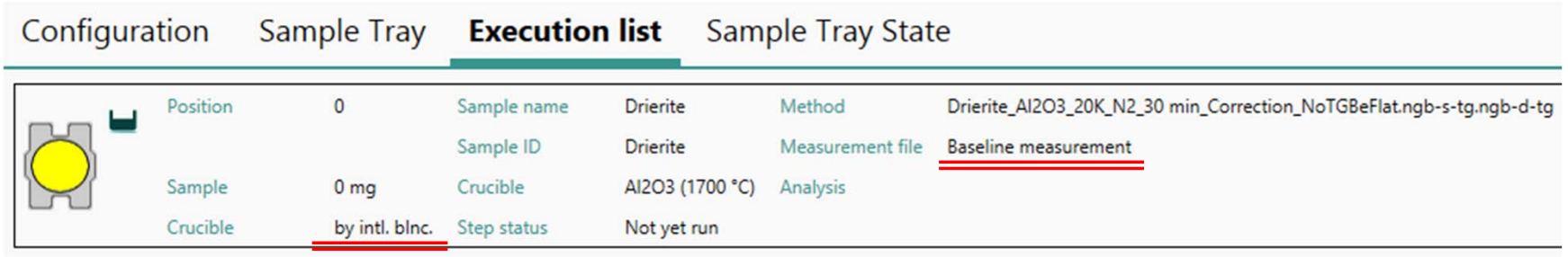



15. Add to ASC at the bottom

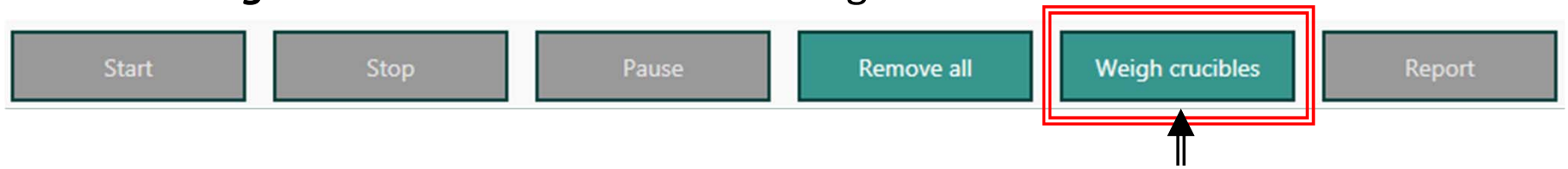


# XI. Baseline Corrections – 4/6

16. Ensure your next measurement is a ***Baseline measurement***

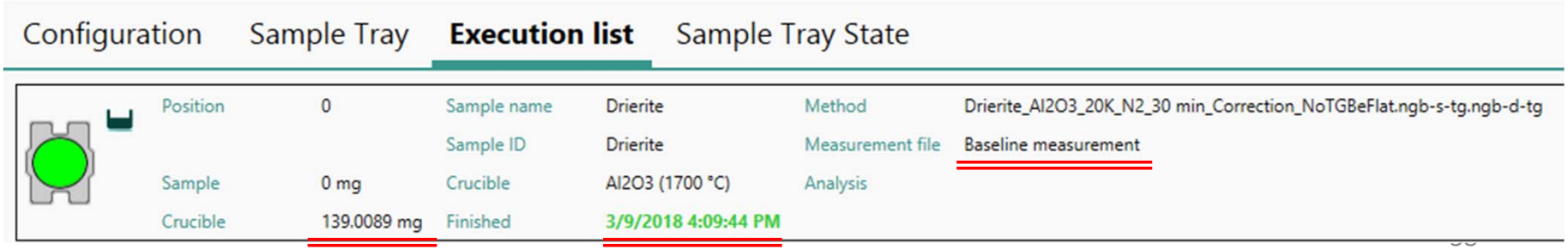



17. Click ***Weigh crucibles*** to have the ASC weigh ALL EMPTY CRUCIBLES NOW



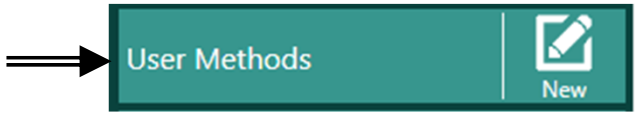

18. Proceed to review the notes and correct all issues as before… Click ***OK***

19. After the Empty Crucible is weighed, click ***Start*** to perform the ***Baseline measurement…***



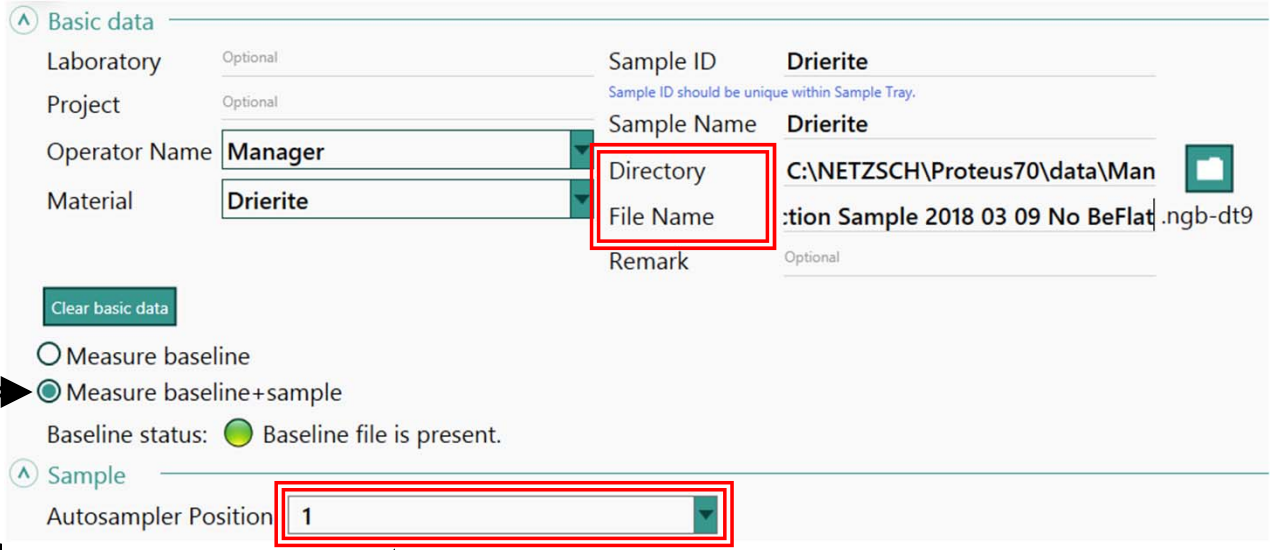
# XI. Baseline Corrections – 5/6

20. Click ***User Methods*** again



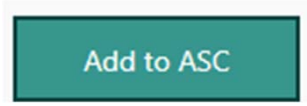

21. You can now create a ***File Name*** for your sample

22. Notice that ***Measure baseline + sample*** is now available



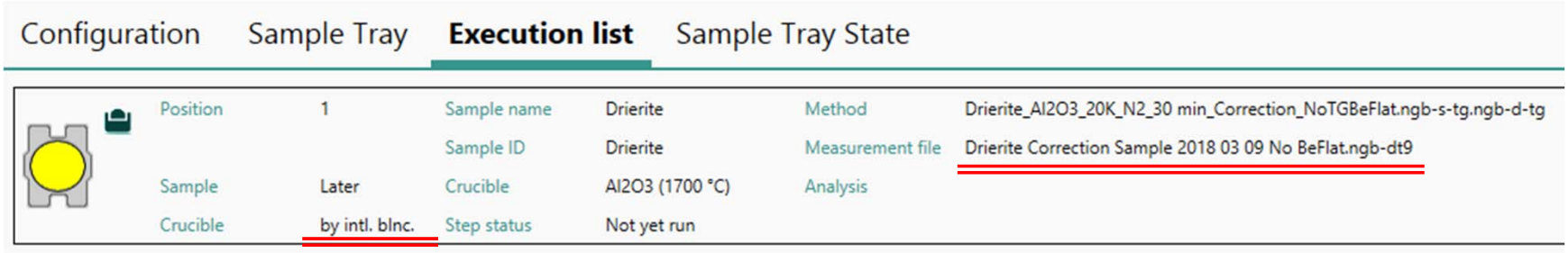

23. The new sample position is also updated to the next position automatically

24. Add to ASC at the bottom

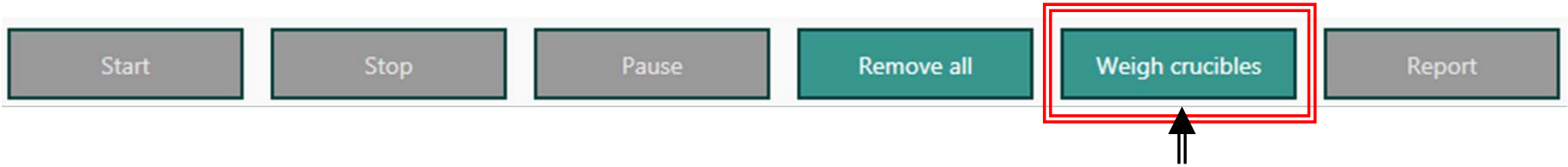


# XI. Baseline Corrections – 6/6

25. Ensure your next measurement is a ***sample measurement***

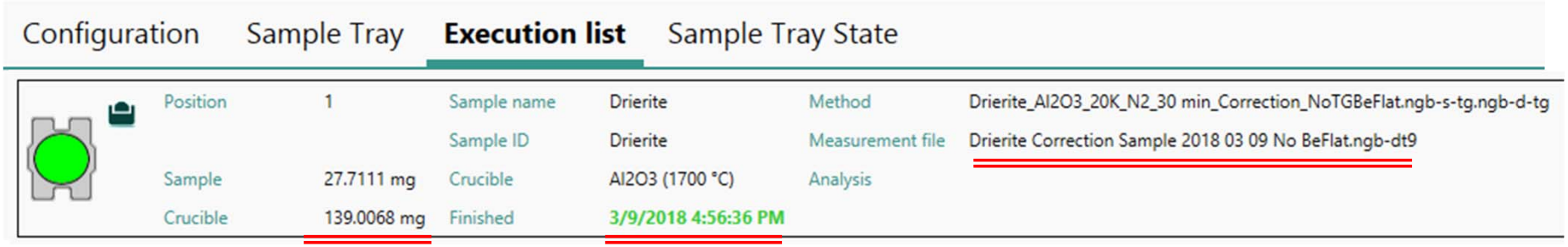



26. Click ***Weigh crucibles*** to have the ASC weigh ALL EMPTY CRUCIBLES NOW




27. Proceed to review the notes and correct all issues as before… Click ***OK***

28. After the Empty Crucible is weighed, click ***Start*** to perform the ***sample measurement…***



# XII. Troubleshooting – 1/1

1. If the ASC sample changer fails to place your crucible back to the tray when experiment is completed, perform the following:
   a) Select "***Stop ASC Sample Tray run***"
   b) Click ***OK***
   c) Press ***Stop*** at top of TGA
   d) Press ***Init*** at top of TGA
   e) The ASC gripper will now drop your crucible
   **f)** **CAREFULLY** grab your crucible with tweezers underneath gripper and place it back in your tray
   g) Avoid spilling sample while moving your crucible
   h) Continue with experiments