Netzsch DSC Training Notebook

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Materials Science and Engineering
University of California, Riverside

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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 DSC Operation

I. Preparation

II. Start

III. Setup & Control

IV. New Method

V. Adding Reference

VI. Opening Method

VII. ASC Manager

VIII. Running Experiments

IX. Results

X. Cleanup

XI. Red Flags & Mistakes

XII. Baseline Corrections

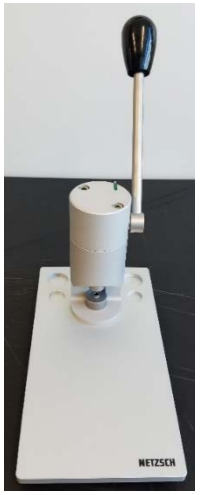
XIII. C_p Measurement: Sapphire Method – coming soon

I. Preparation – 1/3

1. Prior to running a DSC test, it is important to perform the following checks to plan for an efficient run and avoid damage to DSC
   - I. Acceptable ***Calibrated Pans***
     - a) Aluminum Concavus Pans with pierced lid – 5 mm diameter, 30/40 µL
   - II. Acceptable ***Calibrated Gas environments***
     - a) 100% Nitrogen = Purge 2: N2 60 mL/min + Protection: N2 40 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 ***Reference Sample*** and ***Samples*** using the ***Sealing Press***

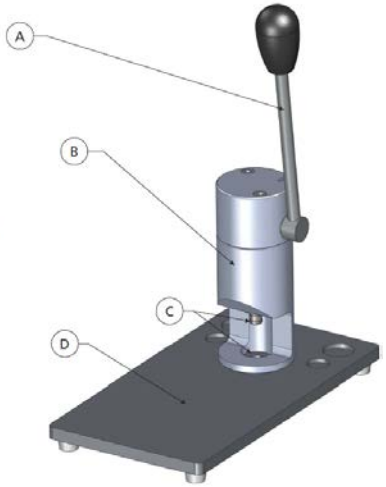


I. Preparation – 2/3

4. Only seal Netzsch Concavus Pans (***Series DSC21400A66.xxx NGB14672***)

   Using ***Sealing Press*** for any other pan will DAMAGE Press!

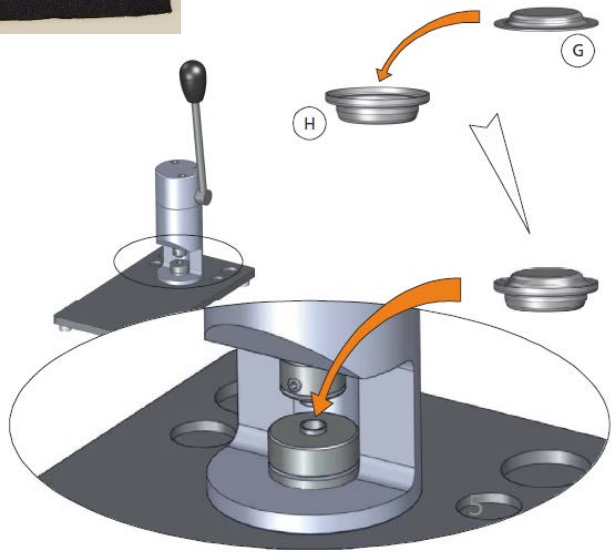
Type of crucible:



5. Punch a tiny hole onto the lid using the provided push-pin + pad

6. Place pan into the bottom part (C) of the toolkit carefully

7. Carefully place lid (G) onto the crucible (H) using tweezers

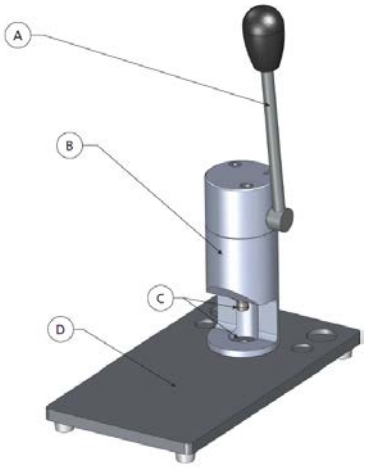


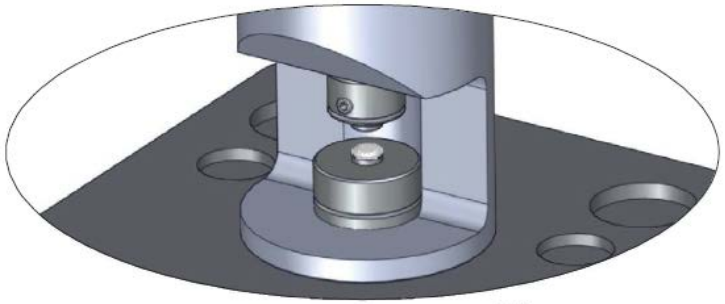
I. Preparation – 3/3

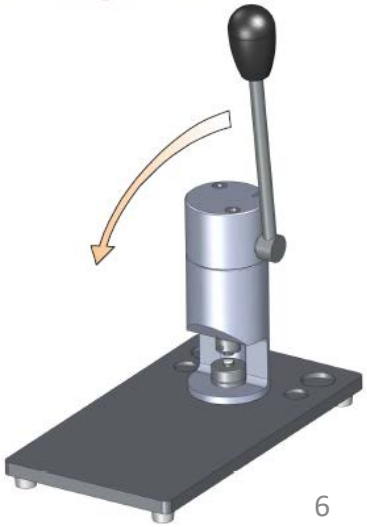
8. Press the lever down (A) with a continuous motion until the limit stop is reached

9. For pressure tight cold welding, it is important to keep the lever in the limit stop position at ***least 5 seconds***

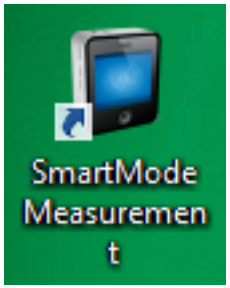
10. Release the lever and the crucible is now cold-welded





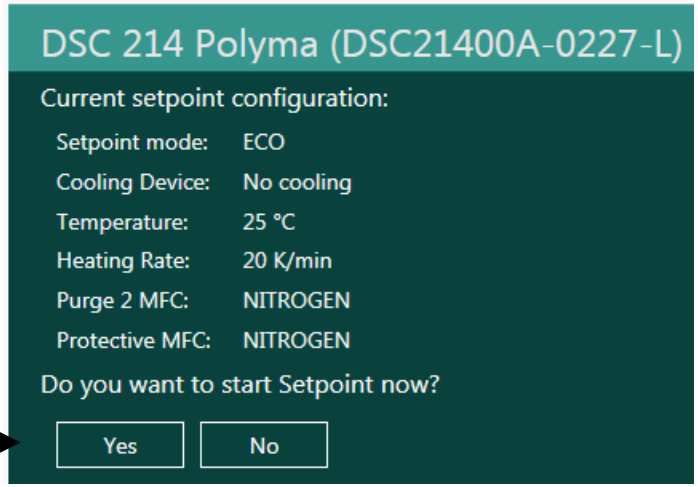


II. Start – 1/2

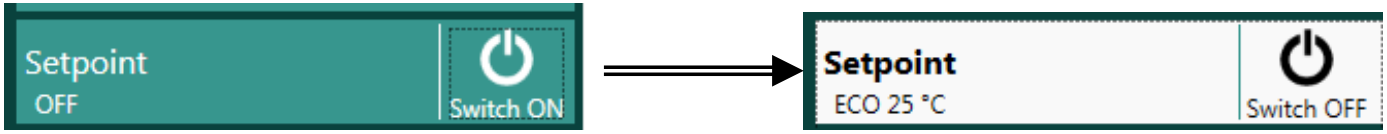


1. Click ***SmartMode Measurement*** to start DSC measurement program

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 DSC with Nitrogen!)



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

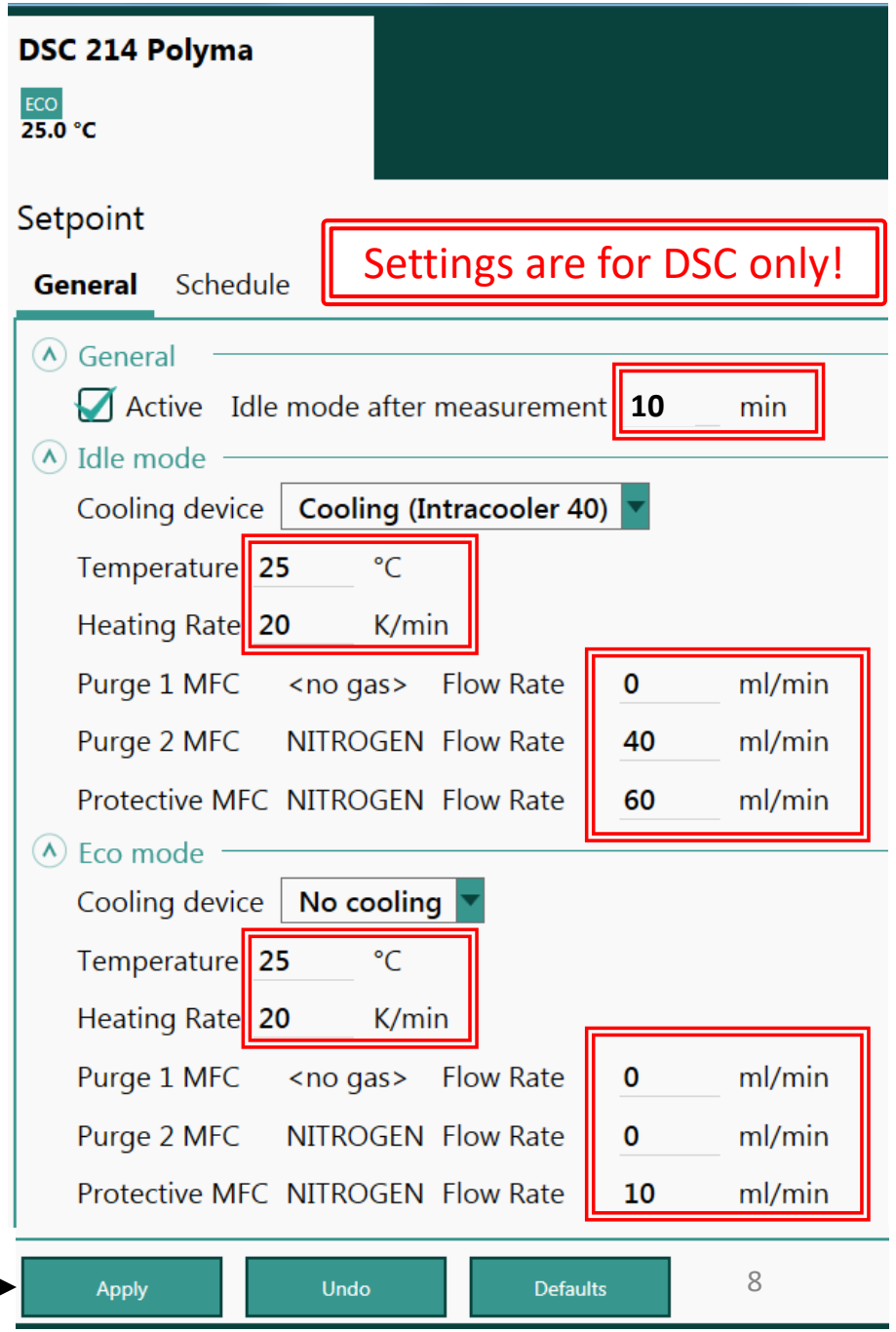
II. Start – 2/2

5. Click on ***Setpoint***

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

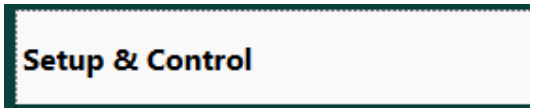
7. If they values are the same, proceed to **III. Setup & Control**

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

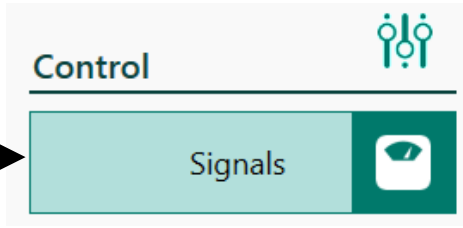


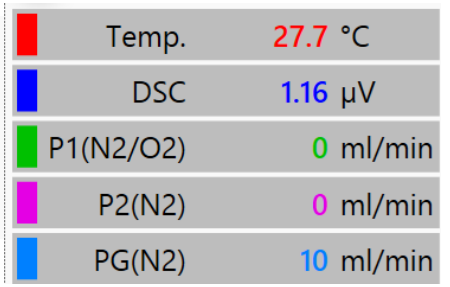
III. Setup & Control – 1/4

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



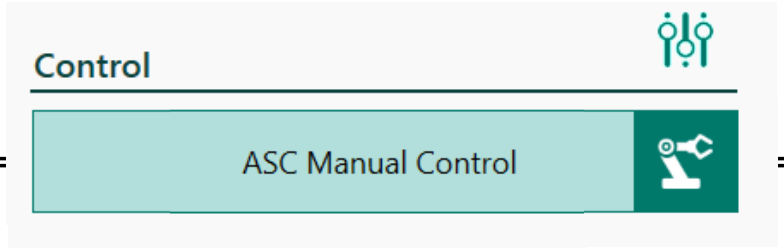
2. Click on ***Signals*** to activate the pop-up window showing the Temperature and Gas Flow signals

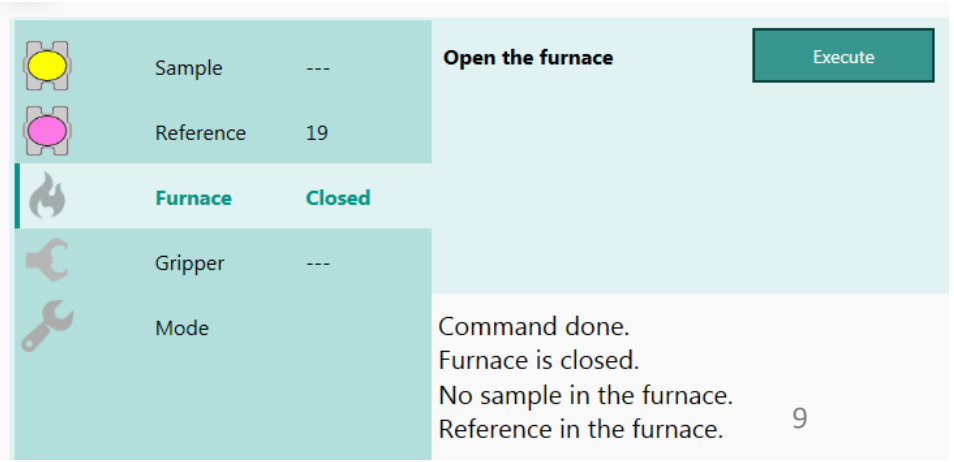




3. Click on the ***Back*** button to return to main menu

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

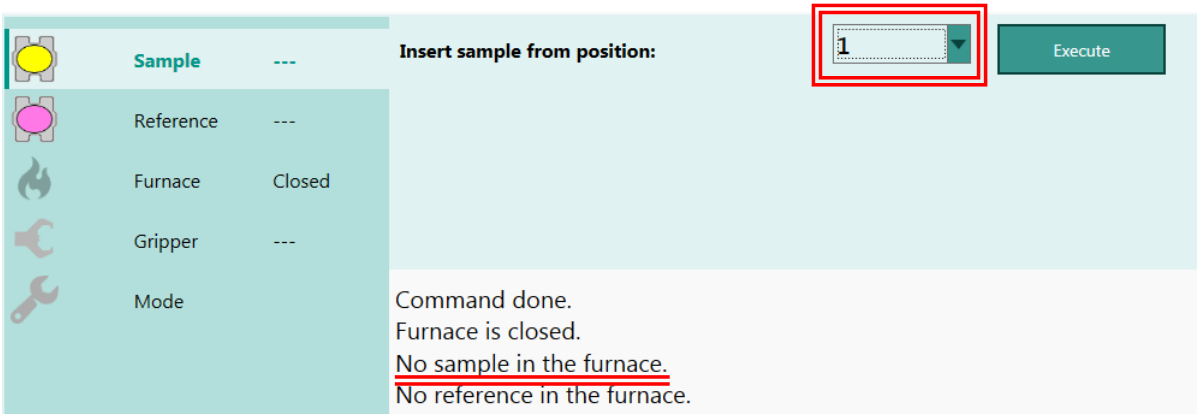




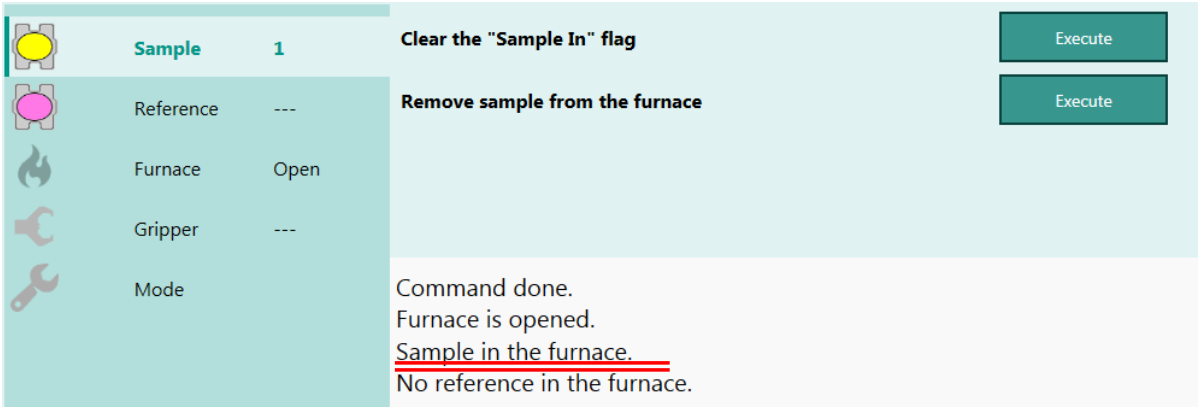
III. Setup & Control – 2/4

4. Review the different commands available below

Condition: **No sample in the furnace**



Condition: **Sample in the furnace**

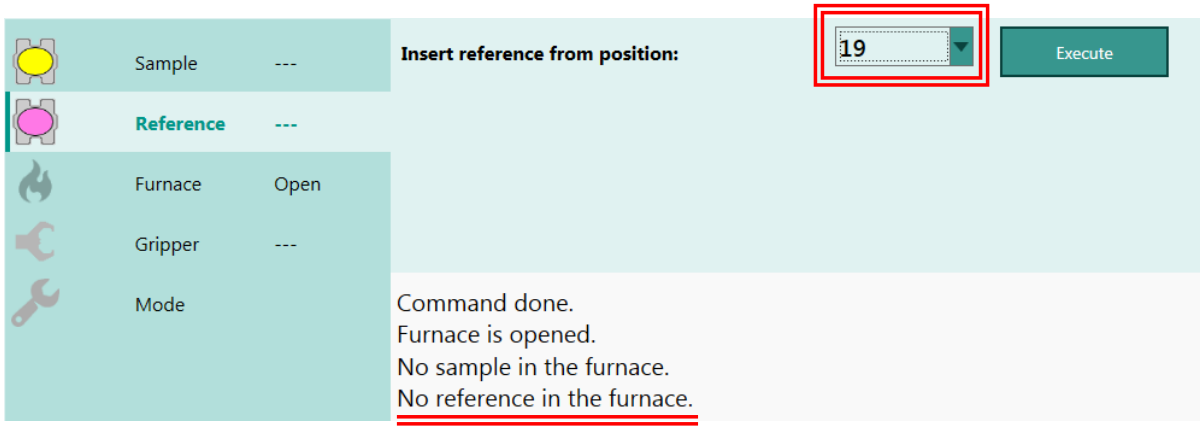


- ***Insert sample from position***: Select sample from position in dropdown to insert into furnace
- ***Clear the "Sample In" flag:*** **ONLY** use this if there is **NO** sample in furnace, to correct this "error"
- ***Remove sample from the furnace***: Use this to remove the current sample from the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

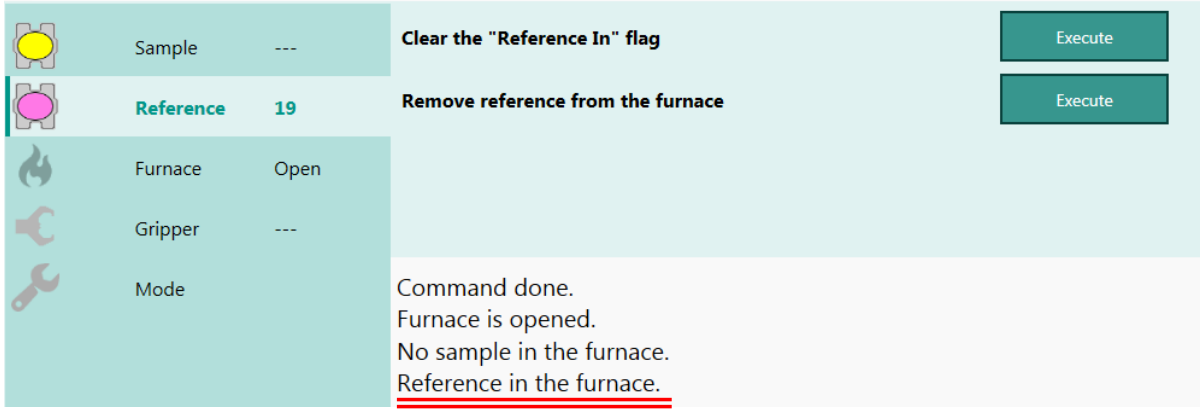
III. Setup & Control – 3/4

4. Review the different commands available below

Condition: **No reference in the furnace**



Condition: **Reference in the furnace**

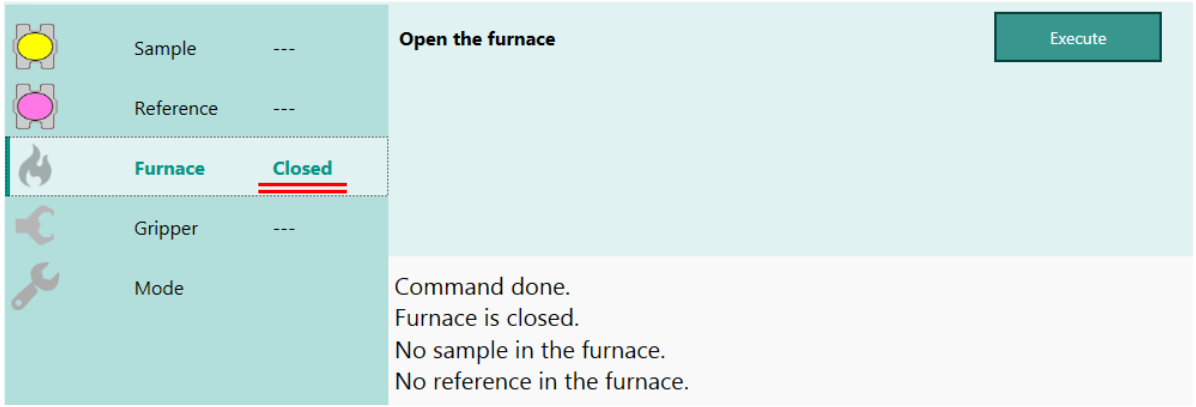


- ***Insert reference from position***: Select reference from position in dropdown to insert into furnace
- ***Clear the "Reference In" flag:*** **ONLY** use this if there is **NO** sample in furnace, to correct this "error"
- ***Remove reference from the furnace***: Use this to remove the current reference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

III. Setup & Control – 4/4

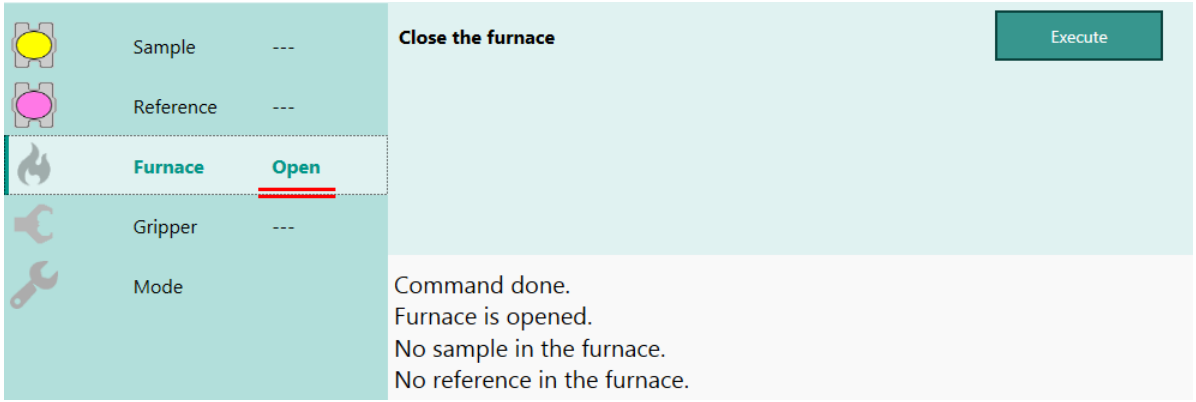
4. Review the different commands available below

Condition: **Furnace is closed**



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

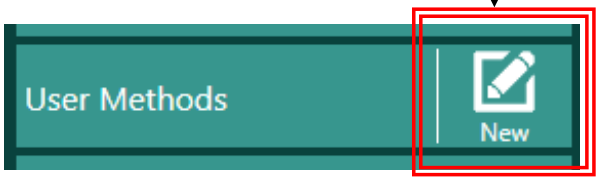
Condition: **Furnace is open**

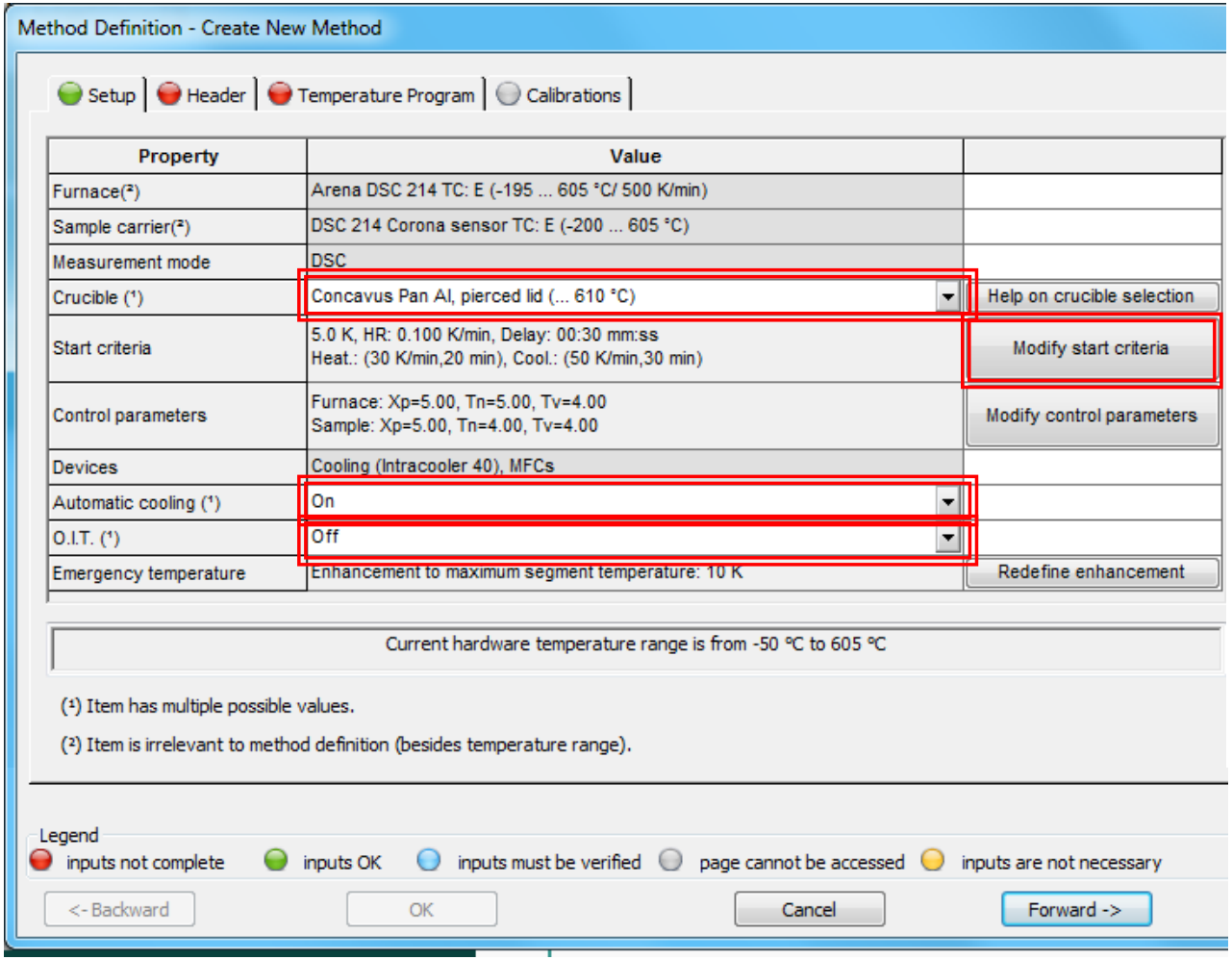


- ***Close the furnace:*** Click to close the furnace

IV. New Method – 1/9

1. Click on ***New*** next to ***User Methods***
2. Confirm settings below are accurate or acceptable:

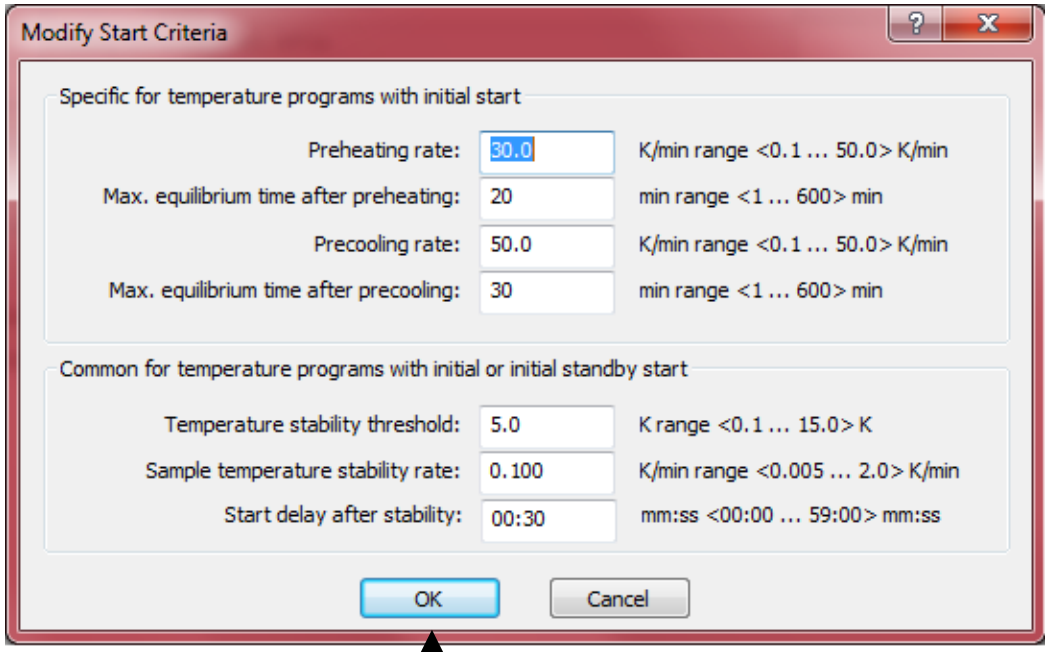




3. Crucible = ***Concavus Pan Al, pierced lid (… 610 °C)***
4. Automatic cooling = ***ON***
5. O.I.T. = ***OFF***
   (unless Oxidative Induction Time test is desired)
6. Click ***Modify start criteria***

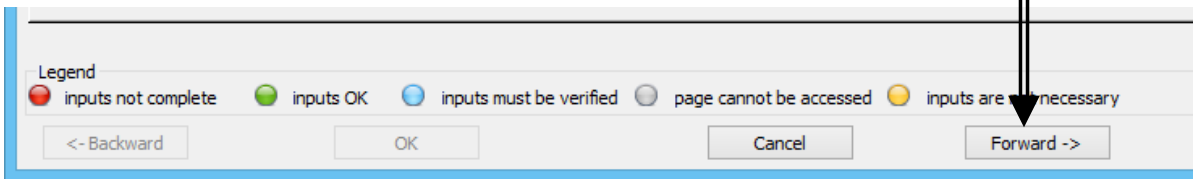
IV. New Method – 2/9

7. 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 = 5.0 K**
   - **Sample temperature stability rate = 0.1 K/min**
   - **Start delay after stability = 30 sec**



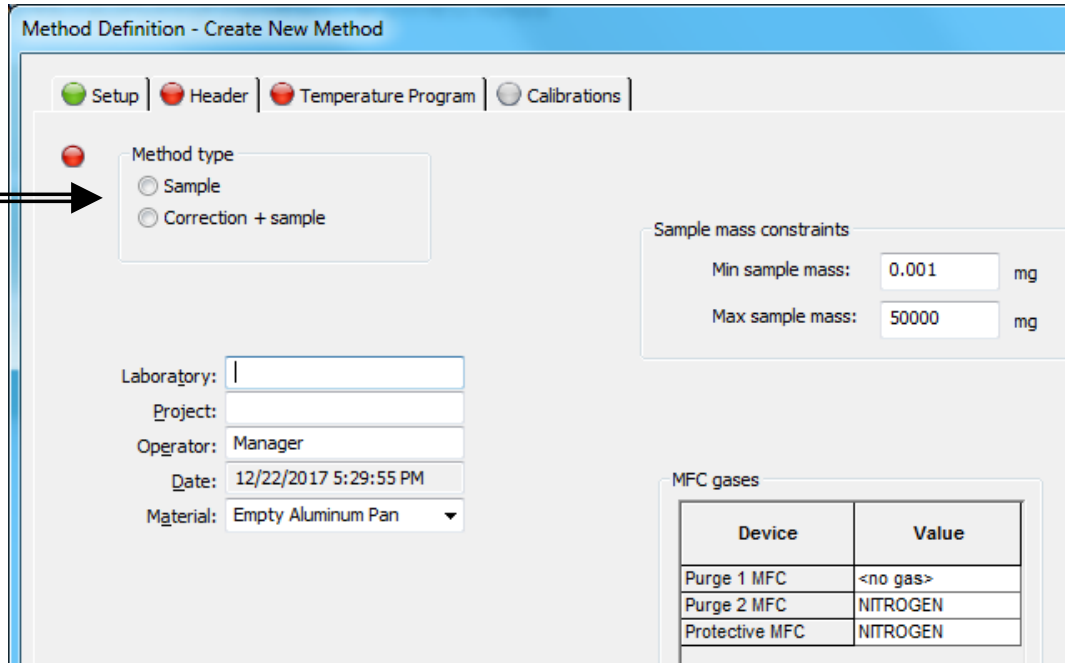
8. Click ***OK*** to accept settings

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

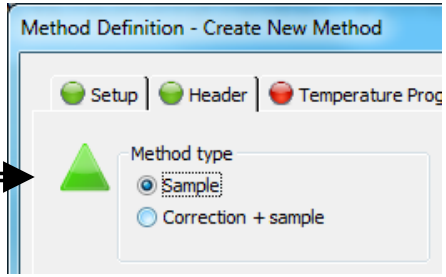


IV. New Method – 3/9

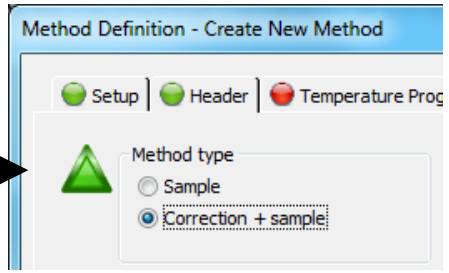
10. Select the desired ***Method*** type:



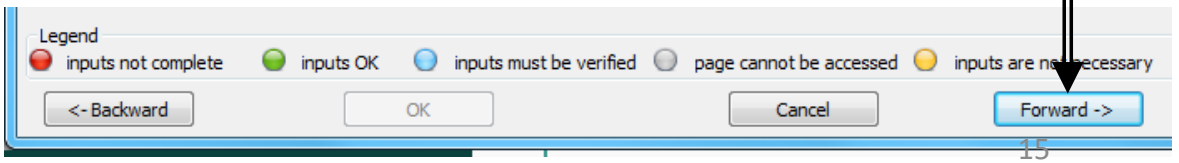
Sample - Single experiment with a sample inside crucible (Default)



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



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



IV. New Method – 4/9

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

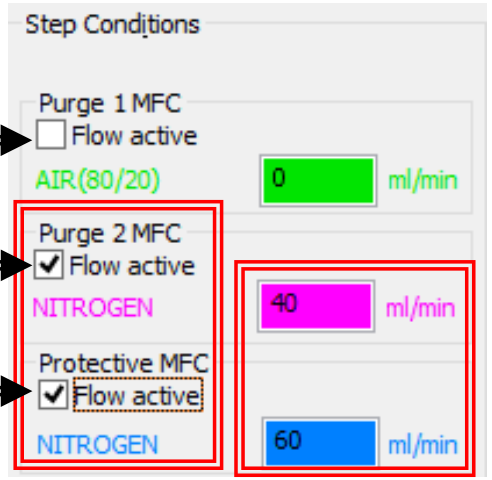
Protective MFC = Nitrogen over electronics

+

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: **40 mL/min N_2 (or Air)**

(Default) Protective: **60 mL/min N_2**

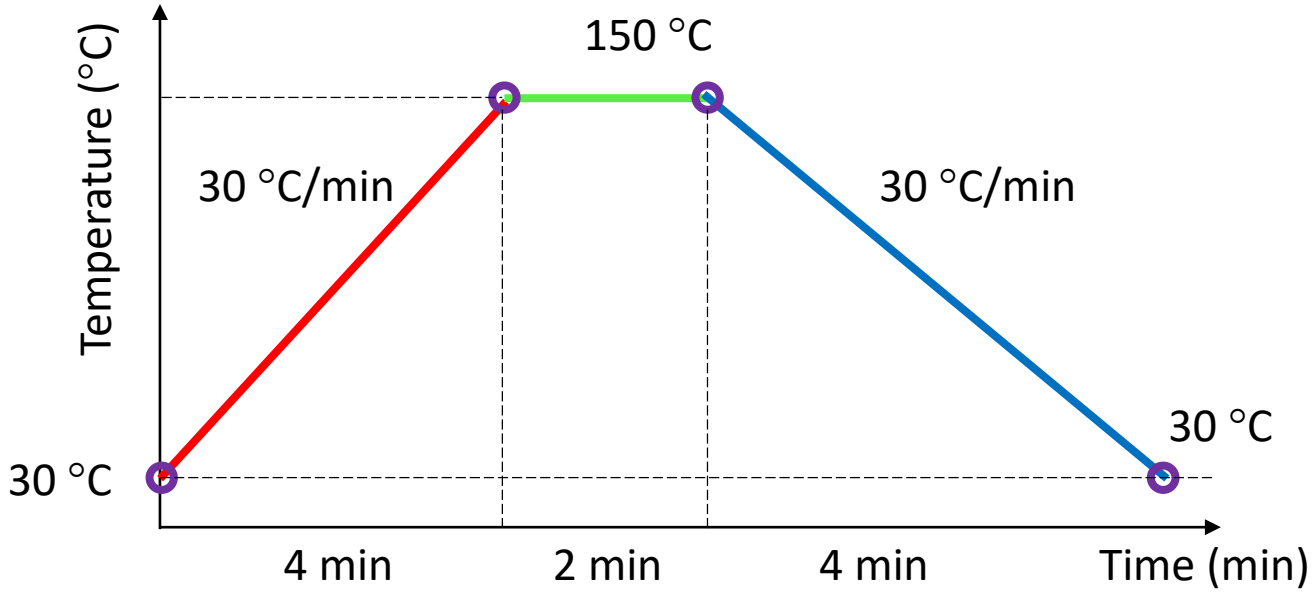
Protective must always ≥ 60 mL/min N_2

IV. New Method – 5/9

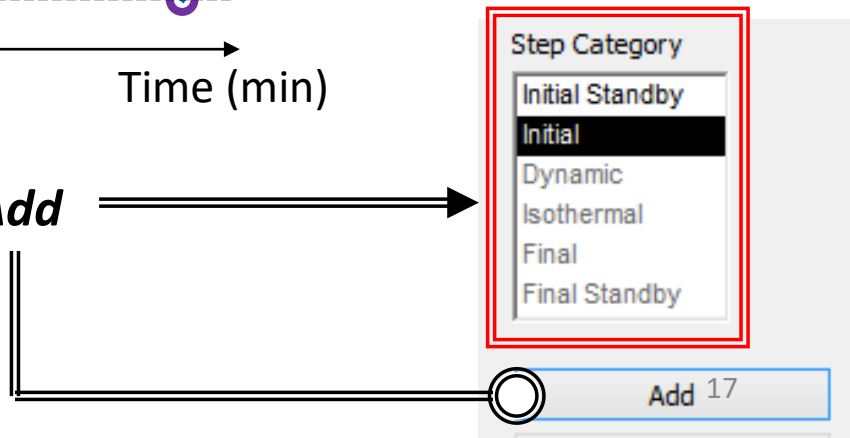
15. Build ***Temperature Program*** with desired ***Step Categories***

For NEW USERS AND TRAINING PURPOSES ONLY!

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



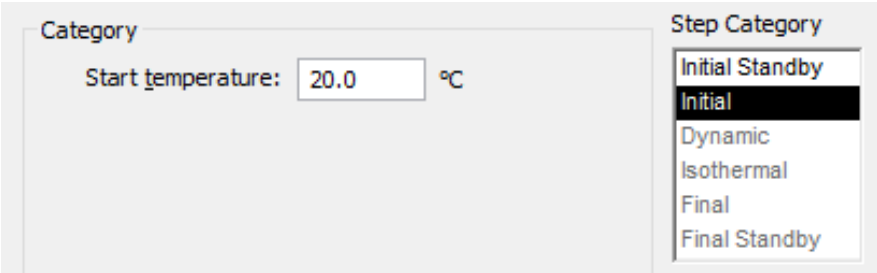
16. Select desired ***Step Categories*** and click ***Add***



IV. New Method – 6/9

17. Add ***Initial*** step

    a) Input Start temperature
    Recommended temp = ***25 – 30℃***



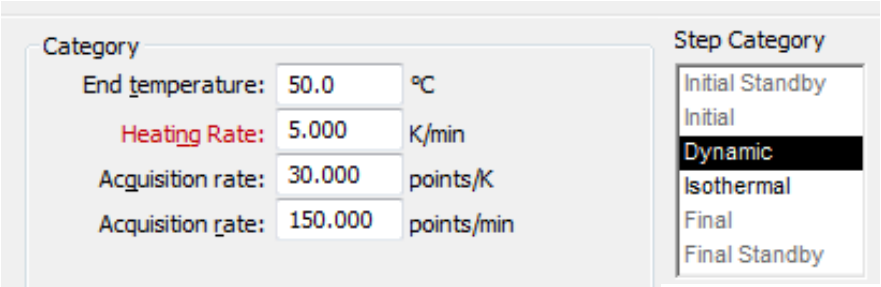
18. Add ***Dynamic*** step

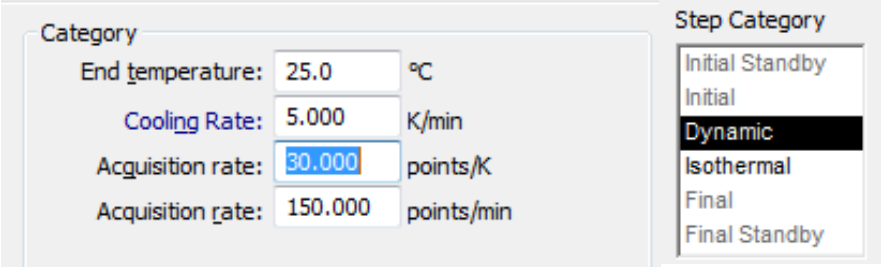
    a) Input End temperature

    b) Input Heating Rate or Cooling Rate

    Note: Heating Rate MUST be equal to calibrated rates

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

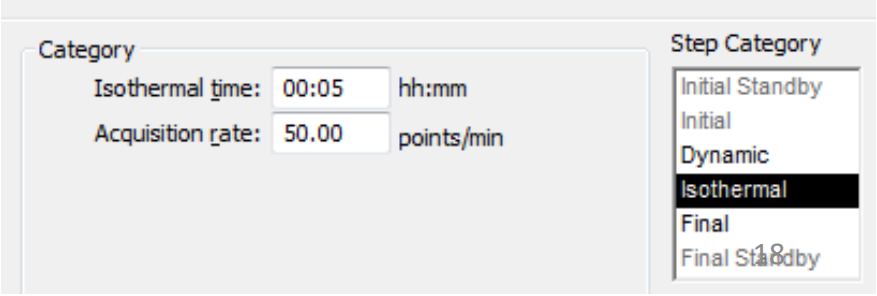




19. Add ***Isothermal*** step

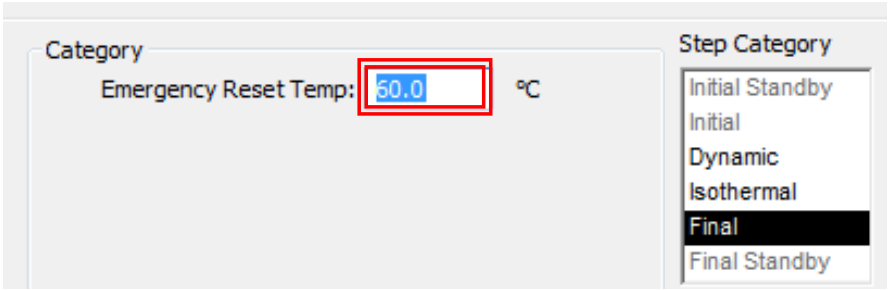
    a) Input Isothermal time

    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

Air Purge Gas Flowrate

Purge 2 N_2 Gas Flowrate

Setup | Header | Temperature Program | Calibrations

Nr	Type	°C	K/min	Time	pts/min	pts/K	AC	N2/O2	N2	N2
1		30.0					1	0	40	60
2		150.0	30.000	0:04:00	600.00	20.00	1	0	40	60
3		150.0		0:02:00	150.00		1	0	40	60
4		30.0	30.000	0:04:00	600.00	20.00	1	0	40	60
5		160.0					0	0	40	60
6		25.0	20.000	0:00:15			1	0	40	60
7		25.0		0:15:00			1	0	40	60

Type of Step

Temp

Heat/Cooling Rate

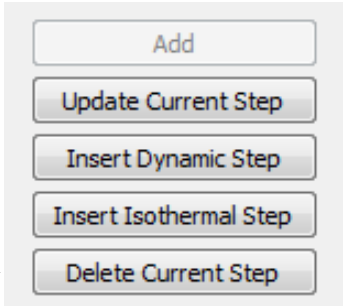
Time for Step

Acquisition Rate

Protective Gas Flowrate

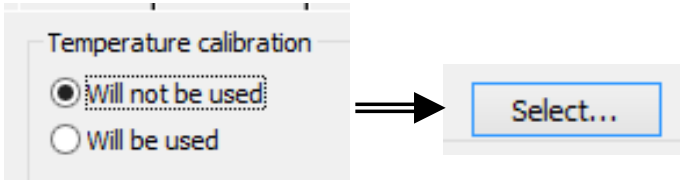
IV. New Method – 8/9

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***



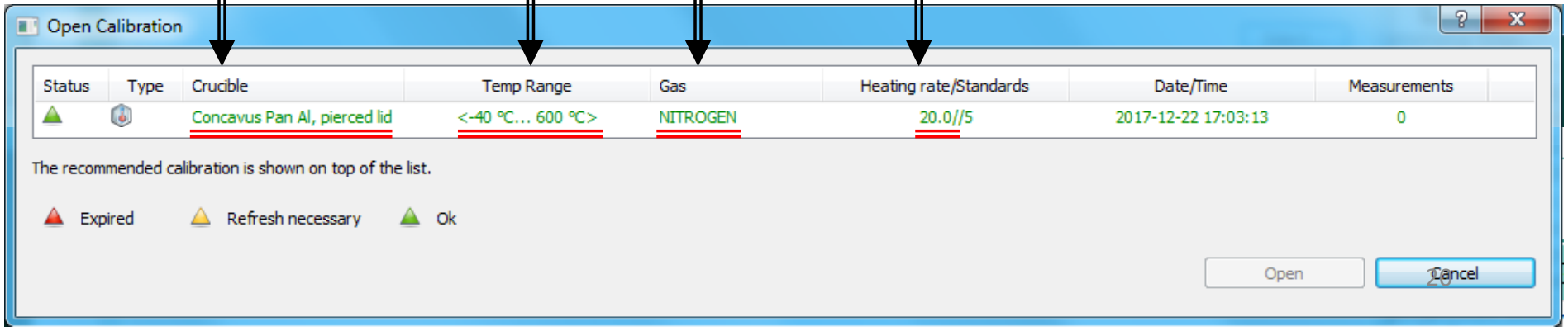
25. Select correct ***Calibration File***, checking **EVERY** condition is correct:

1) Crucible: Al2O3

2) Temp Range: 0 °C …1175 °C

3) Gas: NITROGEN or AIR

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



IV. New Method – 9/9

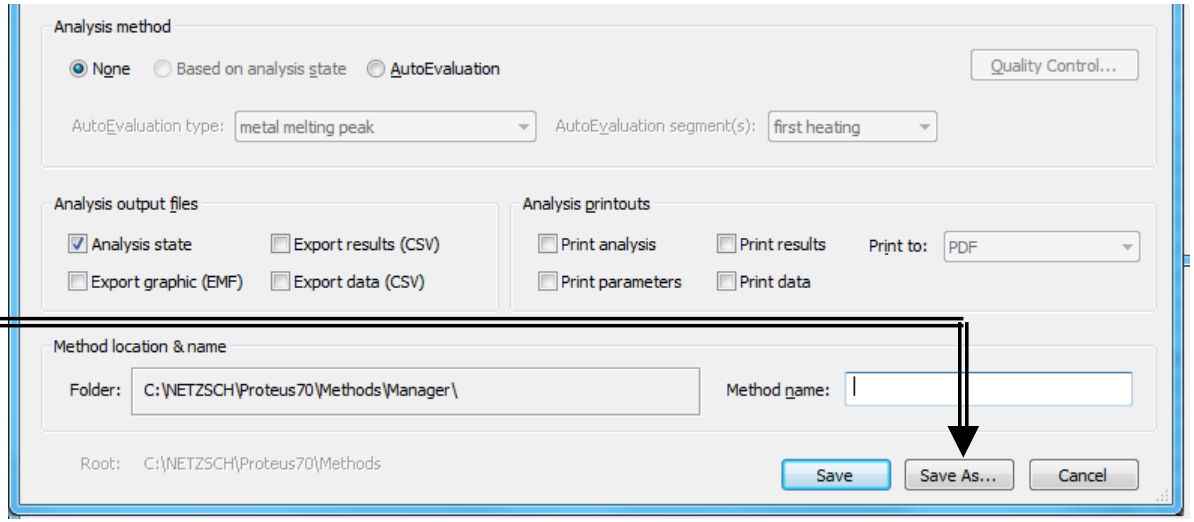
26. Repeat for ***Heat flow***, ***Tau-R***, and ***BeFlat calibration***

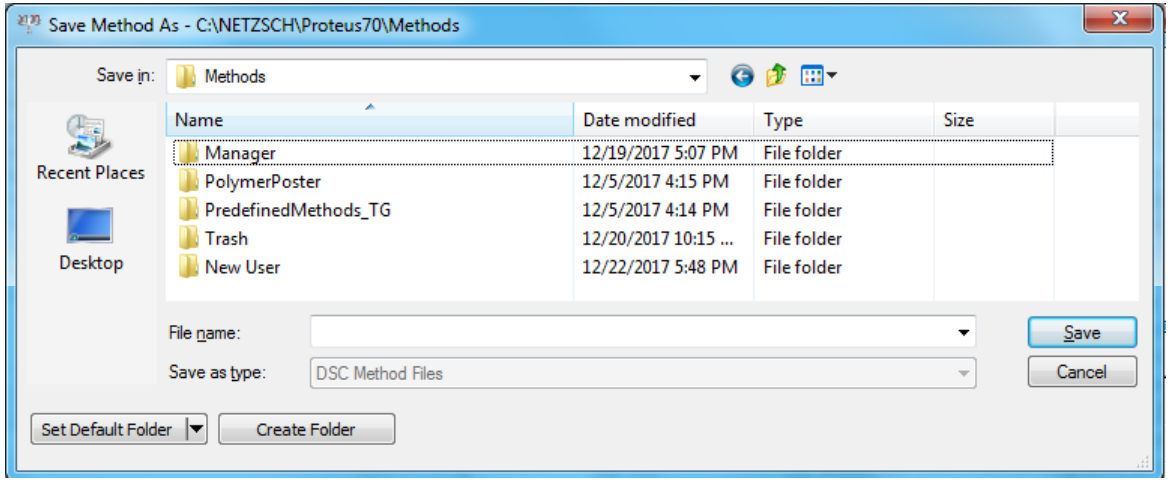
27. Click ***Forward ->*** to advance

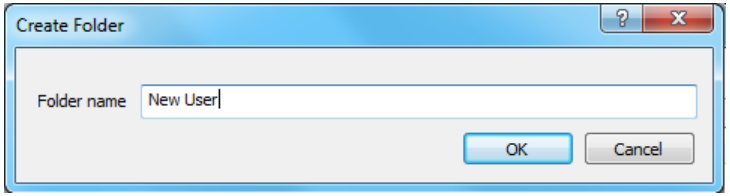
28. Click ***Save As…*** to save Method into desired folder under ***Methods***

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

30. Click ***Save***



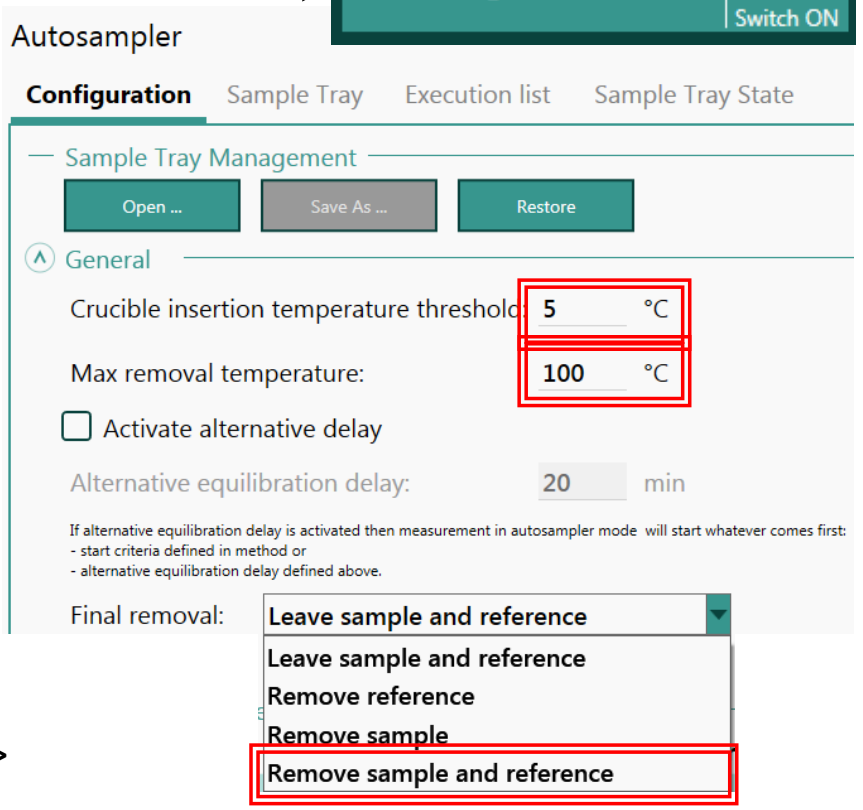


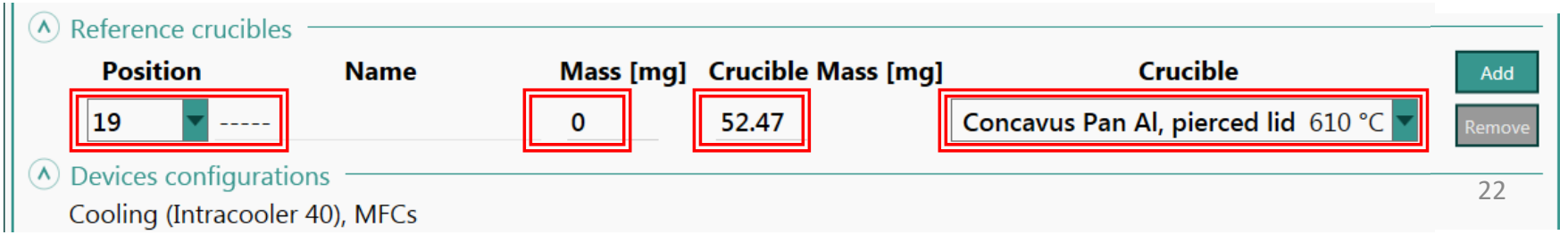


V. Adding Reference – 1/1

1. Click on ***ASC Manager*** and ***Switch ON***
2. Confirm ***Crucible insertion temperature threshold*** (Default = ***5 ℃***)
3. Confirm ***Max removal temperature*** of crucible (Default = ***100 ℃***)
4. Pick ***Final removal*** action for your last sample: (Default = ***Remove sample and reference***)
5. Click ***Add*** to enter Reference crucible info (see Default information below)

- Position = ***19***
- Mass (mg) = ***0***
- Crucible Mass (mg) = <***Use Precision Balance***>
- Crucible = ***Concavus Pan Al, pierced lid 610 ℃***





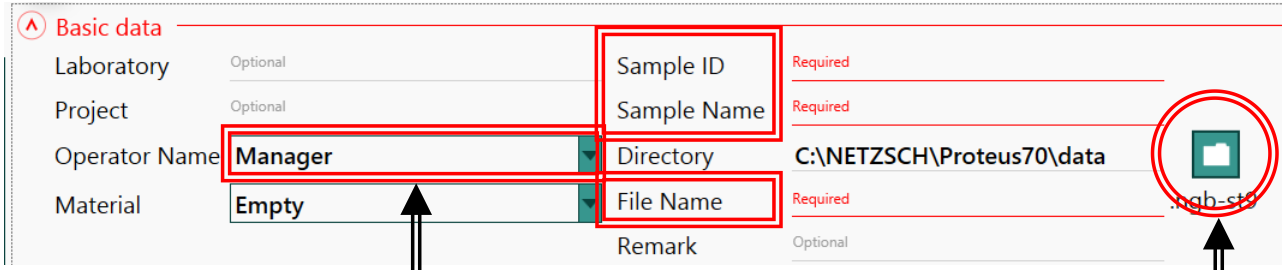
VI. Opening Method – 1/3

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

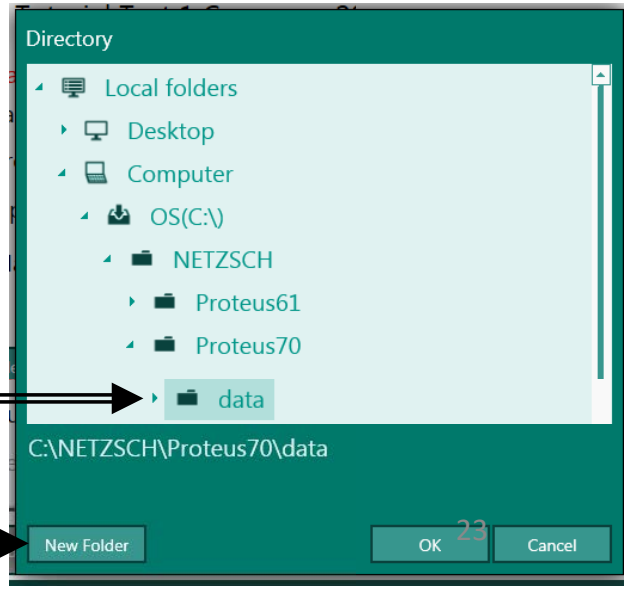


4. Enter ***Operator Name*** with your ***User Name*** for reference

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***

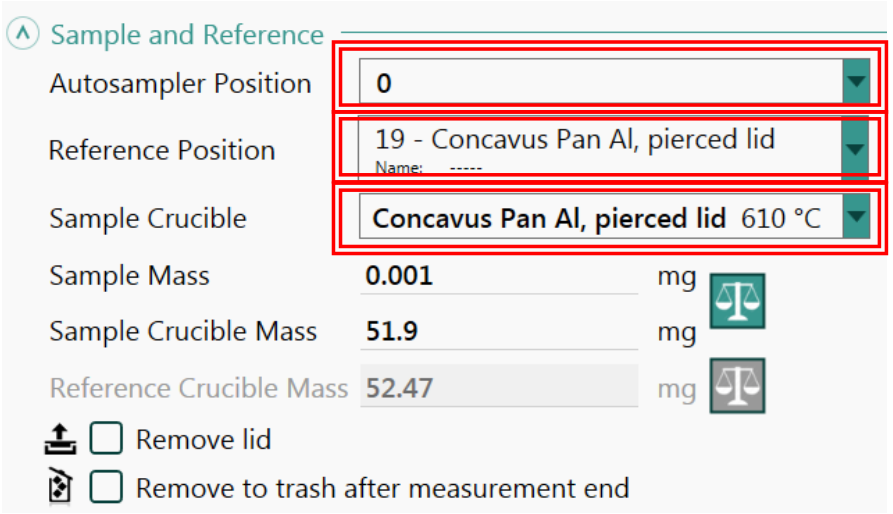


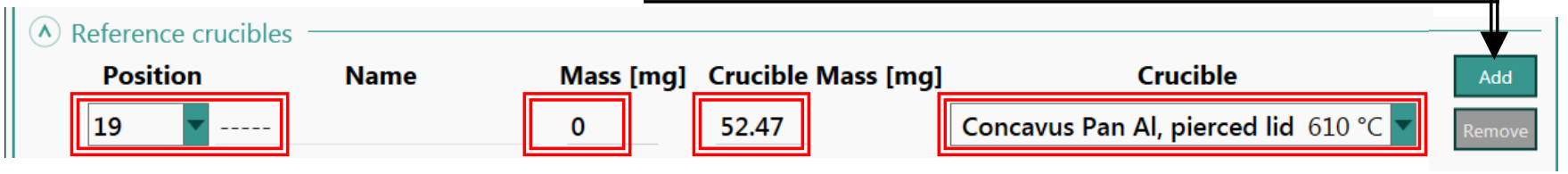
VI. Opening Method – 2/3

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

8. Select the ***Reference Position*** Recommend ***Position 19*** (last position)

9. If ***Reference*** is new or different from before, proceed to ***ASC Manager*** to ***Remove*** previous Reference and ***Add*** new ***Reference***



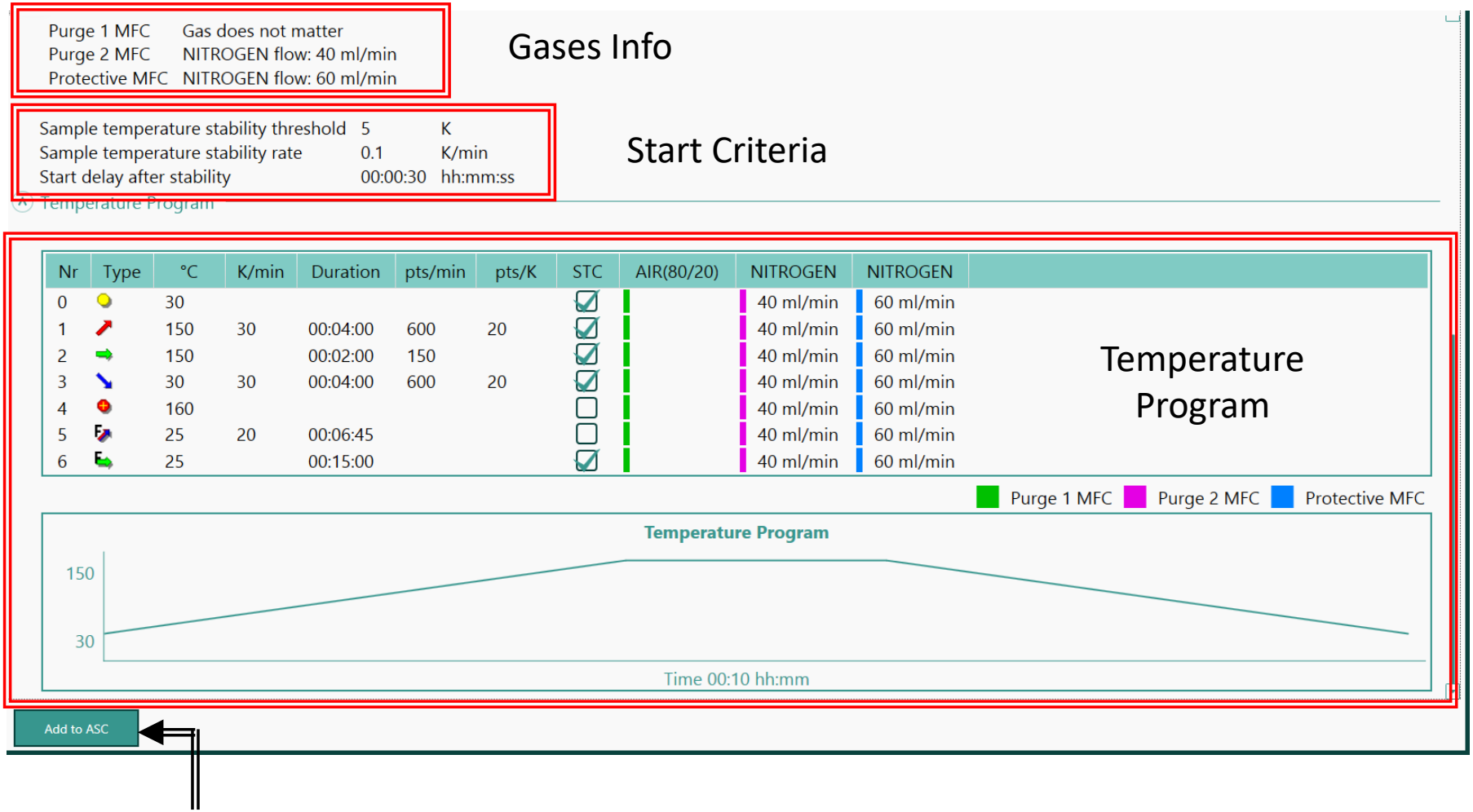


10. Select the type of ***Sample Crucible*** (Default = ***Concavus Pan Al, pierced lid 610 ℃***)

11. Enter the ***Sample Mass*** and ***Sample Crucible Mass*** (Use the Precision Balance next to the DSC)

VI. Opening Method – 3/3

12. Review that following is correct for your desired ***Method:***



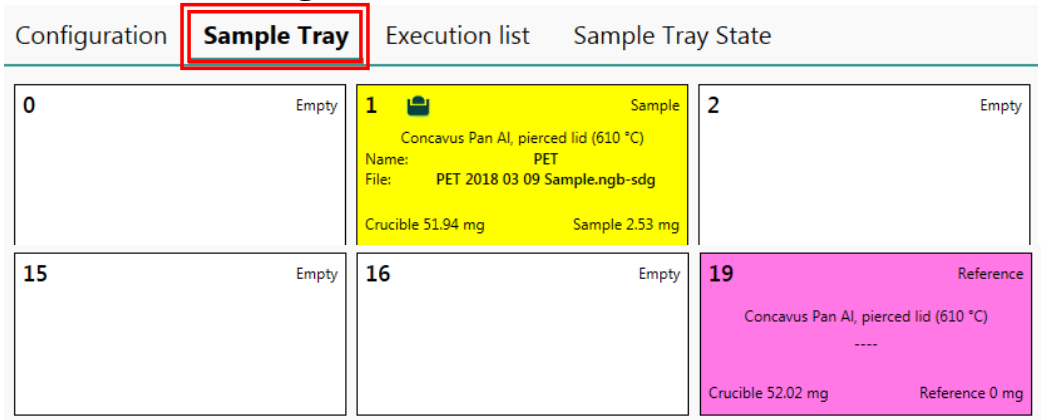
13. If everything is correct, proceed to Add Method to the ASC queue by clicking ***Add to ASC*** at the bottom

VII. ASC Manager – 1/1

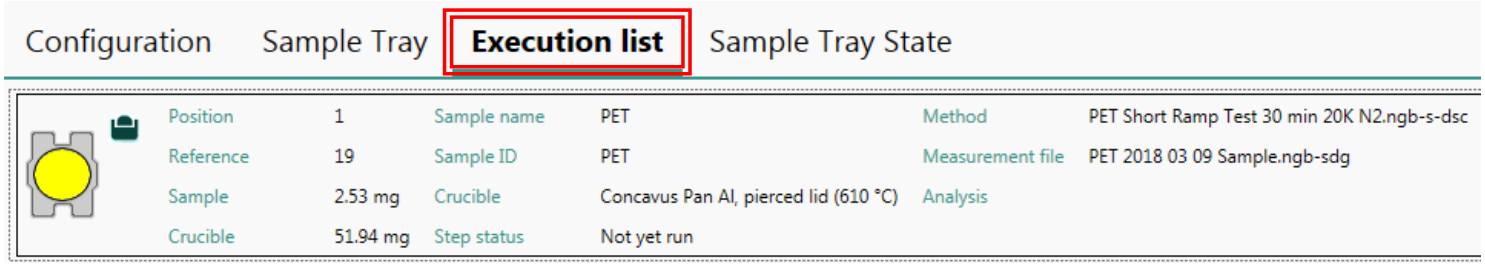
1. Click on ***ASC Manager*** and ***Switch ON*** if not already ON

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

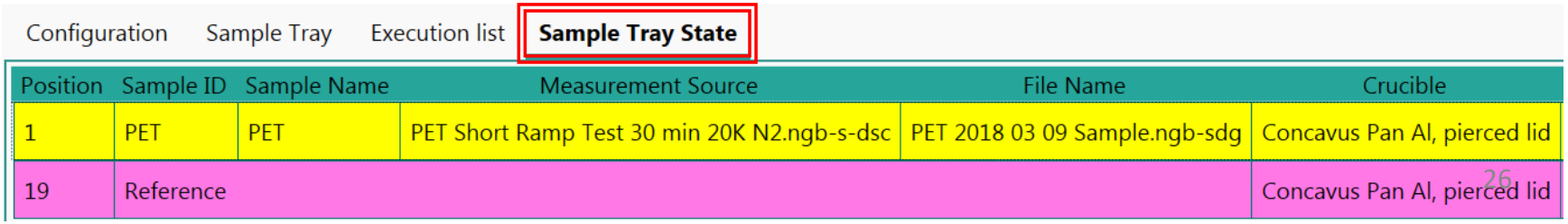
	Defined
	Done
	Done (analysis failed)
	Failed
	Measurement Active
	Reference



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



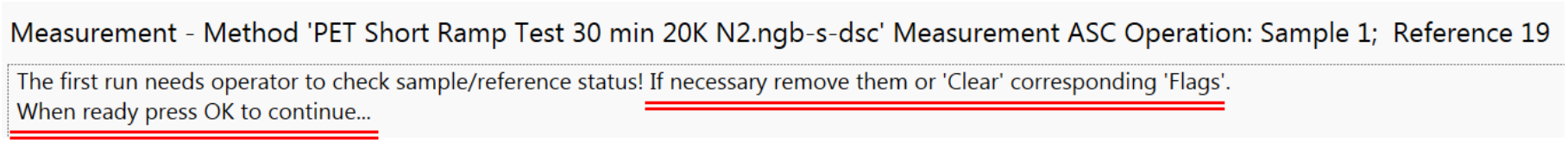
4. Click ***Sample Tray State*** to review details of experiments scheduled



VIII. Running Experiments – 1/3

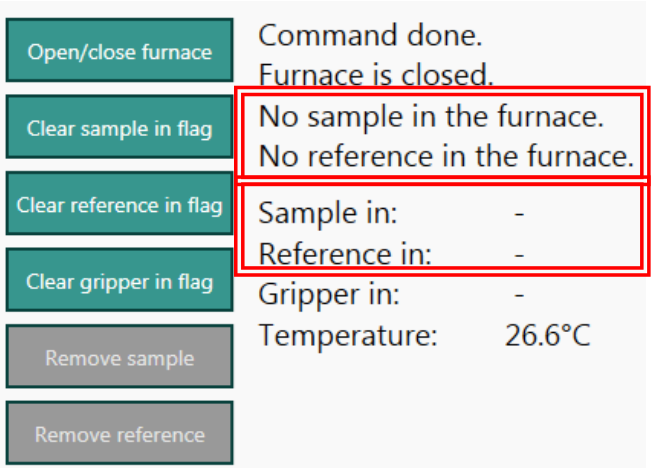
1. Review ***ASC Manager*** settings and confirm all is correct
2. Click ***Start*** when ready

3. Proceed to review the notes described here. IMPORTANT!



4. 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:

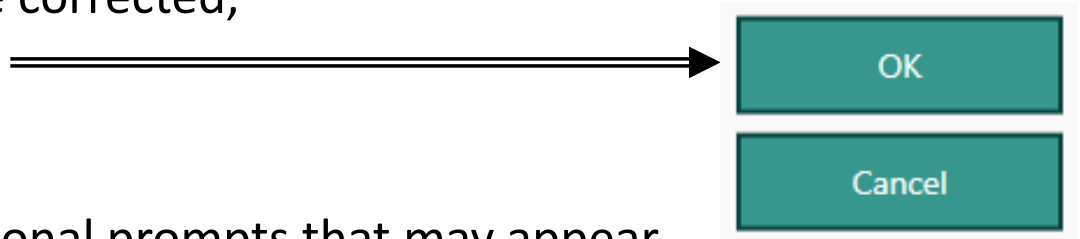
- Is the furnace open or closed?
- Is the correct sample in?
- Is the correct reference in?

VIII. Running Experiments – 2/3

5. 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 **ONLY IF** there is **NO** reference in the furnace
- click to “remove” the current sample
- click to “remove” the current reference

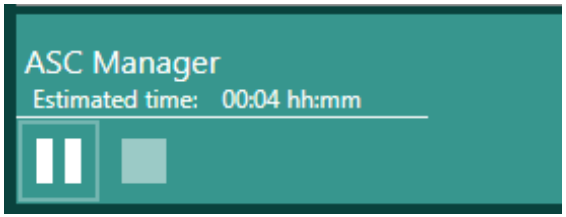
6. After all flags and conditions are corrected, click ***OK*** to begin experiments

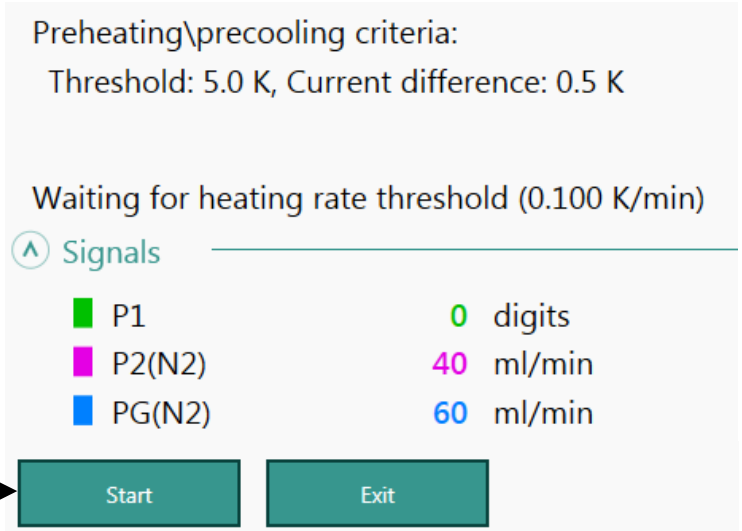


7. Follow any instructions or additional prompts that may appear

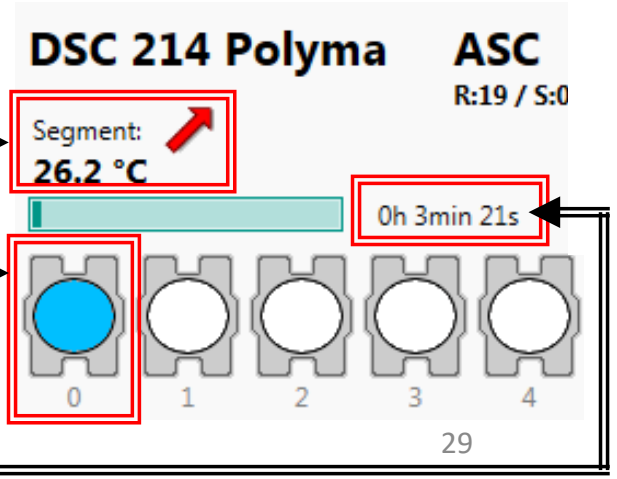
VIII. Running Experiments – 3/3

8. DSC will now begin to preheat/precool to target Initial temperature
9. Click ***Start*** when initial conditions have been met for experiment
10. The ***Estimated time*** for all experiments are shown here



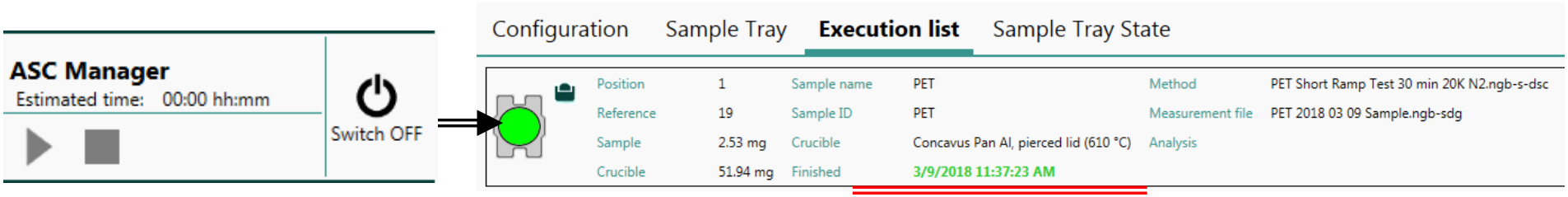


11. The ***current temperature*** and ***segment action*** is shown here
12. Active measurement can be shown here
13. Remaining ***segment time*** and ***completion %*** is shown here

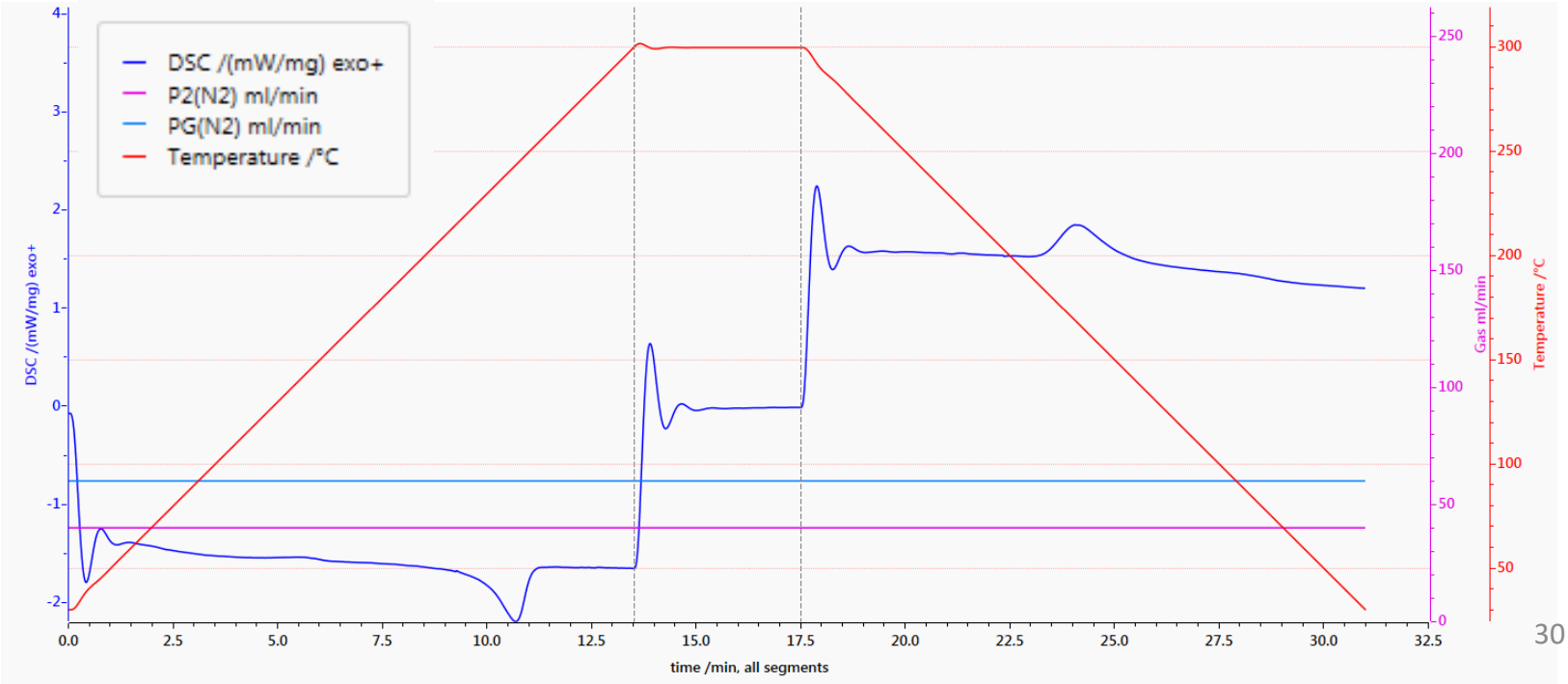


IX. Results – 1/1

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

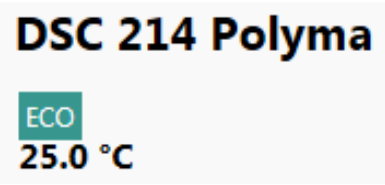


2. Click ***Measurement***, to show the results



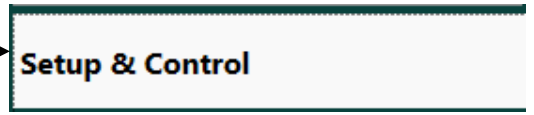
X. Clean Up – 1/1

1. After experiment(s), DSC should cool itself down to 25°C via ***Idle Mode*** or ***Eco Mode*** at top of the screen

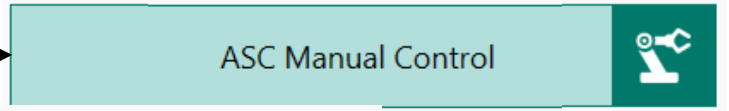


2. If ***Eco Mode*** is not activated, proceed to turn back on ***Setpoint*** first by clicking ***Apply***

3. Click on ***Setup & Control***

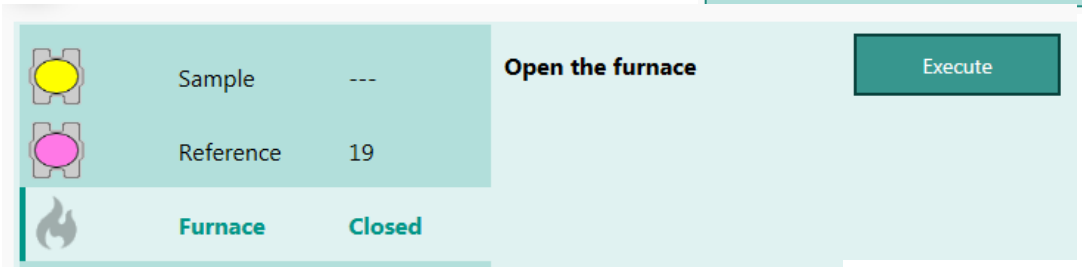


4. Click on ***ASC Manual Control***



5. Check that the following are true, else correct:

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



6. When TGA temperature reaches ***25 ± 5 ℃***, click the ***X*** to close the software

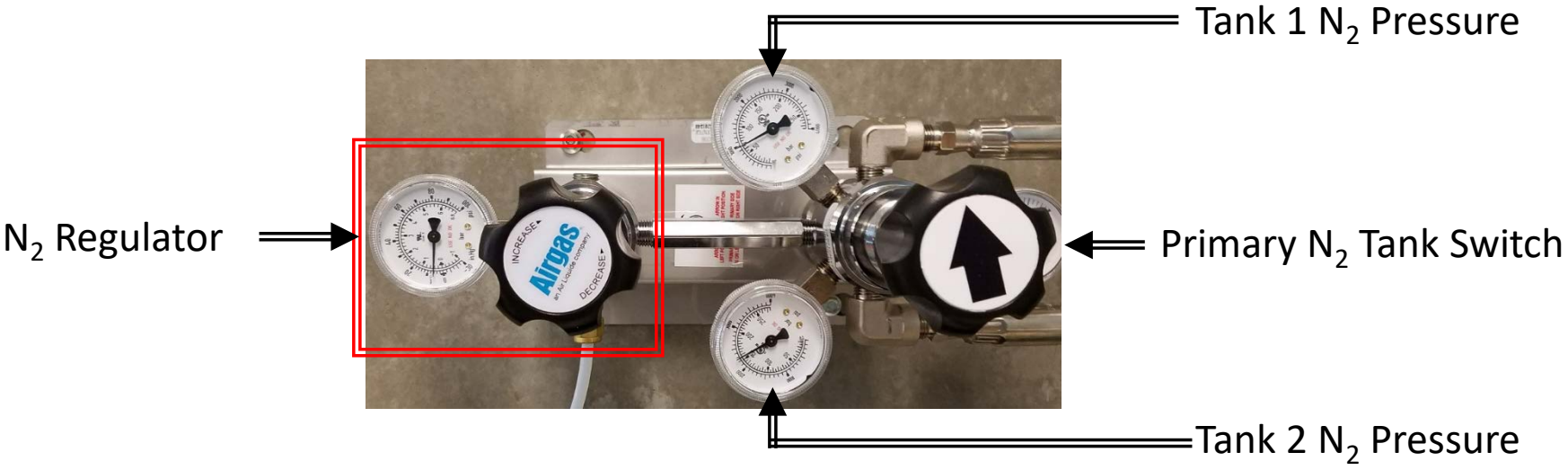
7. Confirm that you wish to ***keep the Setpoint ON*** after you close software

8. ***Log out*** of your ENGR account

9. Clean up the lab bench and place all items back in their respective drawers

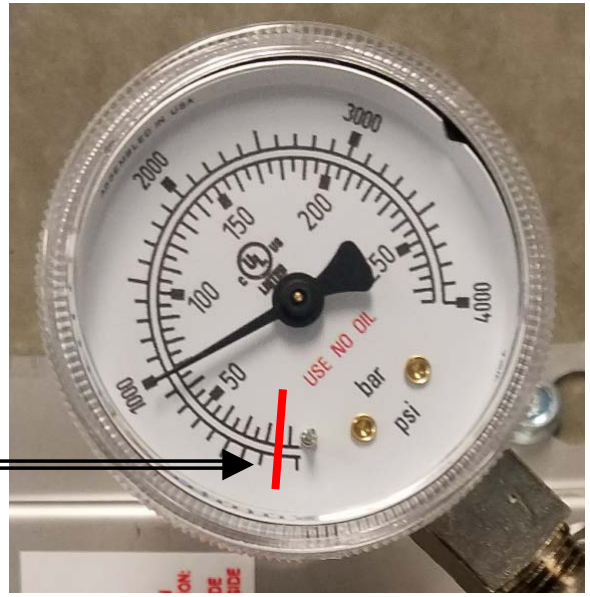
10. Sign out on the ***Sign-In Sheet*** before leaving

XI. Red Flags & Mistakes – 1/3



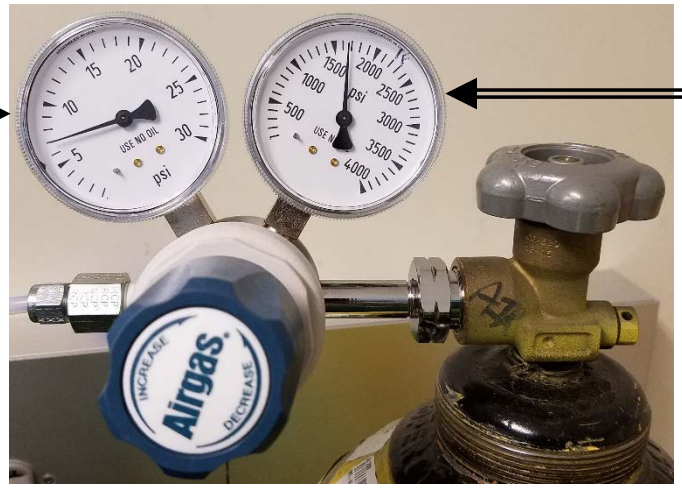
1. DO NOT ADJUST THE REGULATOR AS THIS MAY DAMAGE MASS FLOW CONTROLLERS $$$

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

XI. Red Flags & Mistakes – 3/3

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

DSC sample chamber

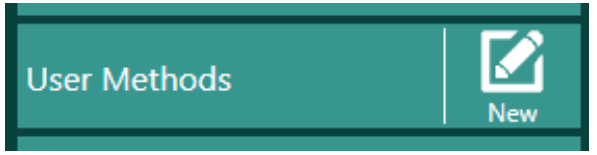
6. Avoid over-filling the crucible in case the sample boils and bubbles over contaminating the DSC sample chamber $$$

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

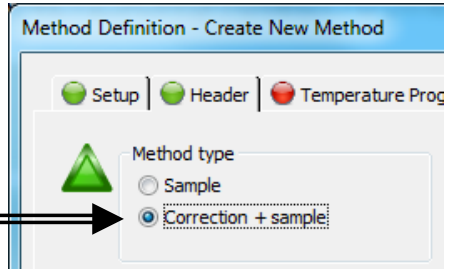
XII. Baseline Corrections – 1/6

The following are modifications to perform a baseline correction

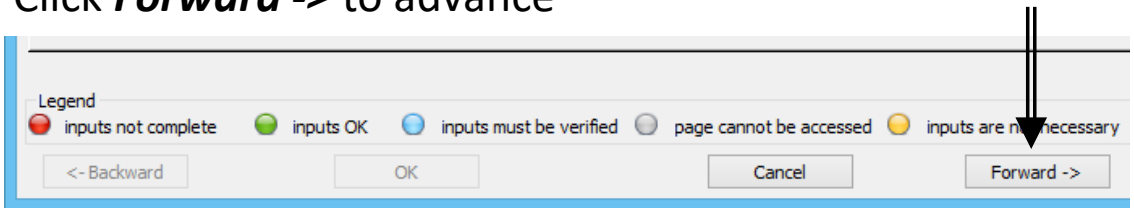
1. Click on New next to User Methods



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



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



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

Setup | Header | Temperature Program | Calibrations | O.I.T.

Nr	Type	°C	K/min	Time	pts/min	pts/K	AC	N2	N2
1		30.0					1	40	60
2		300.0	20.000	0:13:30	600.00	30.00	1	40	60
3		300.0		0:04:00	75.00		1	40	60
4		30.0	20.000	0:13:30	600.00	30.00	1	40	60
5		310.0					0	40	60
6		25.0	20.000	0:00:15			1	40	60
7		25.0		0:15:00			1	40	60

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

XII. Baseline Corrections – 2/6

6. Select the same ***Temperature , Heat flow, and Tau-R calibrations*** as before…

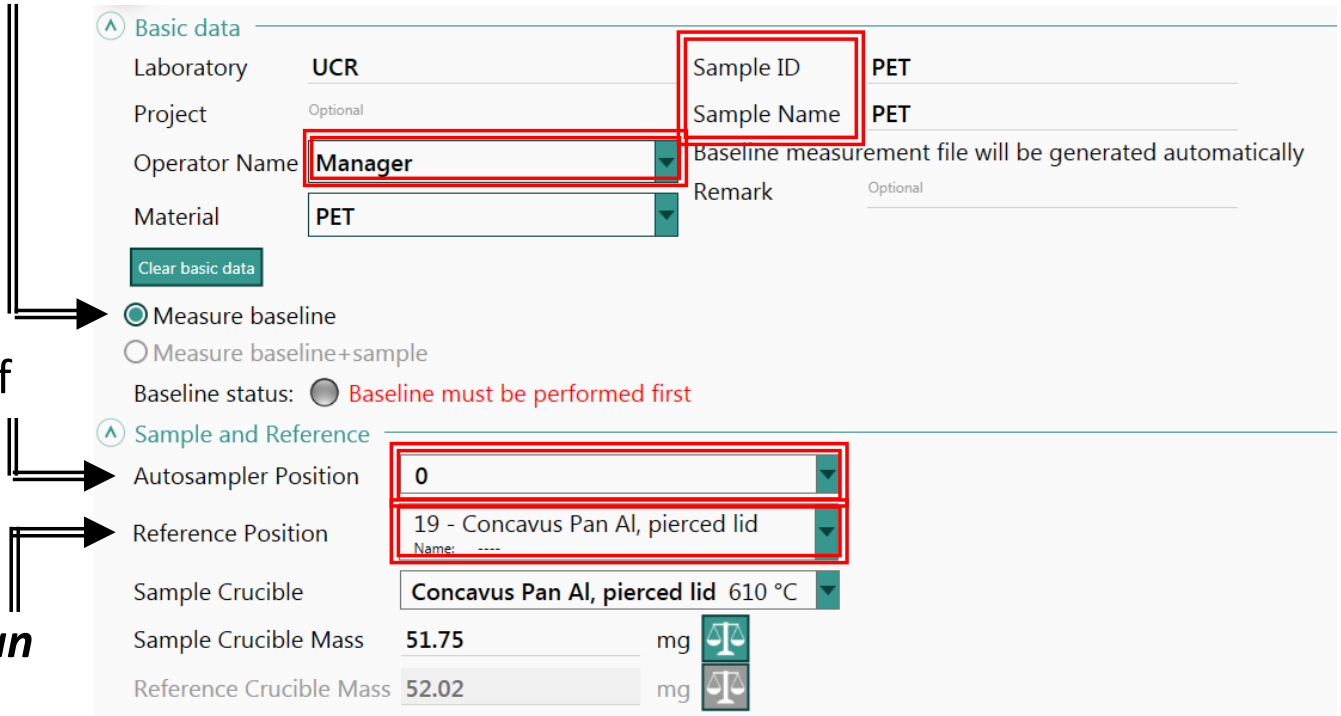
7. The ***TG BeFlat calibration*** will be missing (that's OK)Click ***Forward ->*** to advance

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

XII. Baseline Corrections – 3/6

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

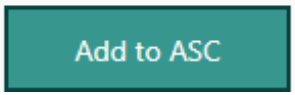
10. You will have to perform a baseline measurement first (unless you have already ran it)



11. Select the position of your ***Empty Pan***

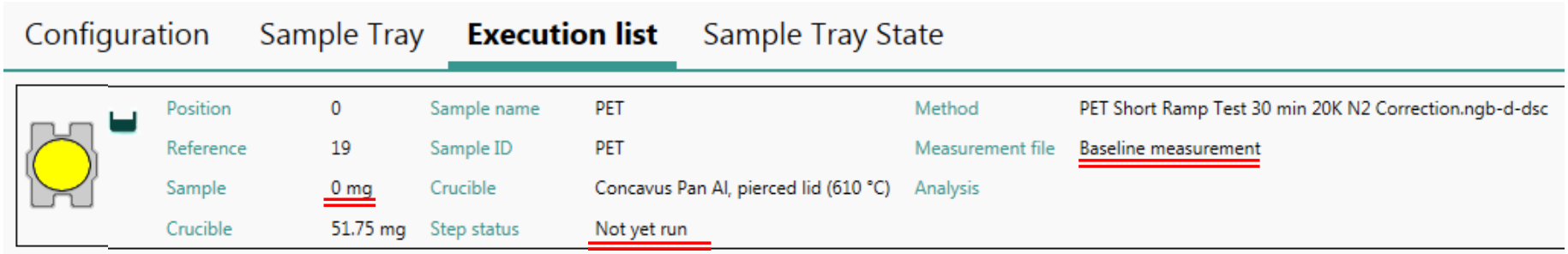
12. Select the position of your ***Reference Pan***

13. Add to ASC at the bottom



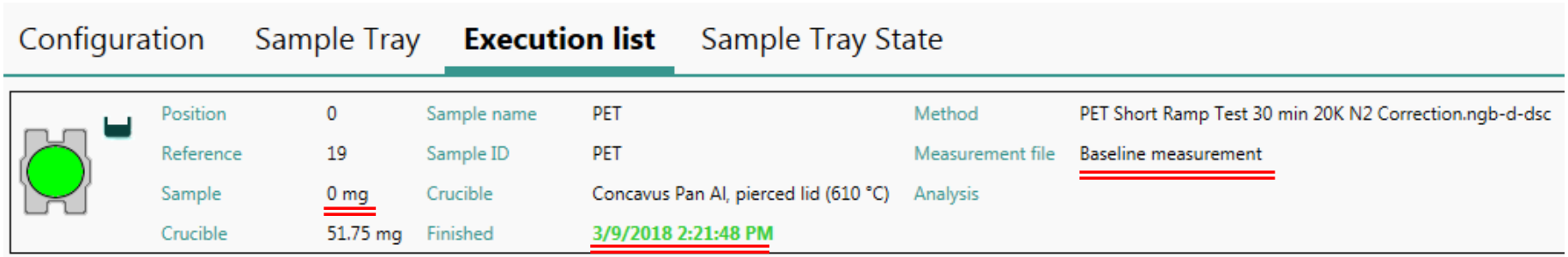
XII. Baseline Corrections – 4/6

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



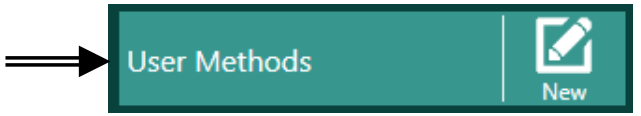
15. Click ***Start*** to perform the ***Baseline measurement…***

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



XII. Baseline Corrections – 5/6

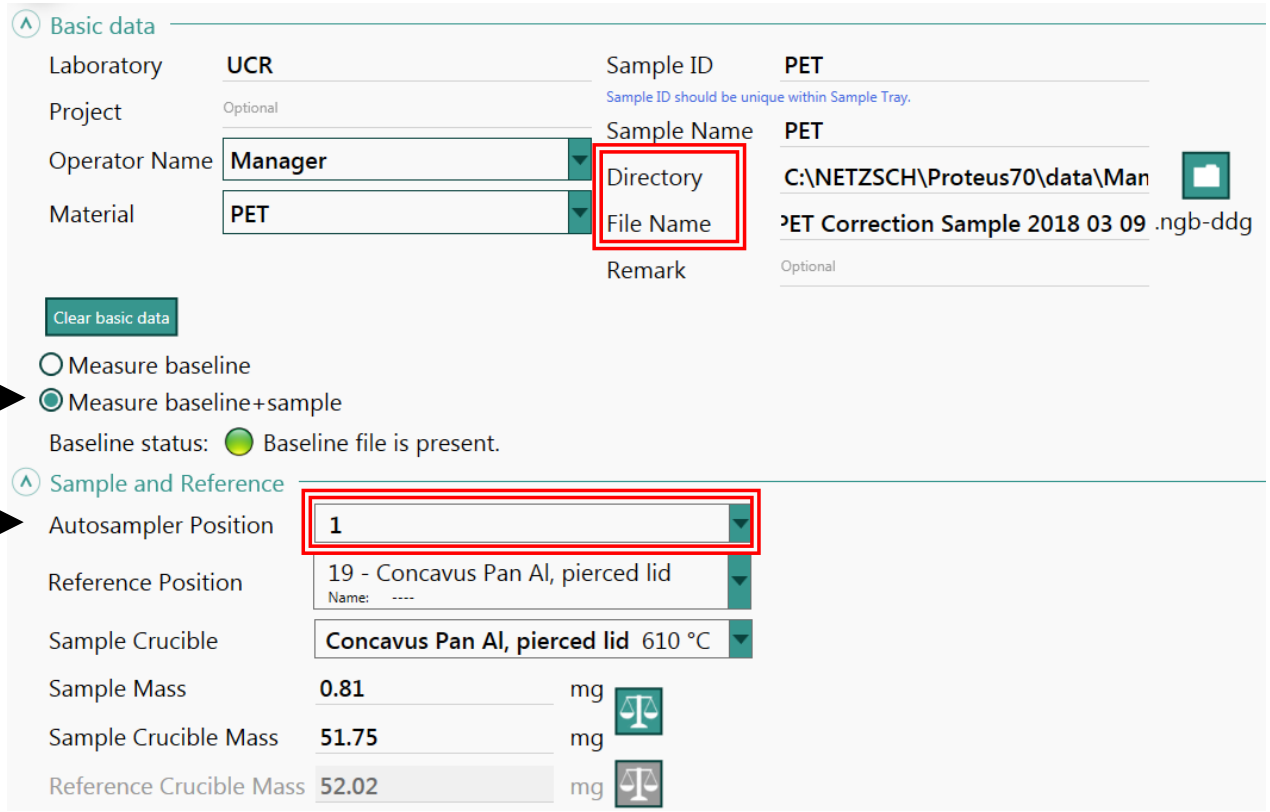
17. Click ***User Methods*** again



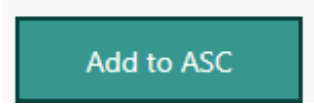
18. You can now create a ***File Name*** for your sample

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

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

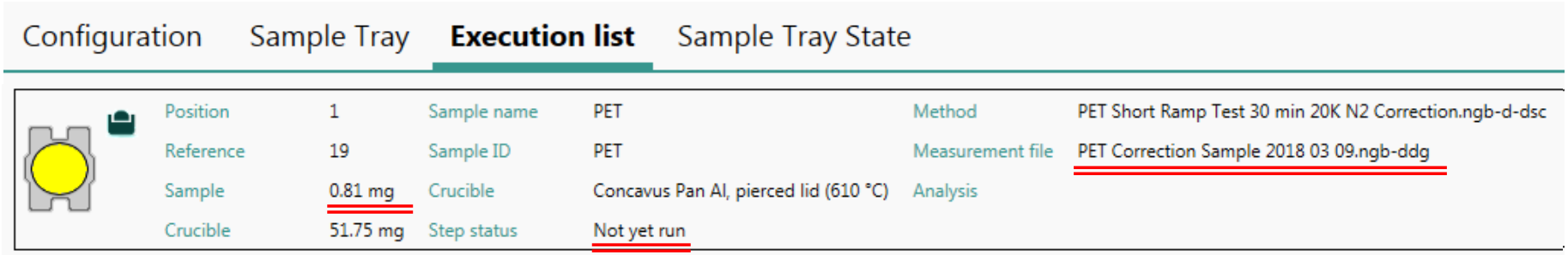


21. Add to ASC at the bottom



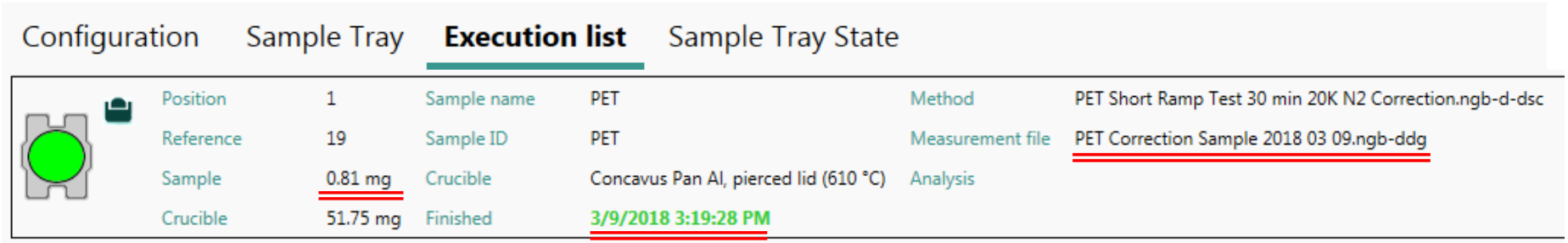
XII. Baseline Corrections – 6/6

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



23. Click ***Start*** to perform the ***sample measurement…***

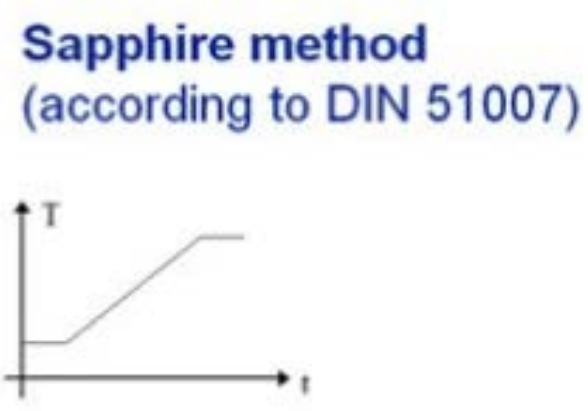
24. Proceed to review the notes and correct all issues as before… Click ***OK***



XI. C_p: Sapphire Method – 1/1

1. Review the documents found on the website on performing Cp measurements

   - Netzsch C_p Sapphire Method
   - Netzsch Tips for C_p Measurement
   - Mettler Toledo Measuring C_p Guide



2. Every determination of the C_p by DSC is comprised of 3 measurements:
   a. Baseline
   b. Standard – Sapphire (see Lab Manager)
   c. Sample

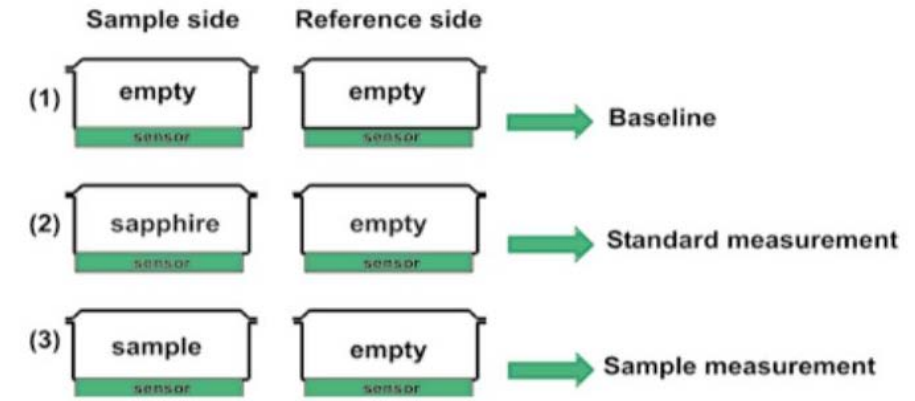


Fig. 1. Setup of a measurement series for determination of the specific heat