

Netzsch TGA Training Notebook

Lab Manager: Dr. Perry Cheung
MSE Fee-For-Service Facility
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 TGA Operation

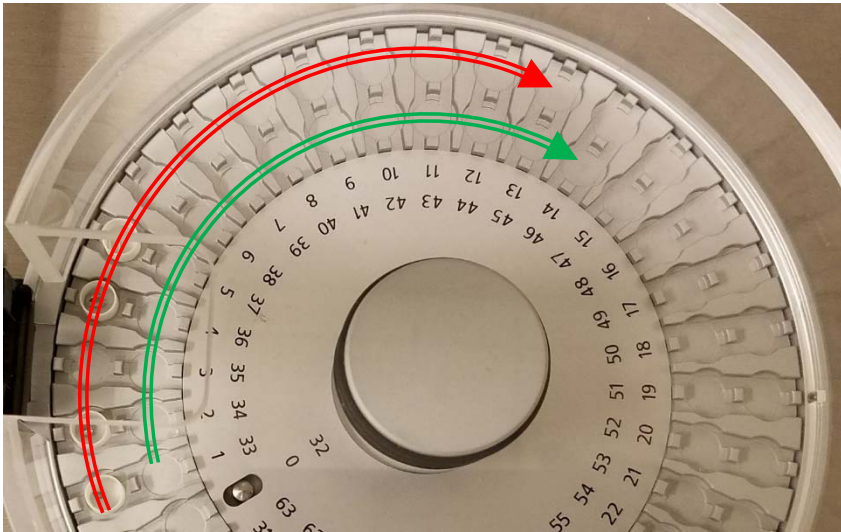
- I. Preparation
- II. Start
- III. Setup & Control
- IV. New Method (no baseline measurement)
- V. Opening Method
- VI. ASC Manager
- VII. Running Experiments
- VIII. Results
- IX. Cleanup
- X. Red Flags & Mistakes
- XI. Troubleshooting
- XII. Baseline Corrections
- XIII. OIT Experiments – coming soon

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**

TG 209F1 Libra
ECO
25.0 °C
Furnace closed (unlocked)

Setpoint
General Schedule

Settings are for TGA only!

General
Active Idle mode after measurement 60 min

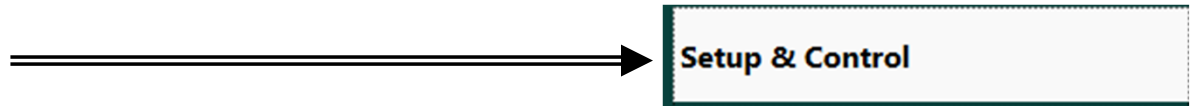
Idle mode
Cooling device No cooling
Temperature 25 °C
Heating Rate 20 K/min
Purge 1 MFC <no gas> Flow Rate 0 ml/min
Purge 2 MFC NITROGEN Flow Rate 50 ml/min
Protective MFC NITROGEN Flow Rate 10 ml/min

Eco mode
Cooling device No cooling
Temperature 25 °C
Heating Rate 20 K/min
Purge 1 MFC <no gas> Flow Rate 0 ml/min
Purge 2 MFC NITROGEN Flow Rate 50 ml/min
Protective MFC NITROGEN Flow Rate 10 ml/min

Apply Undo Defaults 7

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

A screenshot of a software interface showing a menu item labeled "Control" with a small icon of three people. Below it is a sub-menu item labeled "Signals" with a small icon of a signal. To the right of the "Signals" menu is a table displaying various signals and their values.

Temp.	25.5 °C
TG	0.0620 mg
P1(N2/O2)	0 ml/min
P2(N2)	0 ml/min
PG(N2)	10 ml/min
Vacuum	0.0 %

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**

A screenshot of a software interface showing a menu item labeled "Control" with a small icon of three people. Below it is a sub-menu item labeled "ASC Manual Control" with a small icon of a microscope. Below the "ASC Manual Control" menu is a form with the following fields:

Sample	---
Furnace	Closed
Mode	

Insert sample from position:

☐ Punch

Execute

Command done.
No sample in the furnace.

8

III. Setup & Control – 2/3

5. Review the different commands available below

Condition: **No sample in the furnace**

The screenshot shows a control interface with a left sidebar containing icons for Sample, Furnace, and Mode. The main area has a dropdown menu labeled 'Insert sample from position:' with a red box around it. Below the dropdown is a checkbox labeled 'Punch'. To the right is an 'Execute' button. At the bottom, a status bar displays 'Command done. No sample in the furnace.' with the latter part underlined in red.

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

Condition: **Sample in the furnace**

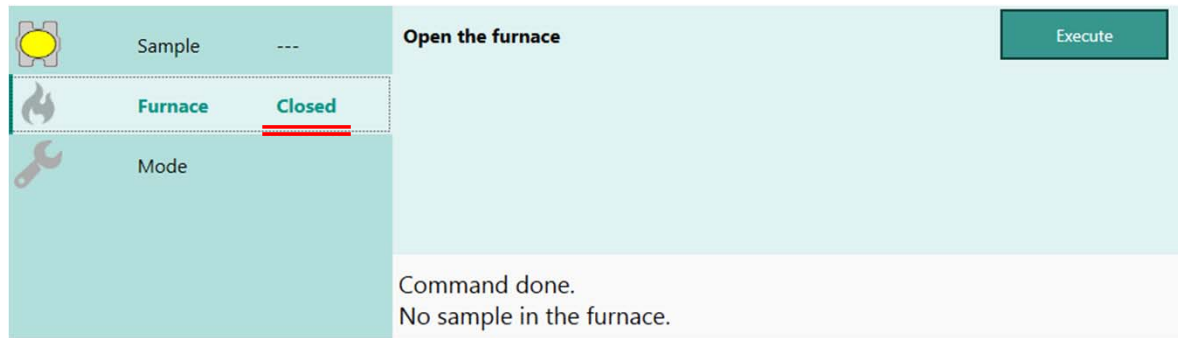
The screenshot shows a control interface with a left sidebar containing icons for Sample, Furnace, and Mode. The main area has two buttons: 'Clear the "Sample In" flag' and 'Remove sample from the furnace', each with an 'Execute' button to its right. At the bottom, a status bar displays 'Command done. Sample in the furnace.' with the latter part underlined in red.

- ***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

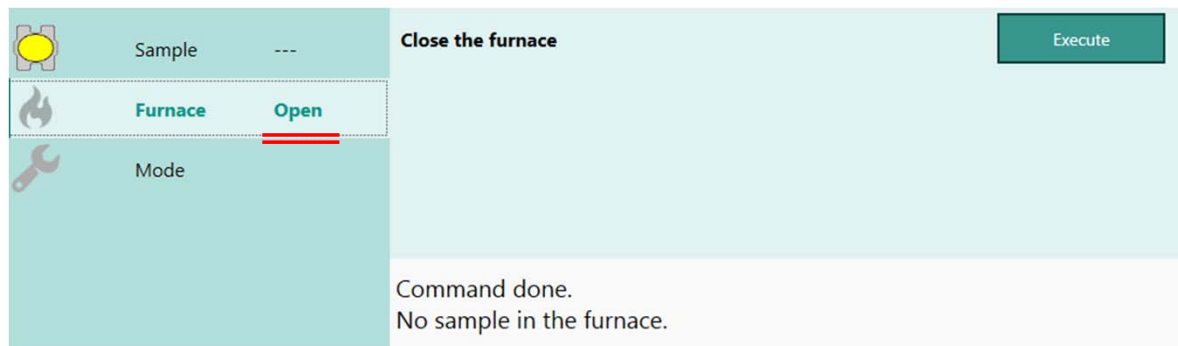
Condition: **Furnace is closed**



The screenshot shows a control panel with three sections: 'Sample' (with a yellow circle icon and a dropdown menu), 'Furnace' (with a flame icon and the word 'Closed' in red), and 'Mode' (with a wrench icon). To the right, the command 'Open the furnace' is displayed with an 'Execute' button. Below the command, a status message reads: 'Command done. No sample in the furnace.'

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

Condition: **Furnace is open**

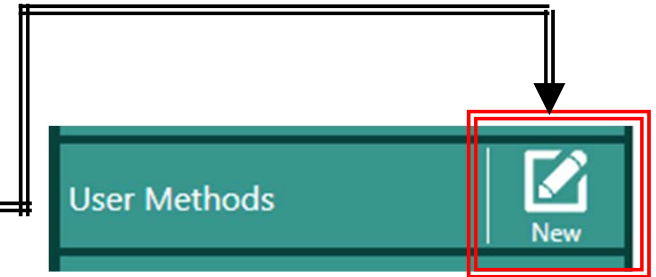


The screenshot shows the same control panel as above, but the 'Furnace' section now displays 'Open' in red. The command 'Close the furnace' is shown with an 'Execute' button. The status message at the bottom reads: 'Command done. No sample in the furnace.'

- ***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:



Method Definition - Create New Method

Setup | Header | Temperature Program | Calibrations

Property	Value	
Furnace(*)	Ceramic TG 209F1 TC: S (0 ... 1175 °C/ 300 K/min)	
Sample carrier (*)	TG 209F1 std (Al2O3 support) TC: P (-200 ... 1200 °C)	
Measurement mode	TG	
Crucible (*)	Al2O3 (... 1700 °C)	Help on crucible selection
Start criteria	7.5 K, Delay: 00:30 mm:ss Stability criteria enabled unconditionally: HR: 4.000 K/min, TG signal stability rate: 0.1000 mg/min Heat.: (30 K/min, 20 min), Cool.: (50 K/min, 30 min)	Modify start criteria
Control parameters	Furnace: Xp=4.00, Tn=4.00, Tv=4.00 Sample: Xp=4.00, Tn=4.00, Tv=4.00	Modify control parameters
Devices	MFCs AUTOVAC 400 (Rotary pump)	
TG BeFlat support (*)	On	
Super-Res (*)	Off	
Emergency temperature	Enhancement to maximum segment temperature: 25 K	Redefine enhancement

Current hardware temperature range is from 0 °C to 1175 °C

(*) Item has multiple possible values.
(?) Item is irrelevant to method definition (besides temperature range).

Legend
☐ inputs not complete ☒ inputs OK ☐ inputs must be verified ☐ page cannot be accessed ☐ inputs are not necessary

<- Backward OK Cancel Forward ->

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

Modify Start Criteria

Specific for temperature programs with initial start

Preheating rate: 30.0 K/min range <0.1 ... 50.0> K/min

Max. equilibrium time after preheating: 20 min range <1 ... 600> min

Precooling rate: 50.0 K/min range <0.1 ... 50.0> K/min

Max. equilibrium time after precooling: 30 min range <1 ... 600> min

Common for temperature programs with initial or initial standby start

Temperature stability threshold: 7.5 K range <0.1 ... 15.0> K

Stability criteria

☒ Activate check

Sample temperature stability rate: 0.1000 K/min range <0.005 ... 2.0> K/min

TG signal stability rate: 0.1000 mg/min range <0.0001 ... 2.0> mg/min

Start delay after stability: 00:30 mm:ss <00:00 ... 59:00> mm:ss

Stability criteria will be activated unconditionally if measured sample temperature is not within threshold otherwise activation depends on 'Activate check' state

OK Cancel

9. Click **OK** to accept settings

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

Legend

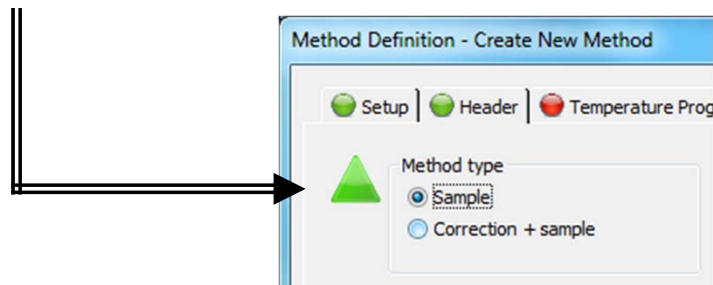
inputs not complete inputs OK inputs must be verified page cannot be accessed inputs are necessary

<- Backward OK Cancel Forward ->

IV. New Method – 3/9

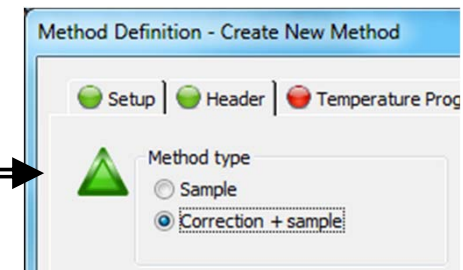
11. Select the desired **Method** type:

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



Device	Value
Purge 1 MFC	<no gas>
Purge 2 MFC	NITROGEN
Protective MFC	NITROGEN

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

The screenshot shows a 'Step Conditions' control panel with several settings. Red boxes and arrows highlight specific areas: a box around the 'STC' checkbox with an arrow from the text 'STC = Sample Temp. Controller'; a box around the 'Purge 1 MFC' section with an arrow from 'Air over sample = Purge 1 MFC'; a box around the 'Purge 2 MFC' section with an arrow from 'Nitrogen over sample = Purge 2 MFC'; and a box around the 'Protective MFC' section with an arrow from 'Protective MFC = Nitrogen over electronics'. The 'Flow active' checkboxes for Purge 2 and Protective MFC are checked. The flow rate for Purge 2 is set to 50 ml/min (labeled 'NITROGEN') and for Protective MFC is set to 10 ml/min (labeled 'NITROGEN'). The 'Purge 1 MFC' section shows 'AIR(80/20)' with a flow rate of 0 ml/min. A 'Vacuum' checkbox is at the bottom and is unchecked.

Condition	Flow active	Gas	Flow rate (ml/min)
STC	<input checked="" type="checkbox"/>	-	-
Purge 1 MFC	<input type="checkbox"/>	AIR(80/20)	0
Purge 2 MFC	<input checked="" type="checkbox"/>	NITROGEN	50
Protective MFC	<input checked="" type="checkbox"/>	NITROGEN	10
Vacuum	<input type="checkbox"/>	-	-

14. Enter in desired gas flowrates:

(Default) Purge 2 (or 1) Gas: **50 mL/min N₂** (or Air)

(Default) Protective: **10 mL/min N₂**

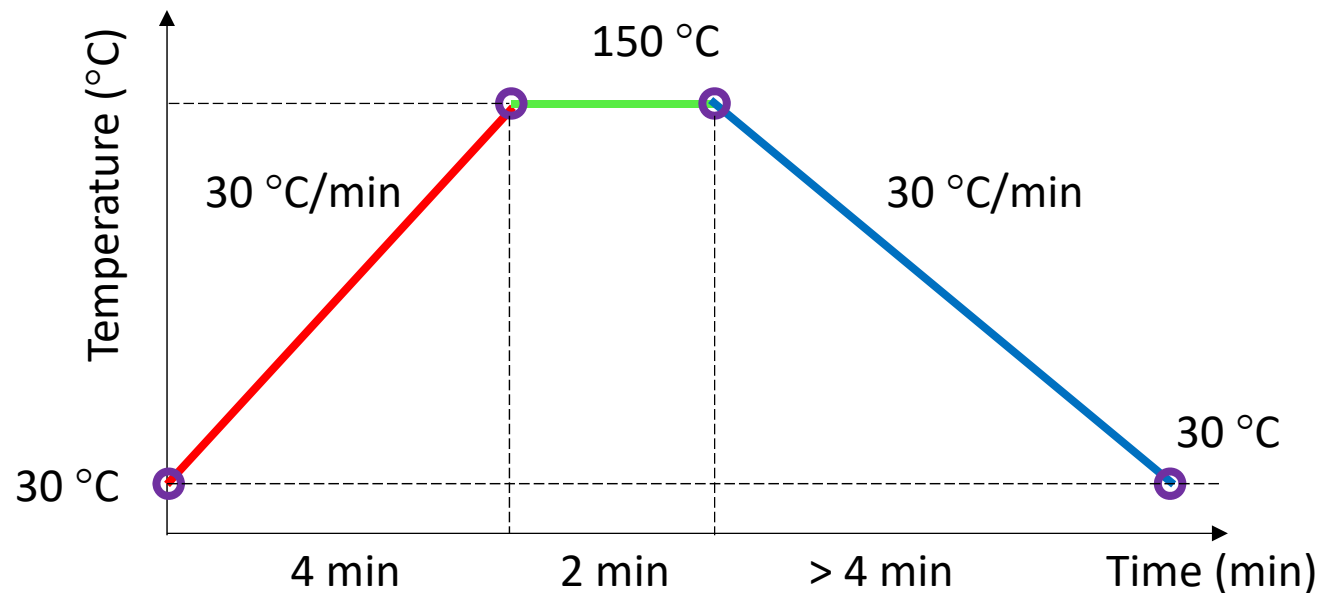
Protective must always \geq 10 mL/min N₂

IV. New Method – 5/9

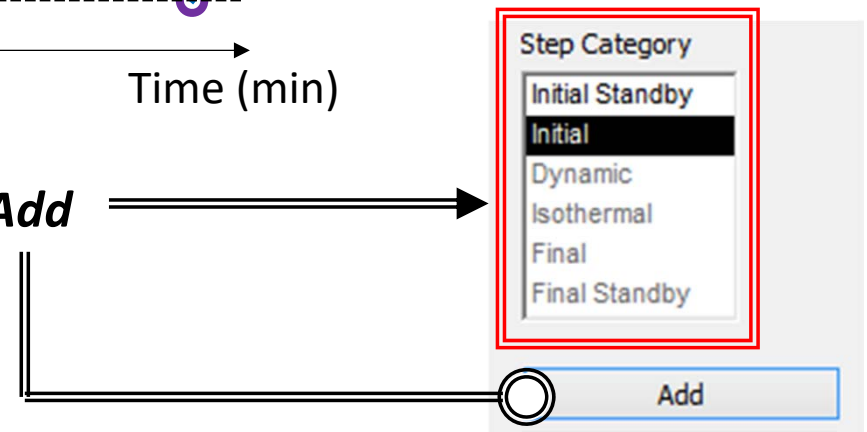
15. Build **Temperature Program** with desired **Step Categories**

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)

- Input Start temperature
Recommended temp = **25 – 30 °C**
- Check Use AUTOVAC Controller if you want to remove residual oxygen from chamber

The screenshot shows the 'Category' section with 'Start temperature' set to 20.0 °C. A checkbox for 'Use AUTOVAC Controller' is present and unchecked. On the right, the 'Step Category' list includes 'Initial Standby', 'Initial' (highlighted), 'Dynamic', 'Isothermal', 'Final', and 'Final Standby'.

18. For *Dynamic* step (changing temp.)

- Input End temperature
- Input Heating Rate or Cooling Rate

Note: Heating Rate MUST be equal to calibrated rates

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

The screenshot shows the 'Category' section with 'End temperature' set to 50.0 °C and 'Heating Rate' set to 5.000 K/min. Two acquisition rates are listed: 30.000 points/K and 150.000 points/min. On the right, the 'Step Category' list includes 'Initial Standby', 'Initial', 'Dynamic' (highlighted), 'Isothermal', 'Final', and 'Final Standby'.

The screenshot shows the 'Category' section with 'End temperature' set to 25.0 °C and 'Cooling Rate' set to 5.000 K/min. Two acquisition rates are listed: 30.000 points/K and 150.000 points/min. On the right, the 'Step Category' list includes 'Initial Standby', 'Initial', 'Dynamic' (highlighted), 'Isothermal', 'Final', and 'Final Standby'.

19. For *Isothermal* step (constant temp.)

- Input Isothermal time
- Input Acquisition rate
(default values will be automatically inserted)

The screenshot shows the 'Category' section with 'Isothermal time' set to 00:05 hh:mm and an acquisition rate of 50.00 points/min. On the right, the 'Step Category' list includes 'Initial Standby', 'Initial', 'Dynamic', 'Isothermal' (highlighted), 'Final', and 'Final Standby'.

IV. New Method – 7/9

20. Add **Final** step (Auto filled)

- Input Emergency Reset Temp
(default values will be automatically inserted)

Category
Emergency Reset Temp: 60.0 °C

Step Category
Initial Standby
Initial
Dynamic
Isothermal
Final
Final Standby

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

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

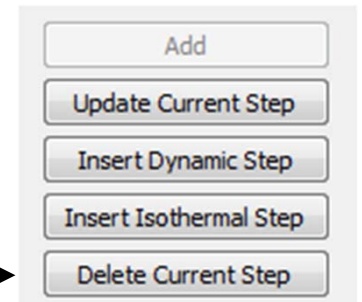
Nr	Type	°C	K/min	Time	pts/min	pts/K	STC	N2/O2	N2	N2	Vac
1	Yellow circle	30.0			0x E+F		✓	0	50	10	
2	Red arrow	150.0	30.000	0:04:00	300.00	10.00	✓	0	50	10	
3	Green arrow	150.0		0:02:00	150.00		✓	0	50	10	
4	Blue arrow	30.0	30.000	0:04:00	300.00	10.00	✓	0	50	10	
5	Red plus	160.0					✓	0	50	10	

Annotations:

- Sample Temperature Controller (points to STC column)
- Air Purge Gas Flowrate (points to N2/O2 column)
- Purge 2 N₂ Gas Flowrate (points to N2 column)
- Type of Step (points to Type column)
- Temp (points to °C column)
- Heat/Cooling Rate (points to K/min column)
- Time for Step (points to Time column)
- Acquisition Rate (points to pts/min and pts/K columns)
- Protective Gas Flowrate (points to N2 column)

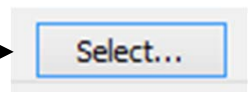
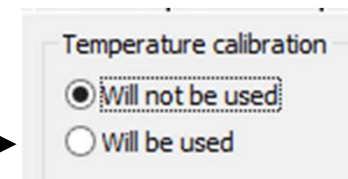
IV. New Method – 8/9

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** 



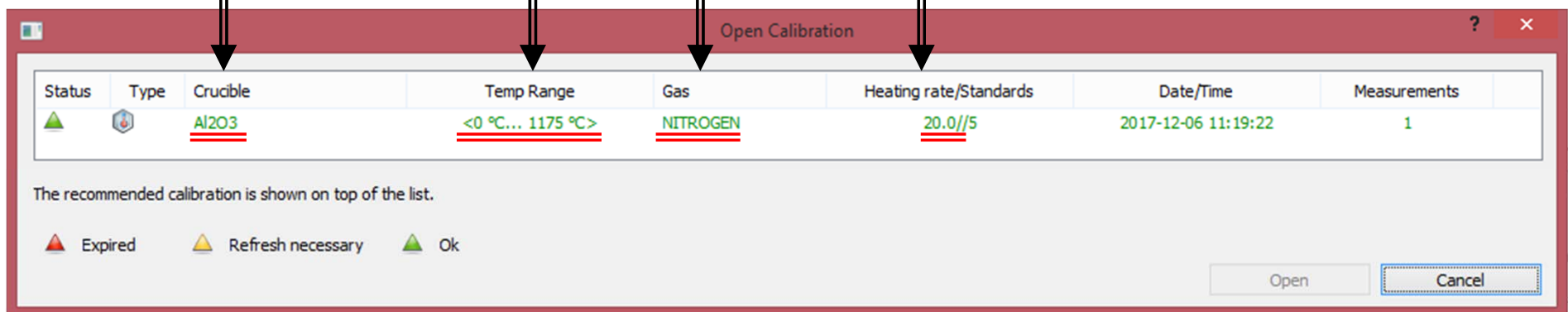
25. Select correct **Calibration File**, checking **EVERY** condition is correct:

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

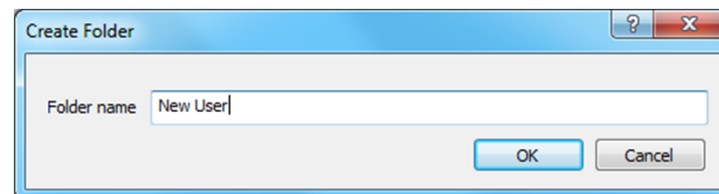
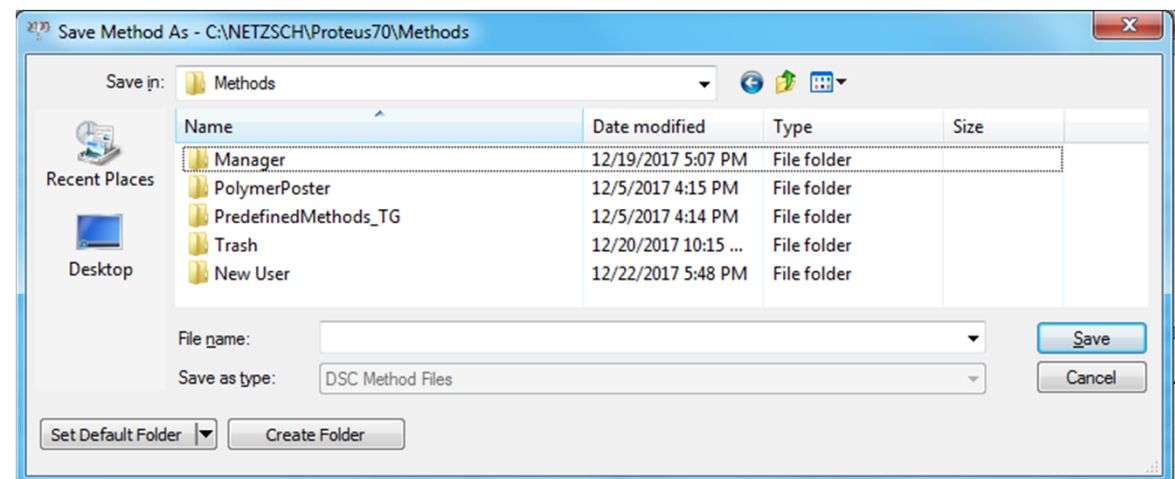
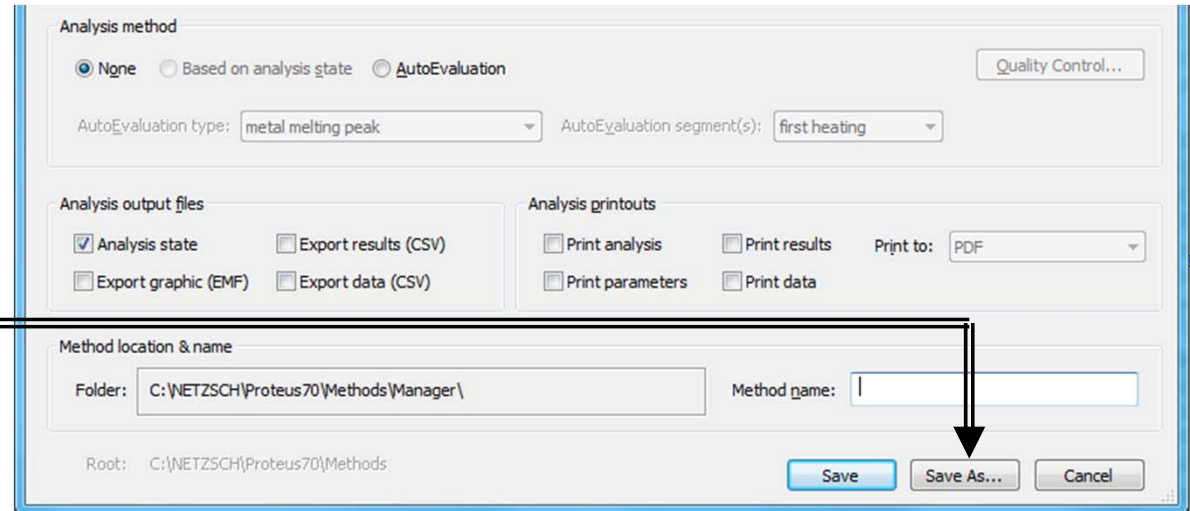
26. Click **Forward** -> to advance

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:\Netsch\Proteus70\Methods\"PI NAME\"YOUR FOLDER"
3. Enter **Required Information** such as:

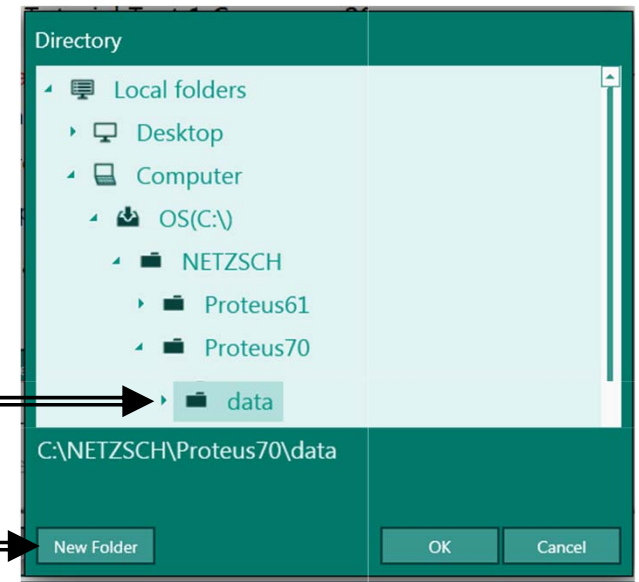


Sample ID
Sample Name
File Name

A form titled 'Basic data' with a red triangle icon. It contains several input fields: 'Laboratory' (Optional), 'Project' (Optional), 'Operator Name' (with a dropdown menu showing 'Manager'), 'Material' (with a dropdown menu showing 'Empty'), 'Sample ID' (Required), 'Sample Name' (Required), 'Directory' (with the text 'C:\NETZSCH\Proteus70\data'), 'File Name' (Required), and 'Remark' (Optional). Red boxes highlight the 'Sample ID', 'Sample Name', 'Operator Name', 'Directory', and 'File Name' fields. A folder icon is circled in red on the right side of the form.

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 °C**)

Sample

Autosampler Position: 0

Sample Crucible: Al2O3 1700 °C

Sample Mass: Later

Sample Crucible Mass: by intl. blnc.

☒ Do hole

Additional Info

Just before measurement

Manual

Just before measurement

Prewrite all positions

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

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

Additional Info

Calibrations

Temperature calibration: <0 °C... 1175 °C>, Crucible: Al2O3, Gas: NITROGEN

TG BeFlat will be used

Gases Info

Purge 1 MFC Gas does not matter

Purge 2 MFC NITROGEN flow: 50 ml/min

Protective MFC NITROGEN flow: 10 ml/min

Start Criteria

Sample temperature stability threshold 7.5 K

Sample temperature stability rate 0.1 K/min

Start delay after stability 00:00:30 hh:mm:ss

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

Temperature Program

Nr	Type	°C	K/min	Duration	pts/min	pts/K	STC	AIR(80/20)	NITROGEN	NITROGEN
0	●	30					<input checked="" type="checkbox"/>		50 ml/min	10 ml/min
1	↗	150	30	00:04:00	300	10	<input checked="" type="checkbox"/>		50 ml/min	10 ml/min
2	→	150		00:02:00	150		<input checked="" type="checkbox"/>		50 ml/min	10 ml/min
3	↘	30	30	00:04:00	300	10	<input checked="" type="checkbox"/>		50 ml/min	10 ml/min
4	●	160					<input checked="" type="checkbox"/>		50 ml/min	10 ml/min

■ Purge 1 MFC ■ Purge 2 MFC ■ Protective MFC

Temperature Program

Time 00:10 hh:mm

Add to ASC

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**)

Autosampler

Configuration Sample Tray Execution list Sample Tray State

— Sample Tray Management —

Open ... Save As ... Restore

⤴ General

Crucible insertion temperature threshold **5** °C

Max removal temperature: **100** °C

☐ Activate alternative delay

Alternative equilibration delay: **20** min

If alternative equilibration delay is activated then measurement in autosampler mode will start whatever comes first:
- start criteria defined in method or
- alternative equilibration delay defined above.

Final removal: **Remove Crucible**

Weighing mode: Just before measurement

⤴ Devices configurations

MFCs, AUTOVAC 400 (Rotary pump)

⤴ Weigh Just Before Measurement Settings

Options valid only for sample and correction+sample measurements

Stability criteria

☒ Time only

Equilibrium time after sample insertion **1** min

☐ TG signal stability and time

TG stability for autostart **1** µg/min

Maximum waiting time for TG stability **10** min







Options for empty crucibles weighing process


Alternative weighing temperature **25** °C

Insertion temperature threshold **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

Configuration	Sample Tray	Execution list	Sample Tray State
	<div><div>0</div><div> Sample</div><div>Al2O3 (1700 °C)</div><div>Name: Drierite</div><div>File: Drierite 2018 03 09.ngb-st9</div><div>by intl. blnc. later</div></div>	<div><div>1</div><div>Empty</div></div>	<div><div>2</div><div>Empty</div></div>

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

Configuration	Sample Tray	Execution list	Sample Tray State
<div>  <div> <div>Position</div> <div>0</div> </div> <div> <div>Sample</div> <div>Later</div> </div> <div> <div>Crucible</div> <div>by intl. blnc.</div> </div> </div>	<div> <div>Sample name</div> <div>Drierite</div> </div> <div> <div>Sample ID</div> <div>Drierite</div> </div> <div> <div>Crucible</div> <div>Al₂O₃ (1700 °C)</div> </div> <div> <div>Step status</div> <div>Not yet run</div> </div>	<div> <div>Method</div> <div>Drierite_Al₂O₃_20K_N2 30 min.ngb-s-tg</div> </div> <div> <div>Measurement file</div> <div>Drierite 2018 03 09.ngb-st9</div> </div> <div> <div>Analysis</div> </div>	

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

Configuration Sample Tray Execution list **Sample Tray State**

Position	Sample ID	Sample Name	Measurement Source	File Name	Crucible	Crucible Mass [mg]	Sample
0	Drierite	Drierite	Drierite_Al2O3_20K_N2 30 min.ngb-s-tg	Drierite 2018 03 09.ngb-st9	Al2O3	by intl. blnc.	Later

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!**

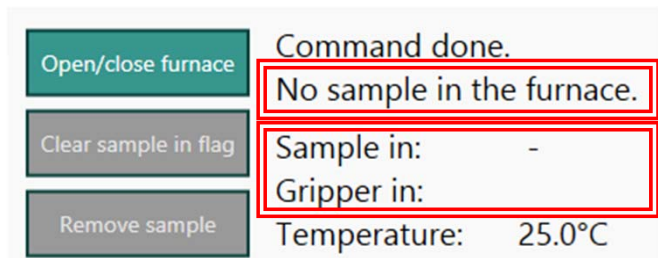
Measurements: 0 ready for execution. Positions: 1 with not yet measured crucible mass

Sample 0: Measuring Crucible Mass...

The first run for crucible mass determination needs operator to check sample status! If necessary remove it or 'Clear' corresponding 'Flag'.
When ready press OK to continue...

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

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

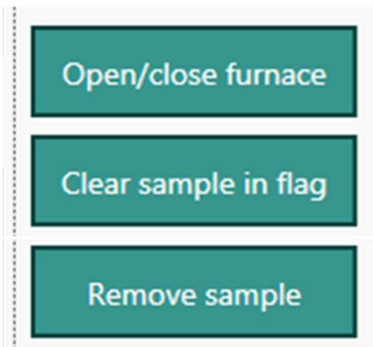


Things to consider:

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

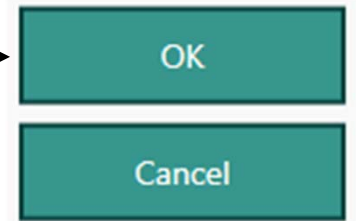
VII. Running Experiments – 2/5

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

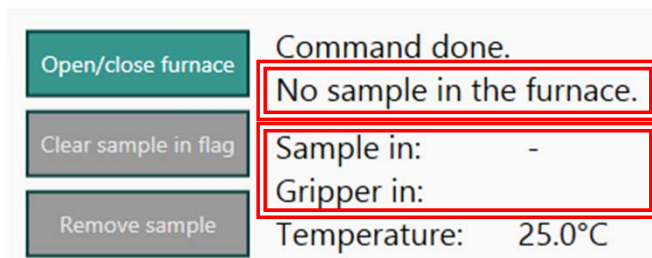


12. Proceed to review the notes described here **IMPORTANT!**

Measurements: 0 ready for execution. Positions: 0 with not yet measured crucible mass
Measurement - Method 'Drierite_Al2O3_20K_N2 30 min.ngb-s-tg' Measurement ASC Operation: Sample 0
The first run needs operator to check sample status! If necessary remove it or 'Clear' corresponding 'Flag'.
When ready press OK to continue...

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

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

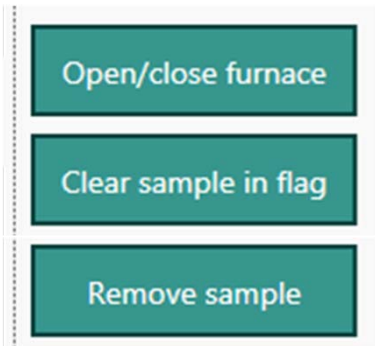


Things to consider:

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

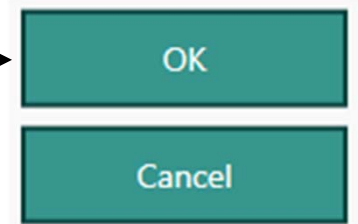
VII. Running Experiments – 4/5

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



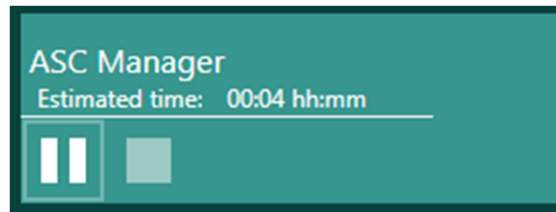
16. Follow any instructions or prompts that appear

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



Preheating\precooling criteria:

Threshold: 50.0 K, Current difference: -0.3 K

Stabilization delay. Remaining time: 0:00 mm:ss

⤴ Weighing process

Wait for achieving stable sample mass value. Rema

Sample Mass 9.4793 mg

DTG signal 0.0000 µg/min

⤴ Signals

TG 150.8409 mg

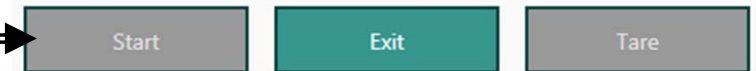
P1 0 digits

P2(N2) 20 ml/min

PG(N2) 10 ml/min

Vacuum 0.0 %

20. The **current temperature** and **segment action** is shown here



21. Active measurement can be shown here

TG 209F1 Libra

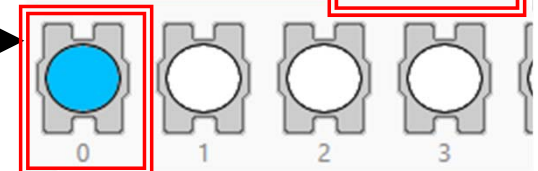
ASC

S:0

Segment: 25.2 °C

2h 44min 28s

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

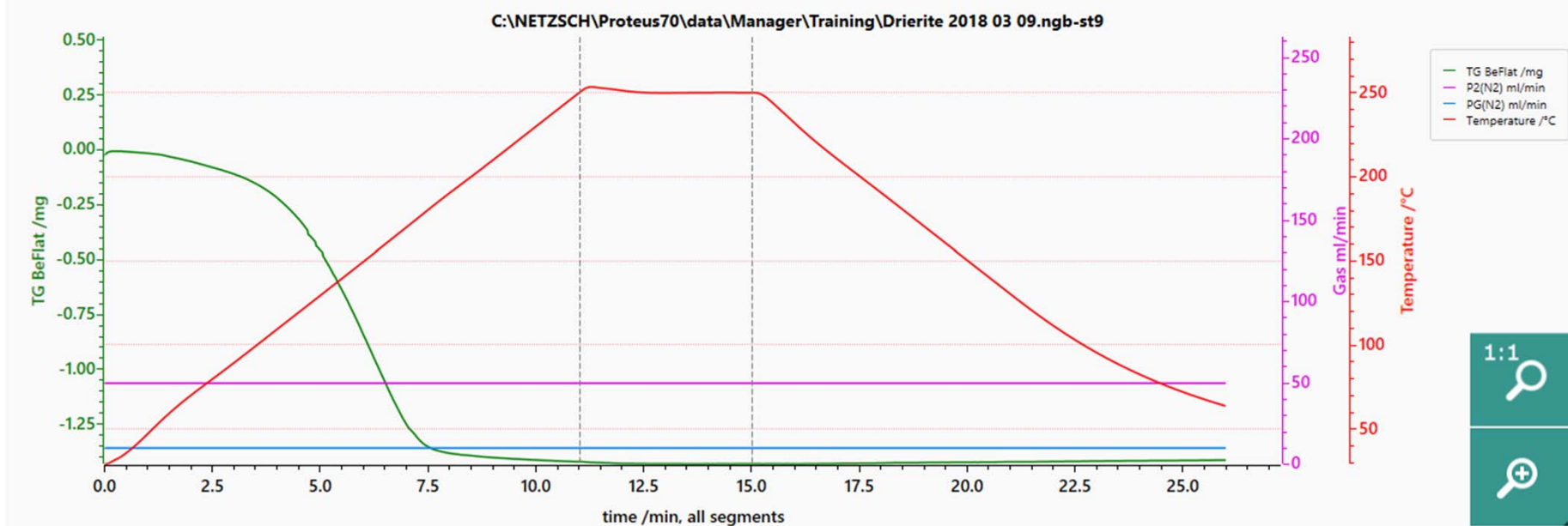
ASC Manager
Estimated time: 00:00 hh:mm
Switch OFF

Configuration	Sample Tray	Execution list	Sample Tray State
Position	0	Sample name	Drierite
		Sample ID	Drierite
Sample	26.3575 mg	Crucible	Al2O3 (1700 °C)
Crucible	139.0119 mg	Finished	3/9/2018 12:05:21 PM

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

Measurement

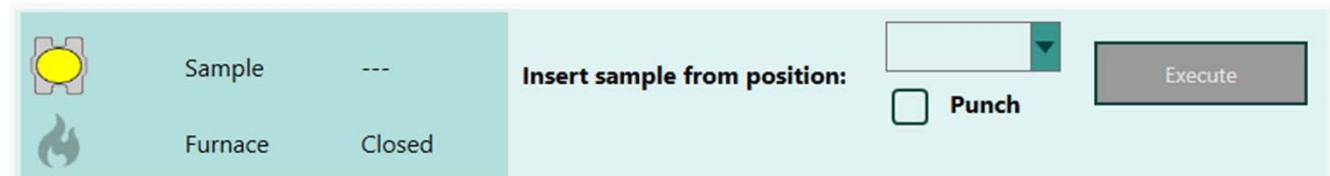
Measurement - Method 'Drierite_Al2O3_20K_N2 30 min.ngb-s-tg' (finished)



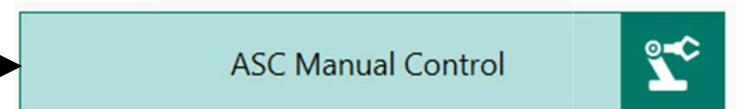
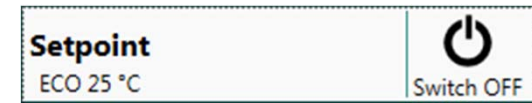
IX. Clean Up – 1/1

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:

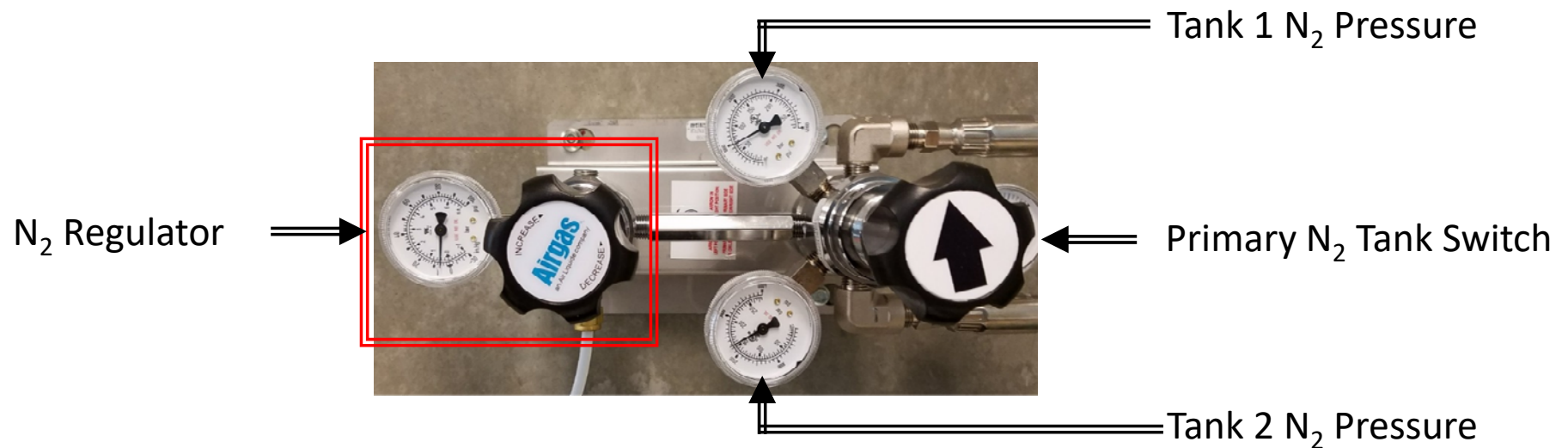
- **No Sample**
- **Furnace is Closed**



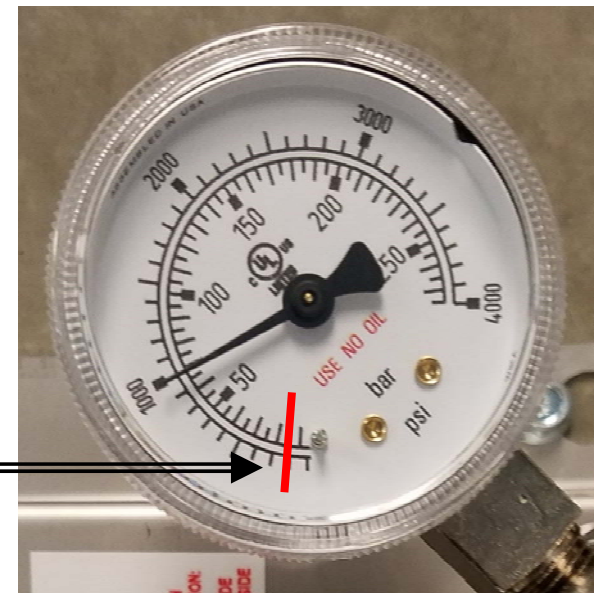
6. When TGA temperature reaches **25 ± 5 °C**, click the **X** to close the software
7. Confirm that you wish to **keep the Setpoint ON** after you close software
8. **Log out** of your ENGR account
9. Clean up the lab bench and place all items back in their respective drawers
10. Sign out on the **Sign-In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



X. Red Flags & Mistakes – 1/3

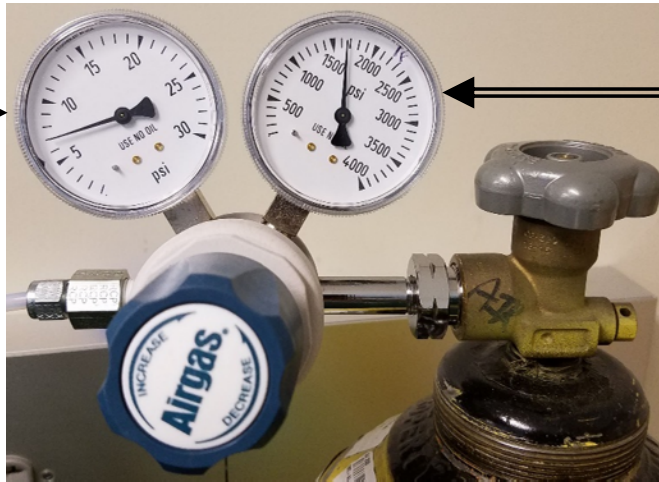


1. DO NOT ADJUST THE REGULATOR AS THIS MAY DAMAGE MASS FLOW CONTROLLERS \$\$\$
2. Check if Tank 1 or 2 N₂ pressure is at least **200 psi**, else contact Lab Manager to replace tank



XI. Red Flags & Mistakes – 2/3

Regulated Air
Pressure \approx 7 psi



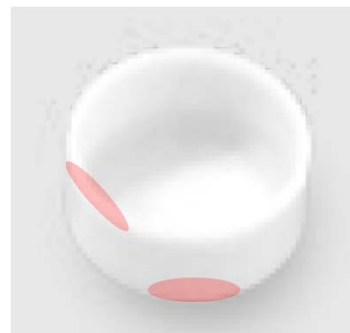
Air Tank Pressure

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 \$\$\$



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



TGA thermocouple

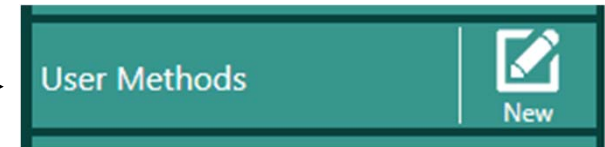


7. If ASC sample changer makes a noise while switching samples, report to Lab Manager immediately!

XI. Baseline Corrections – 1/6

The following are modifications to perform a baseline correction

1. Click on New next to User Methods



Method Definition - Create New Method

Setup | Header | Temperature Program | Calibrations

Property	Value	
Furnace(?)	Ceramic TG 209F1 TC: S (0 ... 1175 °C/ 300 K/min)	
Sample carrier (*)	TG 209F1 std (Al2O3 support) TC: P (-200 ... 1200 °C)	
Measurement mode	TG	
Crucible (*)	Al2O3 (... 1700 °C)	Help on crucible selection
Start criteria	7.5 K, Delay: 00:30 mm:ss Stability criteria enabled unconditionally: HR: 4.000 K/min, TG signal stability rate: 0.1000 mg/min Heat.: (30 K/min, 20 min), Cool.: (50 K/min, 30 min)	Modify start criteria
Control parameters	Furnace: Xp=4.00, Tn=4.00, Tv=4.00 Sample: Xp=4.00, Tn=4.00, Tv=4.00	Modify control parameters
Devices	MFCs, AUTOVAC 400 (Rotary pump)	
TG BeFlat support (*)	On	
Super-Res (*)	Off	
Emergency temperature	Enhancement to maximum segment temperature: 25 K	Redefine enhancement

Current hardware temperature range is from 0 °C to 1175 °C

(*) Item has multiple possible values.
(?) Item is irrelevant to method definition (besides temperature range).

Legend
● inputs not complete ● inputs OK ● inputs must be verified ● page cannot be accessed ● inputs are not necessary

<- Backward OK Cancel Forward ->

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

Legend
☐ inputs not complete ☒ inputs OK ☐ inputs must be verified ☐ page cannot be accessed ☐ inputs are not necessary

<- Backward OK Cancel Forward ->

Method Definition - Create New Method

Setup | Header | Temperature Program

Method type
☐ Sample
☒ Correction + sample

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		<input checked="" type="checkbox"/>	50	10	<input type="checkbox"/>
2		250.0	20.000	0:11:00	300.00	15.00	<input checked="" type="checkbox"/>	50	10	<input type="checkbox"/>
3		250.0		0:04:00	75.00		<input checked="" type="checkbox"/>	50	10	<input type="checkbox"/>
4		30.0	20.000	0:11:00	300.00	15.00	<input checked="" type="checkbox"/>	50	10	<input type="checkbox"/>
5		275.0					<input checked="" type="checkbox"/>	50	10	<input type="checkbox"/>

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...

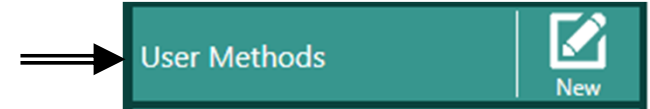
Method Definition - Create New Method

Setup | Header | Temperature Program | Calibrations

Temperature calibration
☒ Will not be used
☐ Will be used

XI. Baseline Corrections – 3/6

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

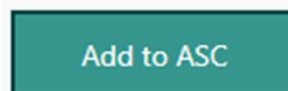


13. You will have to perform a baseline measurement first (unless you have already ran it)

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

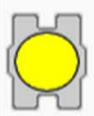
A screenshot of a software interface for baseline corrections. The 'Basic data' section includes fields for Laboratory, Project, Operator Name (set to 'Manager'), and Material (set to 'Drierite'). A 'Clear basic data' button is below these fields. The 'Sample' section includes a radio button for 'Measure baseline' (which is selected), a radio button for 'Measure baseline+sample', and a 'Baseline status' indicator showing 'Baseline must be performed first'. Below this, the 'Autosampler Position' is set to '0', the 'Sample Crucible' is 'Al2O3 1700 °C', and the 'Sample Crucible Mass' is 'by intl. blnc.' with a unit of 'mg' and a dropdown set to 'Just before measurement'. Red boxes highlight the 'Sample ID' and 'Sample Name' fields in the 'Basic data' section, and the 'Autosampler Position' field in the 'Sample' section. Arrows from the instructions point to the 'Measure baseline' radio button and the 'Autosampler Position' field.

15. Add to ASC at the bottom



XI. Baseline Corrections – 4/6

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

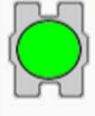
Configuration	Sample Tray	Execution list	Sample Tray State
	Position	0	Sample name Drierite Method Drierite_Al2O3_20K_N2_30 min_Correction_NoTGBFlat.ngb-s-tg.ngb-d-tg
		Sample ID Drierite	Measurement file <u>Baseline measurement</u>
	Sample	0 mg	Crucible Al2O3 (1700 °C) Analysis
	Crucible	<u>by intl. blnc.</u>	Step status Not yet run

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



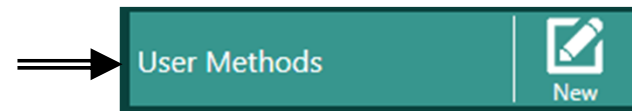
18. Proceed to review the notes and correct all issues as before... Click **OK**

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

Configuration	Sample Tray	Execution list	Sample Tray State
	Position	0	Sample name Drierite Method Drierite_Al2O3_20K_N2_30 min_Correction_NoTGBFlat.ngb-s-tg.ngb-d-tg
		Sample ID Drierite	Measurement file <u>Baseline measurement</u>
	Sample	0 mg	Crucible Al2O3 (1700 °C) Analysis
	Crucible	<u>139.0089 mg</u>	Finished <u>3/9/2018 4:09:44 PM</u>

XI. Baseline Corrections – 5/6

20. Click **User Methods** again



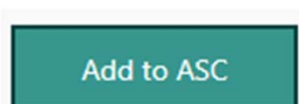
21. You can now create a **File Name** for your sample

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

A screenshot of a software interface for sample data entry. The "Basic data" section is active, showing fields for Laboratory (Optional), Project (Optional), Operator Name (Manager), and Material (Drierite). To the right, Sample ID is Drierite, Sample Name is Drierite, and Directory is C:\NETZSCH\Proteus70\data\Man. The File Name field is highlighted with a red box and contains the text "tion Sample 2018 03 09 No BeFlat". Below these fields are radio buttons for "Measure baseline" and "Measure baseline+sample", with the latter being selected. A "Clear basic data" button is also present. The "Sample" section shows the Autosampler Position as 1, which is also highlighted with a red box. A "Remark" field is optional and empty. The baseline status is indicated as "Baseline file is present." with a green circle icon.

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

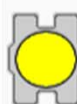
25. Ensure your next measurement is a **sample measurement**

Configuration

Sample Tray

Execution list

Sample Tray State



Position	1	Sample name	Drierite	Method	Drierite_Al2O3_20K_N2_30 min_Correction_NoTGBFlat.ngb-s-tg.ngb-d-tg
		Sample ID	Drierite	Measurement file	<u>Drierite Correction Sample 2018 03 09 No BeFlat.ngb-dt9</u>
Sample	Later	Crucible	Al2O3 (1700 °C)	Analysis	
Crucible	<u>by intl. blnc.</u>	Step status	Not yet run		

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**

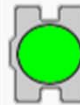
28. After the Empty Crucible is weighed, click **Start** to perform the **sample measurement...**

Configuration

Sample Tray

Execution list

Sample Tray State



Position	1	Sample name	Drierite	Method	Drierite_Al2O3_20K_N2_30 min_Correction_NoTGBFlat.ngb-s-tg.ngb-d-tg
		Sample ID	Drierite	Measurement file	<u>Drierite Correction Sample 2018 03 09 No BeFlat.ngb-dt9</u>
Sample	27.7111 mg	Crucible	Al2O3 (1700 °C)	Analysis	
Crucible	139.0068 mg	Finished	3/9/2018 4:56:36 PM		

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

