

Netzsch DSC 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 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 μ L
 - 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***



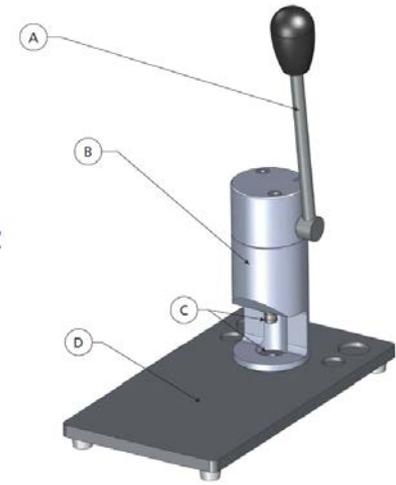
I. Preparation – 2/3

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

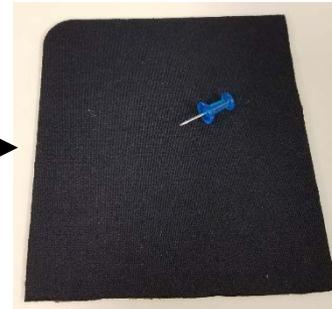
Using **Sealing Press** for any other pan will **DAMAGE** Press! ⇒



Type of crucible:

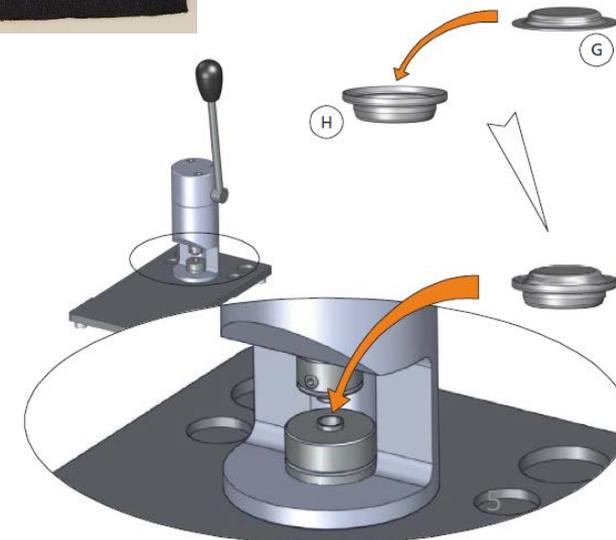


5. Punch a tiny hole onto the lid using the provided push-pin + pad ⇒



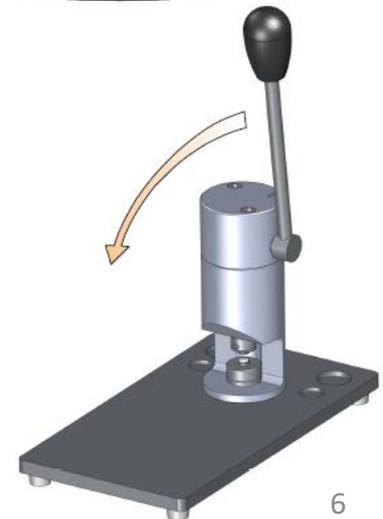
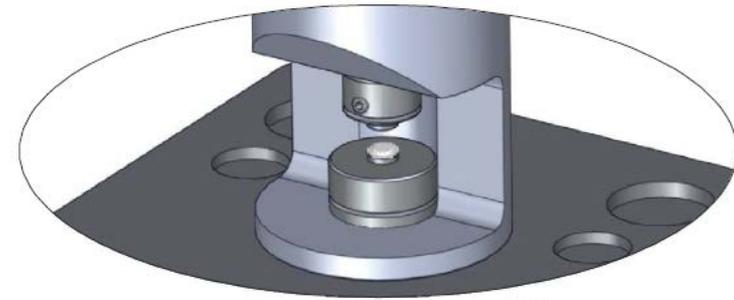
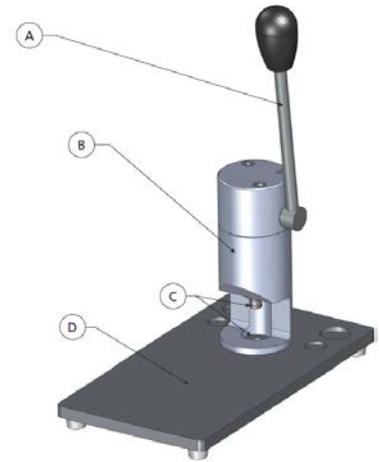
6. Place pan into the bottom part (C) of the toolkit carefully

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!)

DSC 214 Polyma (DSC21400A-0227-L)

Current setpoint configuration:

Setpoint mode:	ECO
Cooling Device:	No cooling
Temperature:	25 °C
Heating Rate:	20 K/min
Purge 2 MFC:	NITROGEN
Protective MFC:	NITROGEN

Do you want to start Setpoint now?

Setpoint
OFF

 Switch ON

Setpoint
ECO 25 °C

 Switch OFF

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

II. Start – 2/2

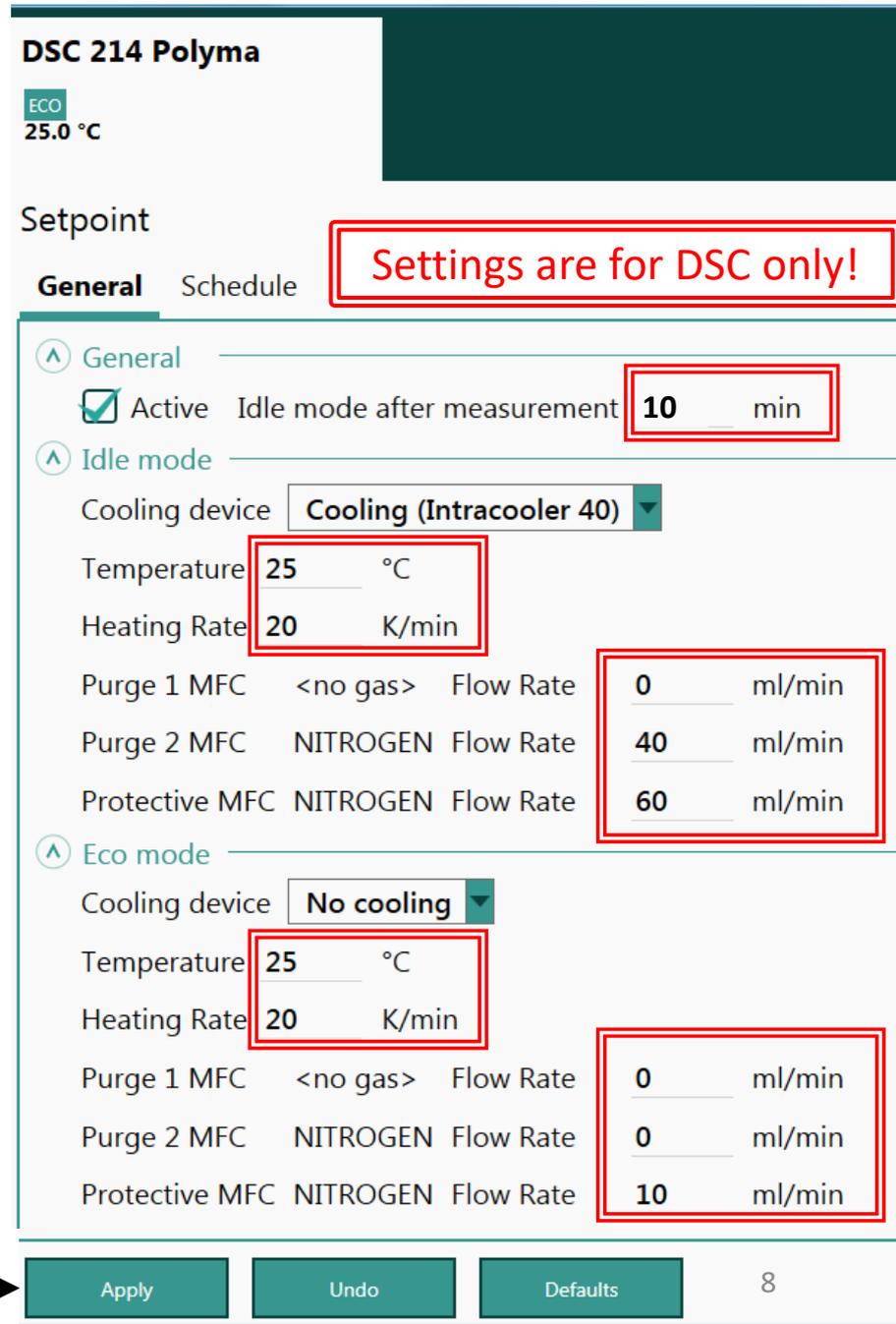
5. Click on **Setpoint**



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**



DSC 214 Polyma
ECO
25.0 °C

Setpoint

Settings are for DSC only!

General Schedule

General

- Active Idle mode after measurement **10** min

Idle mode

- Cooling device **Cooling (Intracooler 40)**
- Temperature **25** °C
- Heating Rate **20** K/min
- Purge 1 MFC <no gas> Flow Rate **0** ml/min
- Purge 2 MFC NITROGEN Flow Rate **40** ml/min
- Protective MFC NITROGEN Flow Rate **60** ml/min

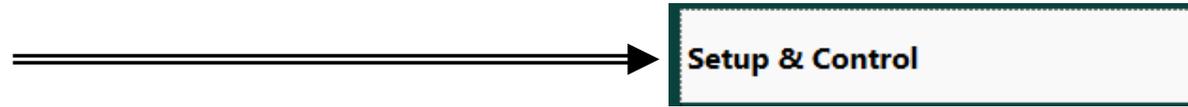
Eco mode

- 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 **0** ml/min
- Protective MFC NITROGEN Flow Rate **10** ml/min

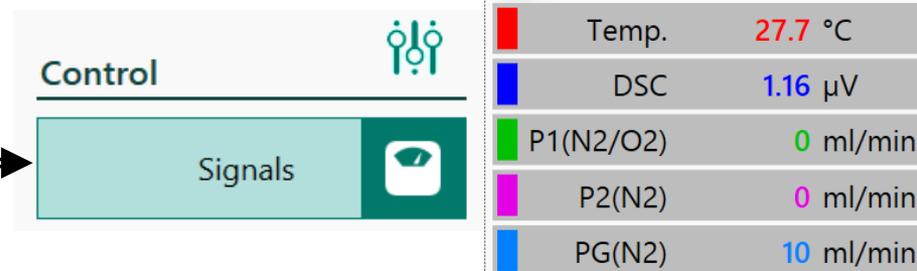
Apply Undo Defaults 8

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



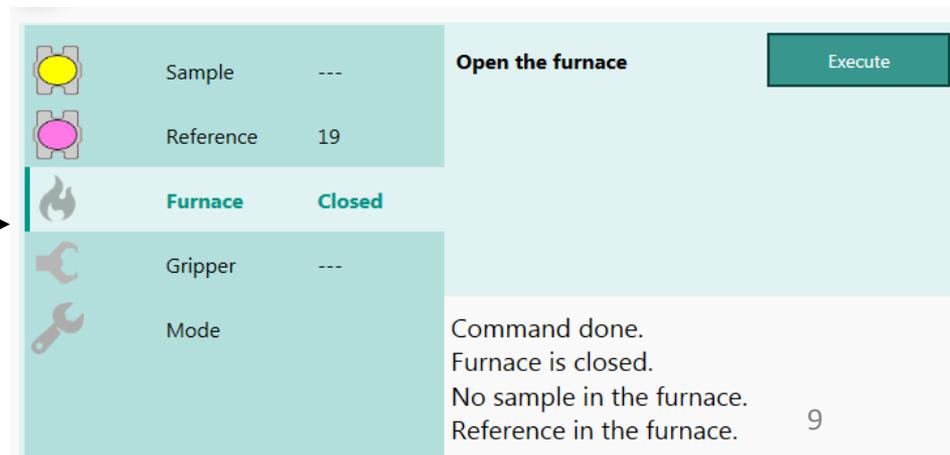
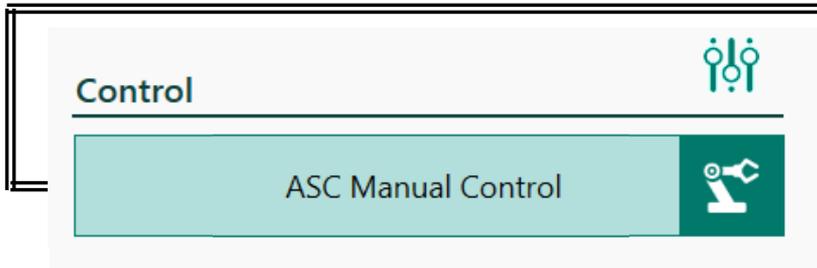
A screenshot of the 'Signals' pop-up window. It features a 'Control' header with a robot icon and a 'Signals' button with a monitor icon. To the right is a table of sensor readings:

Temp.	27.7 °C
DSC	1.16 μV
P1(N2/O2)	0 ml/min
P2(N2)	0 ml/min
PG(N2)	10 ml/min

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 the 'ASC Manual Control' menu. It lists several control options with their current status:

Sample	---	Open the furnace	Execute
Reference	19		
Furnace	Closed		
Gripper	---		
Mode			

Below the menu, a confirmation message is displayed: 'Command done. Furnace is closed. No sample in the furnace. Reference in the furnace.'

III. Setup & Control – 2/4

4. Review the different commands available below

Condition: **No sample in the furnace**

The screenshot shows a control panel with a sidebar on the left containing icons and labels for Sample, Reference, Furnace, Gripper, and Mode. The main area displays the command 'Insert sample from position:' with a dropdown menu showing '1' (highlighted with a red box) and an 'Execute' button. Below the command area, the status is shown as 'Command done. Furnace is closed. No sample in the furnace. No reference in the furnace.'

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

Condition: **Sample in the furnace**

The screenshot shows the control panel with the 'Sample' status set to '1'. The main area displays two commands: 'Clear the "Sample In" flag' and 'Remove sample from the furnace', each with an 'Execute' button. Below the commands, the status is shown as 'Command done. Furnace is opened. Sample in the furnace. No reference 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 – 3/4

4. Review the different commands available below

Condition: **No reference in the furnace**

The screenshot shows a control panel with a left sidebar and a main area. The sidebar contains icons and labels for Sample, Reference, Furnace, Gripper, and Mode. The main area has a dropdown menu for 'Insert reference from position:' with the value '19' selected and highlighted by a red box. An 'Execute' button is to the right. Below the main area, a status message reads: 'Command done. Furnace is opened. No sample in the furnace. No reference in the furnace.'

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

Condition: **Reference in the furnace**

The screenshot shows the same control panel. The 'Reference' field in the sidebar now shows '19'. The main area has two buttons: 'Clear the "Reference In" flag' and 'Remove reference from the furnace', both with 'Execute' buttons. The status message reads: 'Command done. Furnace is opened. No sample in the furnace. Reference in the 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**

	Sample	---	Open the furnace	
	Reference	---		
	Furnace	<u>Closed</u>		
	Gripper	---		
	Mode	---		
			Command done. Furnace is closed. No sample in the furnace. No reference in the furnace.	

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

Condition: **Furnace is open**

	Sample	---	Close the furnace	
	Reference	---		
	Furnace	<u>Open</u>		
	Gripper	---		
	Mode	---		
			Command done. Furnace is opened. No sample in the furnace. No reference 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:



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**

Method Definition - Create New Method

Setup | Header | Temperature Program | Calibrations

Property	Value	
Furnace(²)	Arena DSC 214 TC: E (-195 ... 605 °C/ 500 K/min)	
Sample carrier(²)	DSC 214 Corona sensor TC: E (-200 ... 605 °C)	
Measurement mode	DSC	
Crucible (¹)	Concavus Pan Al, pierced lid (... 610 °C)	Help on crucible selection
Start criteria	5.0 K, HR: 0.100 K/min, Delay: 00:30 mm:ss Heat.: (30 K/min,20 min), Cool.: (50 K/min,30 min)	Modify start criteria
Control parameters	Furnace: Xp=5.00, Tn=5.00, Tv=4.00 Sample: Xp=5.00, Tn=4.00, Tv=4.00	Modify control parameters
Devices	Cooling (Intracooler 40), MFCs	
Automatic cooling (¹)	On	
O.I.T. (¹)	Off	
Emergency temperature	Enhancement to maximum segment temperature: 10 K	Redefine enhancement

Current hardware temperature range is from -50 °C to 605 °C

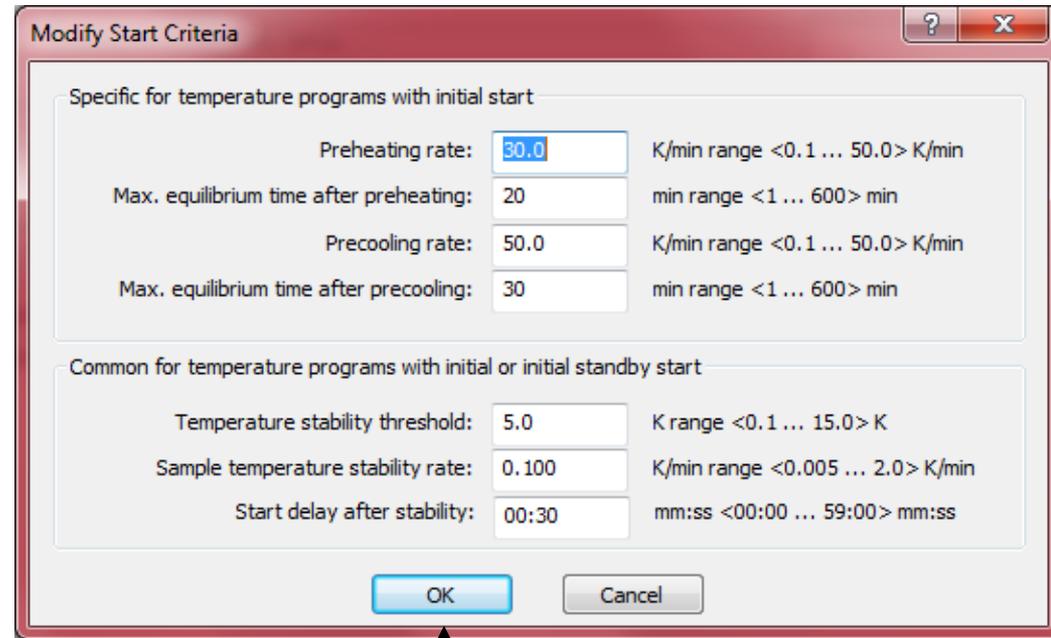
(¹) Item has multiple possible values.
(²) Item is irrelevant to method definition (besides temperature range).

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

<- Backward OK Cancel Forward ->

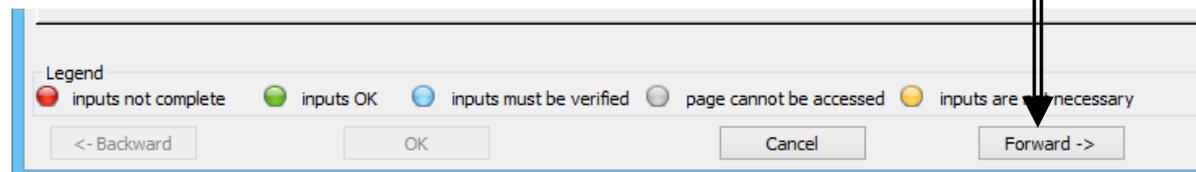
IV. New Method – 2/9

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)

Method Definition - Create New Method

Setup | Header | Temperature Program | Calibrations

Method type

Sample

Correction + sample

Sample mass constraints

Min sample mass: 0.001 mg

Max sample mass: 50000 mg

Laboratory: |

Project: |

Operator: Manager

Date: 12/22/2017 5:29:55 PM

Material: Empty Aluminum Pan

MFC gases

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

Method Definition - Create New Method

Setup | Header | Temperature Program

Method type

Sample

Correction + sample

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

Method Definition - Create New Method

Setup | Header | Temperature Program

Method type

Sample

Correction + sample

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

Legend

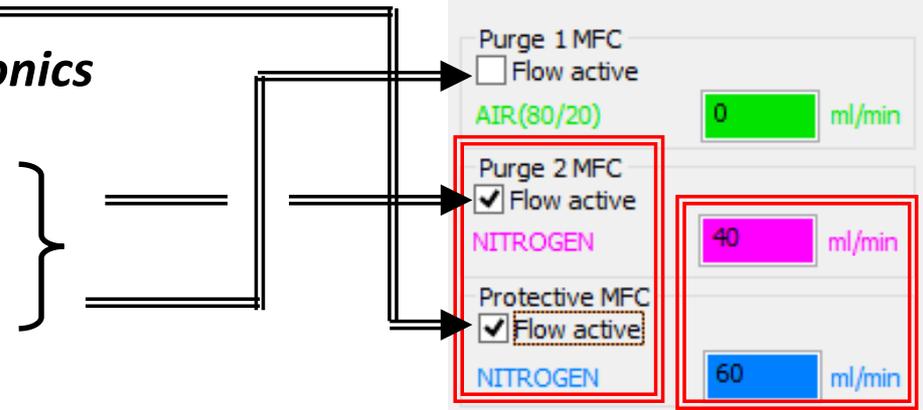
inputs not complete inputs OK inputs must be verified page cannot be accessed inputs are not necessary

<- Backward OK Cancel Forward ->

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 N₂** (or Air)

(Default) Protective: **60 mL/min N₂**

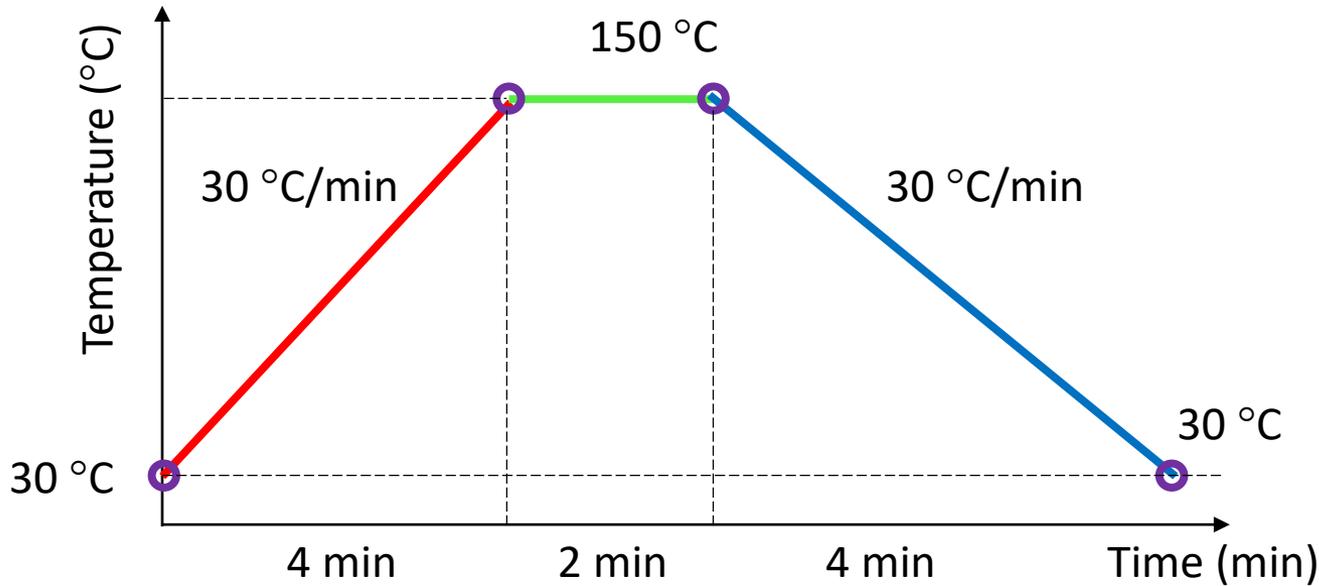
Protective must always \geq 60 mL/min N₂

IV. New Method – 5/9

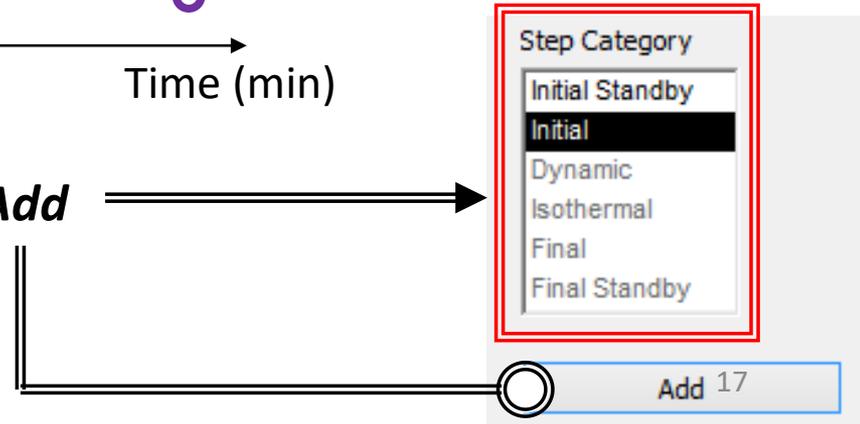
15. Build *Temperature Program* with desired *Step Categories*

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. Add *Initial* step

- Input Start temperature
Recommended temp = **25 – 30 °C**

Category

Start temperature: °C

Step Category

- Initial Standby
- Initial**
- Dynamic
- Isothermal
- Final
- Final Standby

18. Add *Dynamic* step

- 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)

Category

End temperature: °C

Heating Rate: K/min

Acquisition rate: points/K

Acquisition rate: points/min

Step Category

- Initial Standby
- Initial
- Dynamic**
- Isothermal
- Final
- Final Standby

19. Add *Isothermal* step

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

Category

End temperature: °C

Cooling Rate: K/min

Acquisition rate: points/K

Acquisition rate: points/min

Step Category

- Initial Standby
- Initial
- Dynamic**
- Isothermal
- Final
- Final Standby

Category

Isothermal time: hh:mm

Acquisition rate: points/min

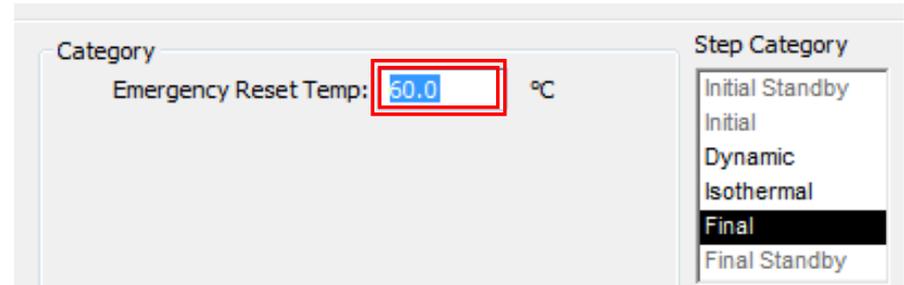
Step Category

- Initial Standby
- Initial
- Dynamic
- Isothermal**
- Final
- Final Standby

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

The screenshot shows the 'Temperature Program' tab selected in the software. Below the table, arrows point from the column headers to descriptive labels. Additionally, arrows from the 'N2/O2' and 'N2' columns point to 'Air Purge Gas Flowrate' and 'Purge 2 N₂ Gas Flowrate' respectively.

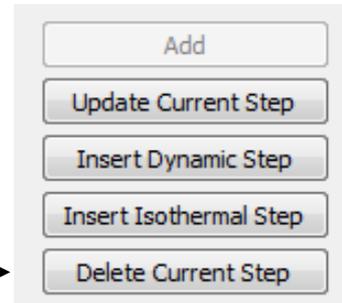
Nr	Type	°C	K/min	Time	pts/min	pts/K	AC	N2/O2	N2	N2
1	●	30.0					1	0	40	60
2	↗	150.0	30.000	0:04:00	600.00	20.00	1	0	40	60
3	→	150.0		0:02:00	150.00		1	0	40	60
4	↘	30.0	30.000	0:04:00	600.00	20.00	1	0	40	60
5	+	160.0					0	0	40	60
6	↖	25.0	20.000	0:00:15			1	0	40	60
7	↙	25.0		0:15:00			1	0	40	60

Labels below the table: Type of Step, Temp, Heat/Cooling Rate, Time for Step, Acquisition Rate, Protective Gas Flowrate.

Labels above the table: Air Purge Gas Flowrate, Purge 2 N₂ Gas Flowrate.

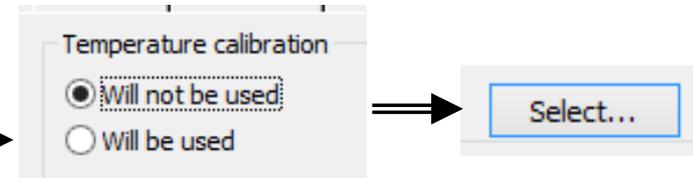
IV. New Method – 8/9

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**



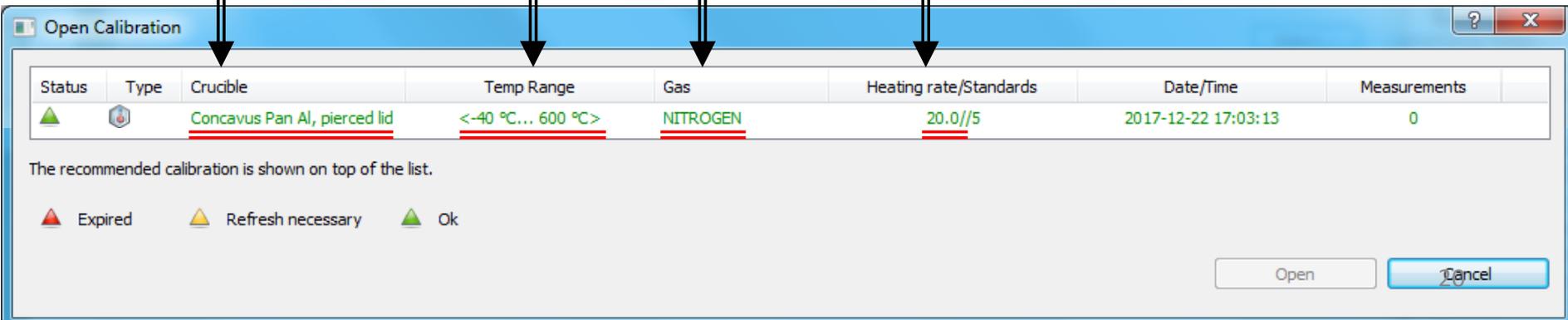
25. Select correct **Calibration File**, checking **EVERY** condition is correct:

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

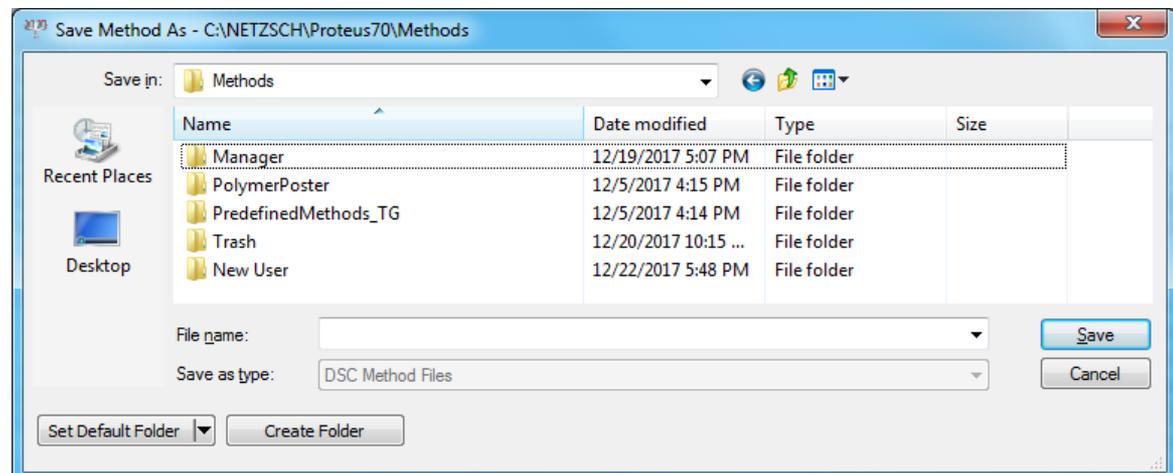
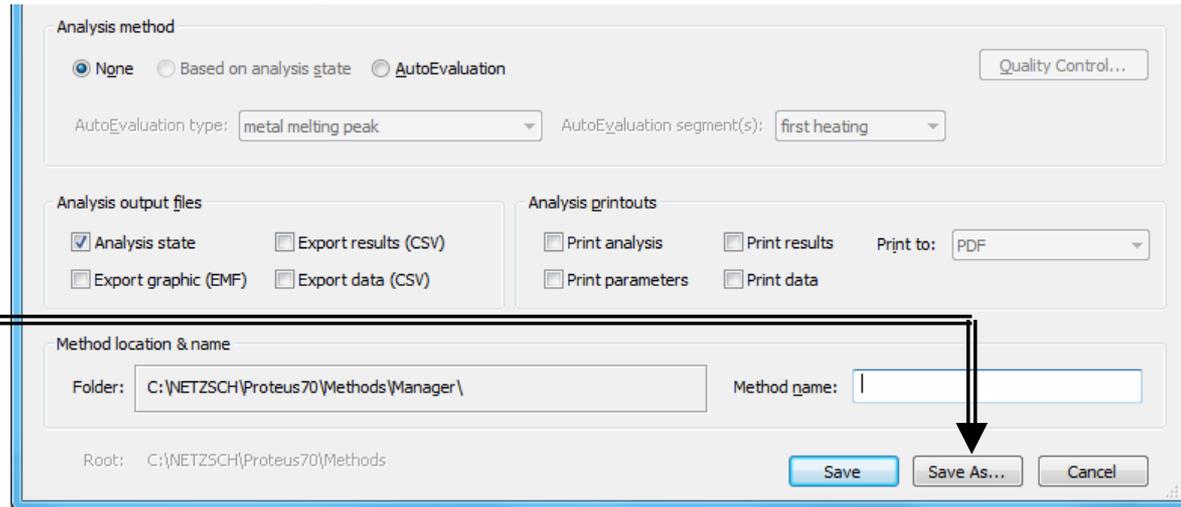
26. Repeat for **Heat flow**, **Tau-R**, and **BeFlat calibration**

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 °C)
3. Confirm **Max removal temperature** of crucible (Default = 100 °C)
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 °C**

ASC Manager Switch ON

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 sample and reference**

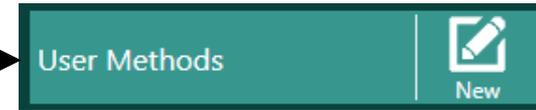
Reference crucibles

Position	Name	Mass [mg]	Crucible Mass [mg]	Crucible	
19		0	52.47	Concavus Pan Al, pierced lid 610 °C	Add Remove

Devices configurations
Cooling (Intracooler 40), MFCs

VI. 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

Basic data	
Laboratory	Optional
Project	Optional
Operator Name	Manager
Material	Empty
Remark	Optional

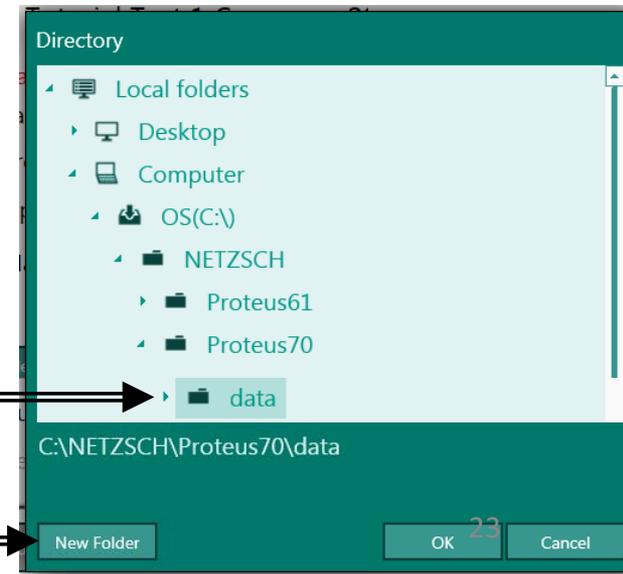
Sample ID	Required
Sample Name	Required
Directory	C:\NETZSCH\Proteus70\data
File Name	Required



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

Sample and Reference

Autosampler Position	0
Reference Position	19 - Concavus Pan Al, pierced lid Name: -----
Sample Crucible	Concavus Pan Al, pierced lid 610 °C
Sample Mass	0.001 mg
Sample Crucible Mass	51.9 mg
Reference Crucible Mass	52.47 mg

Remove lid
 Remove to trash after measurement end

Reference crucibles

Position	Name	Mass [mg]	Crucible Mass [mg]	Crucible	
19	-----	0	52.47	Concavus Pan Al, pierced lid 610 °C	<input type="button" value="Add"/> <input type="button" value="Remove"/>

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

Gases Info

Purge 1 MFC Gas does not matter
Purge 2 MFC NITROGEN flow: 40 ml/min
Protective MFC NITROGEN flow: 60 ml/min

Start Criteria

Sample temperature stability threshold 5 K
Sample temperature stability rate 0.1 K/min
Start delay after stability 00:00:30 hh:mm:ss

Temperature Program

Nr	Type	°C	K/min	Duration	pts/min	pts/K	STC	AIR(80/20)	NITROGEN	NITROGEN
0	●	30					<input checked="" type="checkbox"/>		40 ml/min	60 ml/min
1	↗	150	30	00:04:00	600	20	<input checked="" type="checkbox"/>		40 ml/min	60 ml/min
2	→	150		00:02:00	150		<input checked="" type="checkbox"/>		40 ml/min	60 ml/min
3	↘	30	30	00:04:00	600	20	<input checked="" type="checkbox"/>		40 ml/min	60 ml/min
4	●	160					<input type="checkbox"/>		40 ml/min	60 ml/min
5	↙	25	20	00:06:45			<input type="checkbox"/>		40 ml/min	60 ml/min
6	●	25		00:15:00			<input checked="" type="checkbox"/>		40 ml/min	60 ml/min

■ Purge 1 MFC ■ Purge 2 MFC ■ Protective MFC

Add to ASC

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

Configuration	Sample Tray	Execution list	Sample Tray State
0	Empty	1 Sample Concavus Pan Al, pierced lid (610 °C) Name: PET File: PET 2018 03 09 Sample.ngb-sdg Crucible 51.94 mg Sample 2.53 mg	2 Empty
15	Empty	16 Empty	19 Reference Concavus Pan Al, pierced lid (610 °C) ----- Crucible 52.02 mg Reference 0 mg

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

Configuration	Sample Tray	Execution list	Sample Tray State			
	Position	1	Sample name	PET	Method	PET Short Ramp Test 30 min 20K N2.ngb-s-dsc
	Reference	19	Sample ID	PET	Measurement file	PET 2018 03 09 Sample.ngb-sdg
	Sample	2.53 mg	Crucible	Concavus Pan Al, pierced lid (610 °C)	Analysis	
	Crucible	51.94 mg	Step status	Not yet run		

4. 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
1	PET	PET	PET Short Ramp Test 30 min 20K N2.ngb-s-dsc	PET 2018 03 09 Sample.ngb-sdg	Concavus Pan Al, pierced lid
19	Reference				Concavus Pan Al, pierced lid

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!**

Measurement - Method 'PET Short Ramp Test 30 min 20K N2.ngb-s-dsc' Measurement ASC Operation: Sample 1; Reference 19

The first run needs operator to check sample/reference status! If necessary remove them or 'Clear' corresponding 'Flags'.
When ready press OK to continue...

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

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

Open/close furnace	Command done. Furnace is closed.
Clear sample in flag	No sample in the furnace. No reference in the furnace.
Clear reference in flag	Sample in: - Reference in: -
Clear gripper in flag	Gripper in: -
Remove sample	Temperature: 26.6°C
Remove reference	

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

Open/close furnace

- click to “open” or “close” furnace to check what is inside...

Clear sample in flag

- click **ONLY IF** there is **NO** sample in the furnace

Clear reference in flag

- click **ONLY IF** there is **NO** reference in the furnace

Remove sample

- click to “remove” the current sample

Remove reference

- click to “remove” the current reference

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



OK

Cancel

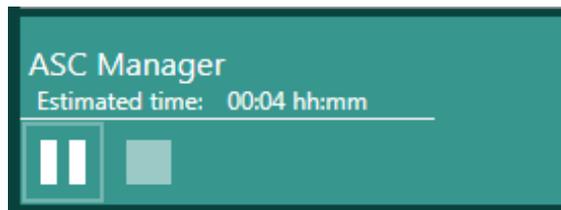
7. Follow any instructions or additional prompts that may appear

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



Preheating\precooling criteria:
Threshold: 5.0 K, Current difference: 0.5 K

Waiting for heating rate threshold (0.100 K/min)

Signals

P1	0 digits
P2(N2)	40 ml/min
PG(N2)	60 ml/min

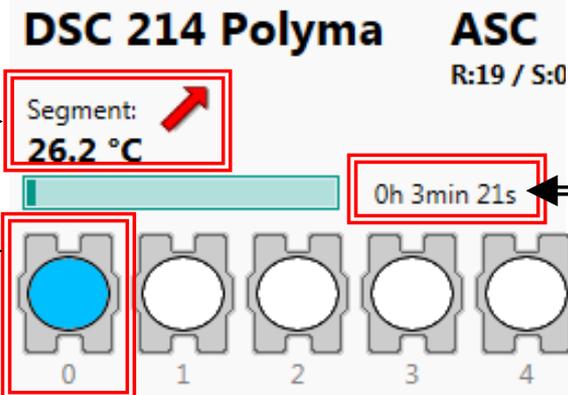
Start

Exit

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

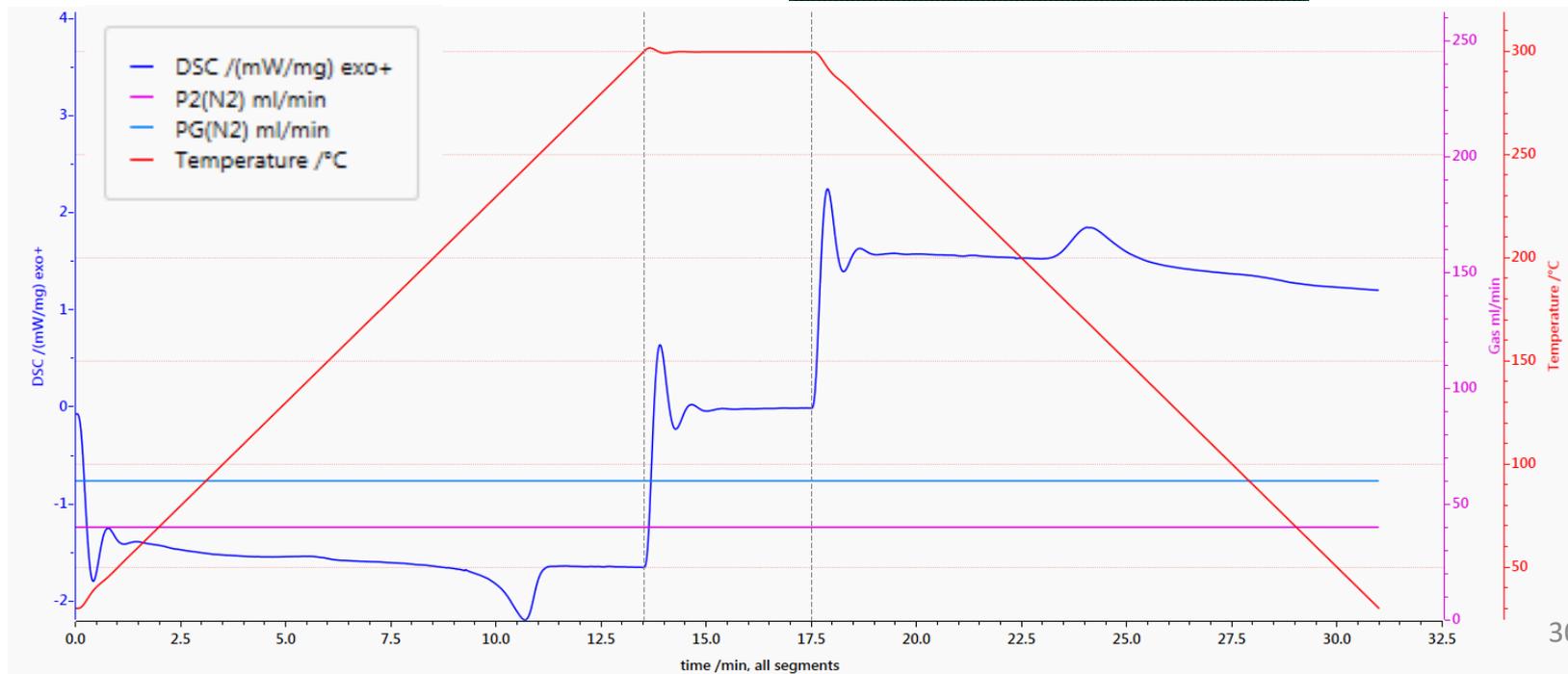
1. Click **ASC Manager**, and completed experiments will be updated with **Green** color code and completion date and time

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

Configuration	Sample Tray	Execution list	Sample Tray State		
Position	1	Sample name	PET	Method	PET Short Ramp Test 30 min 20K N2.ngb-s-dsc
Reference	19	Sample ID	PET	Measurement file	PET 2018 03 09 Sample.ngb-sdg
Sample	2.53 mg	Crucible	Concavus Pan Al, pierced lid (610 °C)	Analysis	
Crucible	51.94 mg	Finished	3/9/2018 11:37:23 AM		

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

Measurement



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**

	Sample	---	Open the furnace	Execute
	Reference	19		
	Furnace	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

DSC 214 Polyma

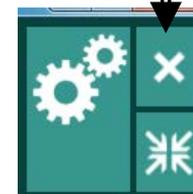
ECO
25.0 °C

Setpoint
ECO 25 °C

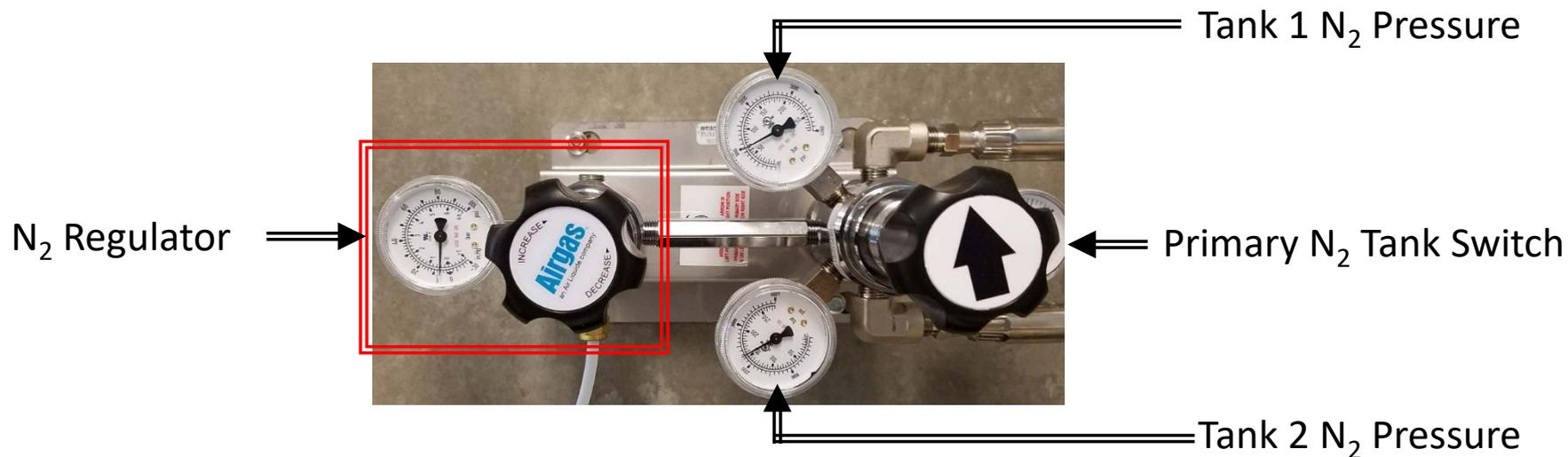


Setup & Control

ASC Manual Control



XI. Red Flags & Mistakes – 1/3

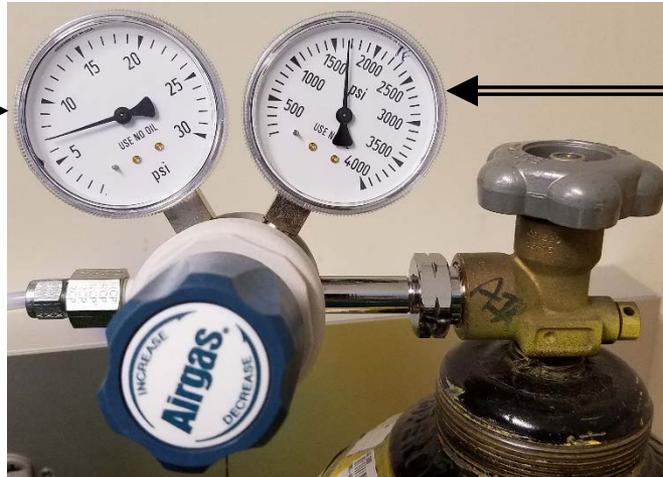


1. DO NOT ADJUST THE REGULATOR AS THIS MAY DAMAGE MASS FLOW CONTROLLERS \$\$\$
2. Check if Tank 1 or 2 N₂ pressure is at least **200 psi**, else contact Lab Manager to replace tank



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

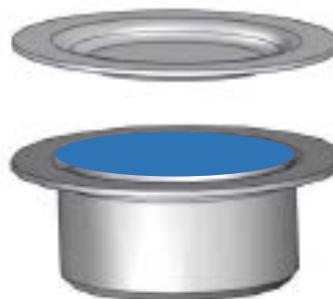


XI. Red Flags & Mistakes – 3/3

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



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



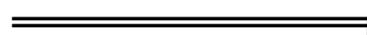
DSC sample chamber

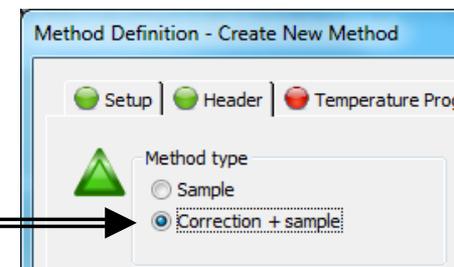
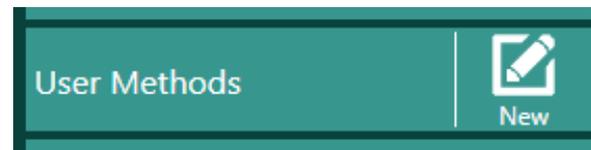


7. If ASC sample changer makes a noise while switching samples, report to Lab Manager immediately!

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



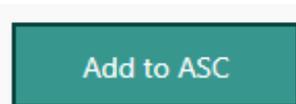
10. You will have to perform a baseline measurement first (unless you have already ran it)

A screenshot of a software interface for baseline corrections. The interface is divided into sections: "Basic data", "Measure baseline", and "Sample and Reference".
- "Basic data" section includes fields for Laboratory (UCR), Project (Optional), Operator Name (Manager), and Material (PET). There are also fields for Sample ID (PET), Sample Name (PET), and Remark (Optional). A "Clear basic data" button is located below the Material field.
- "Measure baseline" section has two radio buttons: "Measure baseline" (selected) and "Measure baseline+sample". Below them is a "Baseline status" field with a radio button and the text "Baseline must be performed first".
- "Sample and Reference" section includes fields for Autosampler Position (0), Reference Position (19 - Concavus Pan Al, pierced lid), Sample Crucible (Concavus Pan Al, pierced lid 610 °C), Sample Crucible Mass (51.75 mg), and Reference Crucible Mass (52.02 mg).
Red boxes highlight the "Sample ID" and "Sample Name" fields, the "Operator Name" dropdown menu, the "Autosampler Position" dropdown menu, and the "Reference Position" dropdown menu. Arrows from the instructions point to these specific elements.

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

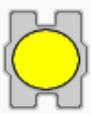
12. Select the position of your **Reference Pan**

13. Add to ASC at the bottom



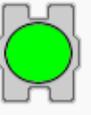
XII. Baseline Corrections – 4/6

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

Configuration	Sample Tray	Execution list	Sample Tray State			
	Position	0	Sample name	PET	Method	PET Short Ramp Test 30 min 20K N2 Correction.ngb-d-dsc
	Reference	19	Sample ID	PET	Measurement file	<u>Baseline measurement</u>
	Sample	<u>0 mg</u>	Crucible	Concavus Pan Al, pierced lid (610 °C)	Analysis	
	Crucible	51.75 mg	Step status	<u>Not yet run</u>		

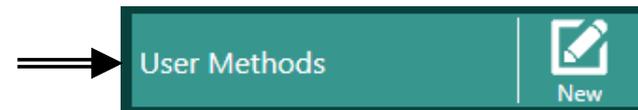
15. Click **Start** to perform the **Baseline measurement...**

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

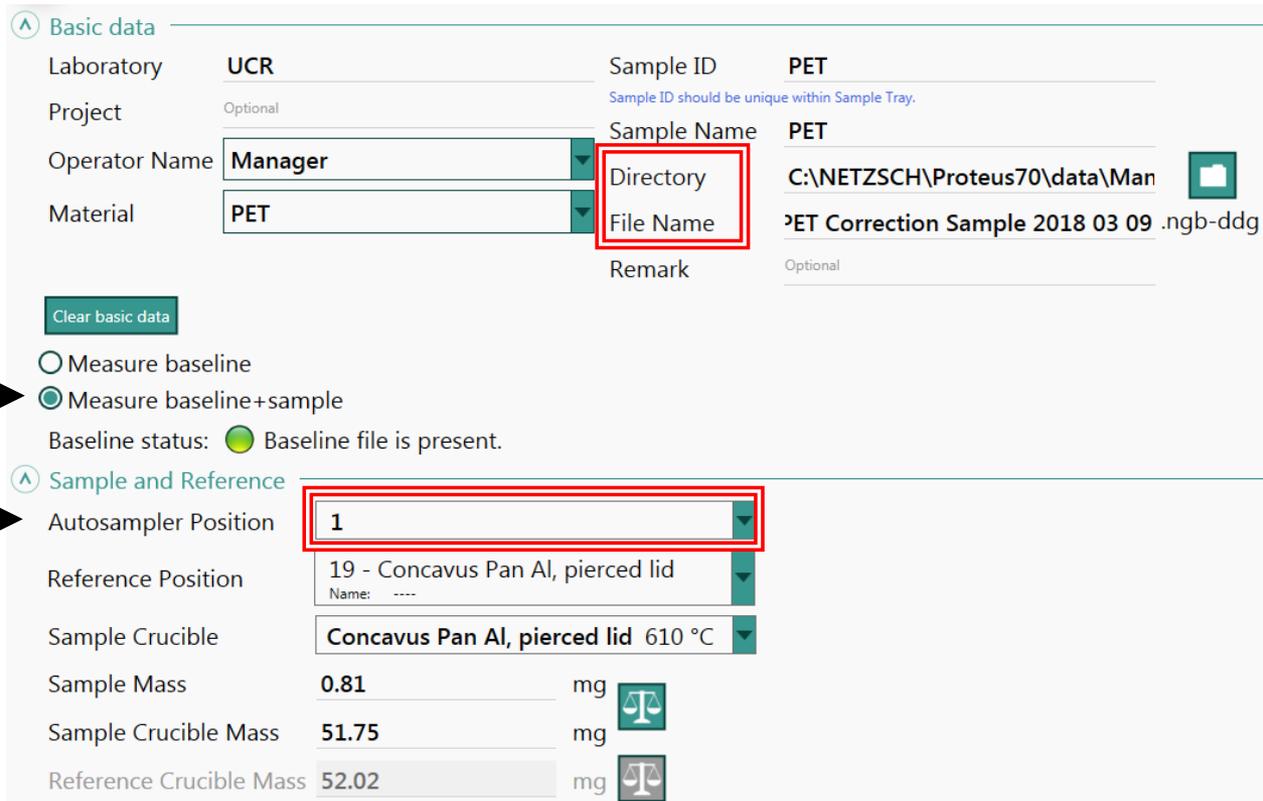
Configuration	Sample Tray	Execution list	Sample Tray State			
	Position	0	Sample name	PET	Method	PET Short Ramp Test 30 min 20K N2 Correction.ngb-d-dsc
	Reference	19	Sample ID	PET	Measurement file	<u>Baseline measurement</u>
	Sample	<u>0 mg</u>	Crucible	Concavus Pan Al, pierced lid (610 °C)	Analysis	
	Crucible	51.75 mg	Finished	<u>3/9/2018 2:21:48 PM</u>		

XII. Baseline Corrections – 5/6

17. Click *User Methods* again



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



Basic data

Laboratory UCR Sample ID PET

Project Optional Sample ID should be unique within Sample Tray.

Operator Name Manager Sample Name PET

Material PET Directory C:\NETZSCH\Proteus70\data\Man

File Name PET Correction Sample 2018 03 09 .ngb-ddg

Remark Optional

Clear basic data

Measure baseline

Measure baseline+sample

Baseline status: ● Baseline file is present.

Sample and Reference

Autosampler Position 1

Reference Position 19 - Concavus Pan Al, pierced lid

Sample Crucible Concavus Pan Al, pierced lid 610 °C

Sample Mass 0.81 mg

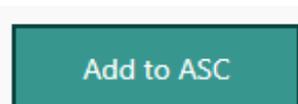
Sample Crucible Mass 51.75 mg

Reference Crucible Mass 52.02 mg

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

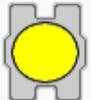
20. The new sample position is also updated to the next position automatically

21. Add to ASC at the bottom



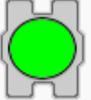
XII. Baseline Corrections – 6/6

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

Configuration	Sample Tray	Execution list	Sample Tray State
	Position: 1 Reference: 19 Sample: <u>0.81 mg</u> Crucible: 51.75 mg	Sample name: PET Sample ID: PET Crucible: Concavus Pan Al, pierced lid (610 °C) Step status: <u>Not yet run</u>	Method: PET Short Ramp Test 30 min 20K N2 Correction.ngb-d-dsc Measurement file: <u>PET Correction Sample 2018 03 09.ngb-ddg</u> Analysis:

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

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

Configuration	Sample Tray	Execution list	Sample Tray State
	Position: 1 Reference: 19 Sample: <u>0.81 mg</u> Crucible: 51.75 mg	Sample name: PET Sample ID: PET Crucible: Concavus Pan Al, pierced lid (610 °C) Step status: <u>3/9/2018 3:19:28 PM</u>	Method: PET Short Ramp Test 30 min 20K N2 Correction.ngb-d-dsc Measurement file: <u>PET Correction Sample 2018 03 09.ngb-ddg</u> Analysis:

XI. C_p : Sapphire Method – 1/1

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

- [Netzsch \$C_p\$ Sapphire Method](#)
- [Netzsch Tips for \$C_p\$ Measurement](#)
- [Mettler Toledo Measuring \$C_p\$ Guide](#)

Sapphire method
(according to DIN 51007)



2. Every determination of the C_p by DSC is comprised of 3 measurements:

- a. Baseline
- b. Standard – Sapphire (see Lab Manager)
- c. Sample

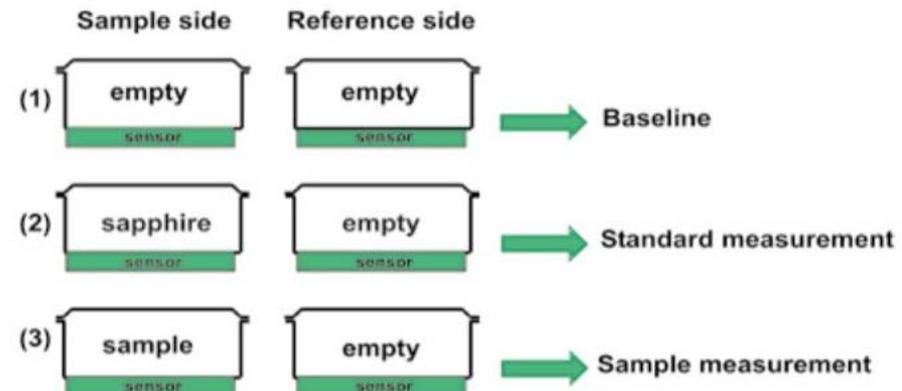


Fig. 1. Setup of a measurement series for determination of the specific heat 41