Netzsch DSC 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 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!

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:
   - **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

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**

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

**Condition: Sample in the furnace**

- **Clear the “Sample In” flag**: ONLY use this if there is NO sample in furnace, to correct this “error”

- **Remove sample from the furnace**: Use this to remove the current sample from the furnace and place back into original sample position
III. Setup & Control – 3/4

4. Review the different commands available below

Condition: **No 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”

Condition: **Reference in the furnace**

- **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:

\[
\text{Protective MFC} = \text{Nitrogen over electronics} + \text{Nitrogen over sample} = \text{Purge 2 MFC}
\]

or

\[
\text{Air over sample} = \text{Purge 1 MFC}
\]

14. Enter in desired gas flowrates:

- (Default) Purge 2 (or 1) Gas: **40 mL/min N}_2 \text{ (or Air)}
- (Default) Protective: **60 mL/min N}_2

Protective must always ≥ **60 mL/min N}_2
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:

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>30 °C/min</td>
<td>2 min</td>
</tr>
<tr>
<td>150 °C</td>
<td>4 min</td>
</tr>
<tr>
<td>30 °C/min</td>
<td>2 min</td>
</tr>
</tbody>
</table>

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 °C**

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

![Temperature Program Table]

<table>
<thead>
<tr>
<th>Nr</th>
<th>Type</th>
<th>°C</th>
<th>K/min</th>
<th>Time</th>
<th>pts/min</th>
<th>pts/K</th>
<th>AC</th>
<th>N2/O2</th>
<th>N2</th>
<th>N2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td>30.0</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1</td>
<td>0</td>
<td>40</td>
<td>60</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td>150.0</td>
<td>30.000</td>
<td>0:04:00</td>
<td>600.00</td>
<td>20.00</td>
<td>1</td>
<td>0</td>
<td>40</td>
<td>60</td>
</tr>
<tr>
<td>3</td>
<td></td>
<td>150.0</td>
<td></td>
<td>0:02:00</td>
<td>150.0</td>
<td></td>
<td>1</td>
<td>0</td>
<td>40</td>
<td>60</td>
</tr>
<tr>
<td>4</td>
<td></td>
<td>30.0</td>
<td></td>
<td>0:04:00</td>
<td>600.00</td>
<td>20.00</td>
<td>1</td>
<td>0</td>
<td>40</td>
<td>60</td>
</tr>
<tr>
<td>5</td>
<td></td>
<td>160.0</td>
<td></td>
<td>0:00:00</td>
<td>600.00</td>
<td></td>
<td>0</td>
<td>0</td>
<td>40</td>
<td>60</td>
</tr>
<tr>
<td>6</td>
<td></td>
<td>25.0</td>
<td>20.000</td>
<td>0:15:00</td>
<td></td>
<td></td>
<td>1</td>
<td>0</td>
<td>40</td>
<td>60</td>
</tr>
<tr>
<td>7</td>
<td></td>
<td>25.0</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1</td>
<td>0</td>
<td>40</td>
<td>60</td>
</tr>
</tbody>
</table>
IV. New Method – 8/9

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

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

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

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

   1) **Crucible:** Al2O3
   2) **Temp Range:** 0 °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**
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

Reference crucibles

<table>
<thead>
<tr>
<th>Position</th>
<th>Name</th>
<th>Mass [mg]</th>
<th>Crucible Mass [mg]</th>
<th>Crucible</th>
</tr>
</thead>
<tbody>
<tr>
<td>19</td>
<td></td>
<td>0</td>
<td>52.47</td>
<td>Concavus Pan Al, pierced lid 610 °C</td>
</tr>
</tbody>
</table>

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:\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 °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

- **Temperature Program**

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

<table>
<thead>
<tr>
<th>Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>Defined</td>
</tr>
<tr>
<td>Done</td>
</tr>
<tr>
<td>Done (analysis failed)</td>
</tr>
<tr>
<td>Failed</td>
</tr>
<tr>
<td>Measurement Active</td>
</tr>
<tr>
<td>Reference</td>
</tr>
</tbody>
</table>

   ![Sample Tray Tab](image)

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

   ![Execution list Tab](image)

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

   ![Sample Tray State Tab](image)
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”

   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
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°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.
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

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 $$$

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...

5. Click Forward -> to advance
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*

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

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

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

<table>
<thead>
<tr>
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<th>Execution list</th>
<th>Sample Tray State</th>
</tr>
</thead>
<tbody>
<tr>
<td>Position</td>
<td>1</td>
<td>Sample name: PET</td>
<td>Method: PET Short Ramp Test 30 min 20K N2 Correction.nbg-d-dsc</td>
</tr>
<tr>
<td>Reference</td>
<td>19</td>
<td>Sample ID: PET</td>
<td>Measurement file: PET Correction Sample 2018 03 09.nbg-ddg</td>
</tr>
<tr>
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<td>Analysis</td>
</tr>
<tr>
<td>Crucible</td>
<td>51.75 mg</td>
<td>Finished: 3/9/2018 3:19:28 PM</td>
<td></td>
</tr>
</tbody>
</table>
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