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 3 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. Cleanup

X. Red Flags & Mistakes
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) Purge 2: N2 60 mL/min
         Protection: N2 40 mL/min

   III. Acceptable **Calibrated Heating Rates**
      a) 5 K/min, 10 K/min, 20 K/min, 30 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

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. Click on *Setpoint* and choose to switch **ON** to turn on the protective gas

3. It’s **IMPORTANT** to check that the *Setpoint* is **ON** prior to any test

4. Confirm that either *IDLE 25 °C* or *ECO 25 °C* is shown under *Setpoint*
II. Start – 2/2

5. Confirm that **Active** is checked and set to **60 min**

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

7. If the values are different, proceed to change them back to what is shown on the right and click **Apply**
III. Setup & Control – 1/2

1. Click on **Setup & Control**

2. Click on **Signals** to show the Temperature and Gas Flow signals

3. Click on **ASC Manual Control** to bring up the menu to control the **Auto Sampler**
IV. New Method – 1/7

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

2. Select the appropriate **Crucible** from dropdown (Default): *Concavus Pan Al, pierced lid (... 610 °C)*

3. Confirm that **Start Criteria** is acceptable (Default): ± 5.0 °K from “Initial” temperature with max delay of 30 min, with Heating of 30 K/min (max 20 min) or Cooling of 50 K/min (max 30 min)

4. Confirm **Automatic Cooling** is set to **ON**

5. Confirm **O.I.T.** is set to **OFF** unless O.I.T. test is desired

6. Click **Forward ->** to advance
IV. New Method – 2/7

7. 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

8. Click **Forward ->** to advance
IV. New Method – 3/7

9. It’s IMPORTANT to check both *Purge 2 Gas* and *Protective Gas*

10. Enter in desired Purge Gas 2 and Protective Gas flowrates
    
    (Default) Purge 2 Gas: **40 mL/min** N₂
    
    (Default) Protective: **60 mL/min** N₂

11. Build *Temperature Program* with desired *Step Category*

12. Input parameters for desired *Category*

13. Click *Add* to insert desired *Step Category*
IV. New Method – 4/7

14. Add *Initial* step
   a) Input Start temperature
      Recommended temp = 25°C

15. Add *Dynamic* step
   a) Input End temperature
   b) Input Heating Rate or Cooling Rate
      Note: Heating Rate should be equal to calibrated rates
   a) Input Acquisition rate
      (default values will be automatically inserted)

16. Add *Isothermal* step
   a) Input Isothermal time
   b) Input Acquisition rate
      (default values will be automatically inserted)

17. Add *Final* step
   a) Input Emergency Reset Temp
      (default values will be automatically inserted)
   b) Additional steps will be automatically inserted to bring final temp back to 25°C
14. Your desired **Temperature Program** details can be reviewed in table

15. Select desired **Step** and modify parameters and then click **Update Current Step**

16. Click **Forward** -> to advance
IV. New Method – 6/7

17. Check **Will be used** for **Temperature calibration**

18. Select the correct Calibration File, checking that the following are correct:

- **Crucible Type:** Concavus Pan Al, pierced lid
- **Gas:** NITROGEN
- **Heating rate:** 5, 10, 20, 30 K/min [for different Heating rates, contact Lab Manager]
19. Repeat for *Heat flow*, *Tau-R*, and *BeFlat calibration*

20. Click **Forward** to advance

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

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

23. 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 in Reference crucible information (see Default information below)
   Position = **19**
   Mass (mg) = **0**
   Crucible Mass (mg) = *Weigh mass of crucible using Precision Balance*
   Crucible = **Concavus Pan Al, pierced lid 610 °C**
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\"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 **User Data Folder** or create **New Folder**
VI. Opening Method – 2/3

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

8. Select the **Reference Position**
   Recommendation: **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 everything is correct for your desired **Method**

13. If everything is correct, proceed to Add Method to the ASC queue by clicking **Add to ASC** at the bottom
VII. ASC Manager – 1/1

1. Click on **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>

![Autosampler Image](image)

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

![Autosampler Image](image)

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

![Autosampler Image](image)
VIII. Running Experiments – 1/2

1. Review ASC Manager *Execution list* and confirm all is correct

2. Click *Start* when ready

3. Proceed to review the notes described here

   **IMPORTANT!**

4. Follow the instructions and perform the corrections necessary

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

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

6. Follow any instructions or prompts that appear
VIII. Running Experiments – 2/2

7. DSC will now begin to preheat/precool to target Initial temperature

8. Click **Start** when initial conditions have been met for experiment

9. The **Estimated time** for all experiments are shown here

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

11. The active measurement can be shown here

12. The remaining **segment time** and **completion %** is shown here
IX. Clean Up – 1/1

1. After experiment(s), DSC should automatically cool itself down to 25°C either through *Idle Mode* or *Eco Mode*

2. Click on **Setup & Control**

3. Click on **ASC Manual Control**

4. Check that the following is true, else correct:
   - *No Reference*
   - *No Sample*
   - *Furnace is Closed*

5. When DSC temperature reaches 25 ± 5°C, click the *X* to close the software

6. Confirm that you wish to *keep the setpoint ON* after you close software

7. Log out of your ENGR account

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

9. 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 the Tank 1 or 2 N\textsubscript{2} pressure is at least \textbf{200 psi}, else contact Lab Manager to replace tank
3. Remove any trace of sample on outside and underneath crucible, as it will contaminate the DSC sample chamber $$$

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

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