Netzsch TGA 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 3 hour block on Faces for your training
Netzsch TGA Operation

I. Preparation
II. Start
III. Setup & Control
IV. New Method
V. Opening Method
VI. ASC Manager
VII. Preparing ASC
VIII. Running Experiments
IX. Cleanup
X. Red Flags & Mistakes
XI. Troubleshooting
I. Preparation – 1/3

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 (Al₂O₃) Crucibles – 6.8 mm diameter, 4 mm height, 85 μL

   II. Acceptable *Calibrated Gas environments*
      a) Purge 2: N₂ 20 mL/min
         Protection: N₂ 20 mL/min

   III. Acceptable *Calibrated Heating Rates*
      a) 5 K/min, 10 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!
II. Start – 1/2

1. Click *SmartMode Measurement* to start TGA 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 activate the pop-up window showing the Temperature and Gas Flow signals

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

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

2. Confirm **Sample carrier** is set to **TG 209F1 std (Al2O3 support)**
   TC: P (-200...1200 °C)

3. Select the appropriate **Crucible** from dropdown (Default): **Al2O3 (... 1700 °C)**

4. 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)

5. Confirm **TG BeFlat support** is set to **ON**

6. Confirm **Super-Res** is set to **OFF** unless **Super-Res** is desired

7. 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 – recommend if results are critical

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

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

10. Enter in desired Purge Gas 2 and Protective Gas flowrates

   (Default) Purge 2 Gas: **20 mL/min N₂**
   (Default) Protective: **20 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
IV. New Method – 5/7

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:** Al2O3
   - **Gas:** NITROGEN
   - **Heating rate:** 5, 10, or 20 K/min
IV. New Method – 7/7

19. Click *Forward* -> to advance

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

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

22. Click *Save*
V. 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**
V. Opening Method – 2/3

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

8. Select the type of **Sample Crucible**
   Default: **Al2O3 1700 °C**

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

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

    - **Calibration Info**
    - **Gases Info**
    - **Start Criteria**
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
VI. ASC Manager – 1/2

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

![ASC Manager 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 Crucible**

5. Pick Stability criteria before experiment starts:
   Default: **Time only for 1 min**
VI. ASC Manager – 2/2

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

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

8. Click **Sample Tray State** to review details of experiments scheduled
1. Clean empty Alumina crucible with appropriate solvent
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. Lightly and gently wipe the ASC sensor with Kimwipe and IPA to remove any dust
5. Avoid touching the grippers as it may bend
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!

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**Sample 6: 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...

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5. Follow the instructions and perform the corrections necessary
6. After all flags and conditions are corrected, click **OK** to begin experiments
7. Follow any instructions or prompts that appear
VIII. Running Experiments – 2/3

8. A prompt will appear when *Empty Crucible Weighing* is completed, click OK

9. Click **Start** at bottom of screen when ready

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

11. Follow the instructions and perform the corrections necessary

12. After all flags and conditions are corrected, click **OK** to begin experiments
VIII. Running Experiments – 3/3

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

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

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

10. The active measurement can be shown here

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

1. After experiment(s), TGA 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 Sample**
   - **Furnace is Closed**

5. When TGA 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 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 TGA thermocouple $$$

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

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