**Netzsch Thermomechanical Analyzer 402 F1**

**Basis Operation Procedure**

**Attention:**

Use Globes + tweezer.

Do not swing the furnace.

Check the Nitrogen gas and do not change the gas pressure.

The temperature program used in Correction measurement and Correction + sample measurement should be the same.

Sample should be flat. Check the sample size, melting point of the sample and the test temperature.

Maximum Length: up to 25mm

Maximum diameter: up to 10 mm

Maximum force: 0.2N

**General information**

Power switch – Instrument “on/off”. Behind the instrument.

Correction sample - High purity aluminum oxide

Correction sample dimension: 6mm dia., 25mm length (mentioned on the back of sample box)

Furnace temperature range: RT- 1550°C (Recommended temperature- up to 1400°C)

Heating rate- 50 K/min

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| Graphical user interface, table  Description automatically generated | Diagram  Description automatically generated |

**Procedure**-

1. Opening the instrument
2. Inserting the standard sample
3. Closing the instrument
4. Turning on the gas
5. Entering measurement parameters
6. Starting the program
7. Saving the measurement file
8. Opening the instrument and taking out the sample
9. Inserting the tested sample and closing the instrument
10. Entering measurement parameters
11. Starting the program and saving the file
12. Finishing the measurement

**1. Opening the instrument**

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| Pressing “Open button+ Safety button” simultaneously moves the furnace into the upper end position (Green LED nest to the open button lit) | Graphical user interface  Description automatically generated |

**2. Inserting the standard sample**

Note: **Sample should be flat**. If not, it might be tilted.

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| * Move the pushrod downwards to get enough space between pushrod and top to set up the sample. * Set up the sample carefully in the center of pushrod using tweezers. * Move pushrod upwards normal for a while using  button and stop using  button when it closes to the top. * Move upwards **slowly** using  button. Stop itself when it touches at the top where is a little flat spot. * Move pushrod down **slowly** using  button (a little bit) and up **slowly** using  button. **Make twice** to ensure the balance of the force to the sample. | A close-up of a faucet  Description automatically generated with medium confidence |

Note: ΔL is no force no expansion at this state but some smaller force is shown in display due to contacting with the top.

**3. Closing the instrument**

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| * To close the instrument, swing the furnace to the middle position. * Pressing “Close button+ Safety button” simultaneously moves the furnace into the lower end position (Green LED next to the open button lit) |  |

**4. Turning on the gas**

Check the nitrogen gas and turn it on. Do not change the pressure.

**5. Enter Measurement Parameters (Run a measurement)**

Open the computer, Password “nopass”. Open the software A picture containing text

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**T**here are 2 types of measurement. I. Correction and II. Correction +sample.

**I. Measurement type- Correction**

If you want to run a correction measurement: Make **New** file.

Define your measurement program. (Set up, Header, Calibration standard, Temperature program, Calibration, Last items)

1. **Set up** (Check instrument configuration)

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* Sample length determination: Select **Manual input**.
* Click on **Forward->** or click on **Header**.

1. **Header**

* Select measurement type: **Correction**
* Define the standard sample: Identity, Name, Shape, Size, and Material.
* Define MFC gases (Purge/protective): Purge 2 MFC-Nitrogen, Protective MFC-Nitrogen
* Click on **Forward ->** or click on **Calibration standard.**

1. **Calibration standards**

* Select temperature calibration (if require). **OR**
* Select table of calibration standard. (C:\NETZSCH\Proteu80\\_Records\cal\Al2o3ne.sd)

1. **Temperature Program**

Table

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* Define all steps of temperature program.
* Select step category (Initial, Dynamic, Isothermal, Dynamic, Final, Final stand-by)
* Click on ‘**Add’** after adding the numbers for each category and then go to the next category.

1. **Calibration**

Check temperature calibration **will not be used**.

1. **Last items** (Define file name)

Save the correction file in C:\NETZSCH\Proteus80\data.

Click **Measure.** The program switches to the adjustment window. Before the program starts, all measurement definition should be **green**.

**6. Starting the program**

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| * Choose **DIL**-5000um, **Offset**- standard or choose one depends on the sample and click on **Set initial force gases** to check the gas **-** click **Yes,** and then **Start** the program. * NGB Measurement box will come up. Click **OK**. * You can also check your program on the display area of the furnace while the process is running condition.   After completing the running process, Click on **Measurement** in the menu and select **Stop stand by**.  Wait for a while to complete the process. | Graphical user interface  Description automatically generated |

**7. Saving the measurement file**

* Click on the **Extra** on the menu bar, select **Run Analysis program**.
* Netzsch Proteus Thermal Analysis 8.03 window comes up.
* Click on the **Extra** on the menu bar and select **Export data. Choose** Click on the **Export** on the measurement data and save the file.

**8. Opening the instrument and taking out the sample**

* Before opening the furnace, cool down the sample or the furnace temperature should be under 50°C.
* Follow the procedure mentioned in section **1. *Opening the instrument***
* Move the pushrod downwards using  to get enough space between pushrod and top to take out the sample.
* Take out the sample carefully using gloves and tweezers.

**9. Inserting the sample and closing the instrument** (Sec. 2 &3)

* Set up the sample carefully in the center of pushrod using tweezers.
* Move pushrod upwards **slowly**. Stop itself when it touches at the top where is a little flat spot. Move pushrod Down (a little bit) and up. **Make twice**.

*Note: Sample should be flat. If not, it might be tilted.*

* Follow the procedure mentioned in section **3. *Closing the instrument***

**10. Entering the program parameters**

**II. Measurement type- Correction + Sample**

If you want to run a correction + Sample measurement: Click on **File** and then select used file from standard correction measurement (**Open circle file)** and **Open**.

Define your measurement program. (**Fast definition**, Set up, Header, Calibration standard, Temperature program, Calibration, Last items)

Note: You can set up your measurement program in **Fast definition** only.

1. **Fast definition**

* Select measurement type: Correction +sample
* Click **Get them** to accept properties of just open measurement OR

Click **Reset them** to define new properties and setting: Identity, Name, Shape, Size, Material

e.g. Dimension of the sample- 6.36mm x 6.36mm x 12.34mm (cubic)

* Click on **Select** to save the file in C:\NETZSCH\Proteus80\data.
* Click on **Forward ->** or **Measure**.

Graphical user interface, application

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*Note: You can see that the rest of measurement definitions are green (Set up, Header, Temperature program, Last items)*

The temperature program must be identical to the just opened temperature program.

**Calibration standards**

Select table of calibration standard. (C:\NETZSCH\Proteu80\\_Records\cal\Al2o3ne.sd)

**Calibration**

Check temperature calibration **will not be used**.

11. **Follow the procedures mentioned in sections:**

6. Starting (measuring) the program

7. Saving the file

**12. Finishing the measurement**

Follow the procedures mentioned in section 8. Opening the instrument and taking out the sample, and

3. Closing the instrument

Turn off the gas after testing. Don’t turn off the pump and water-cooling system.