



# TGA quick Operation Guide

1. Open Nitrogen valve (**Do not adjust** the pressure regulator)
2. Turn ON the instrument (ON/OFF switch is at the instrument back)



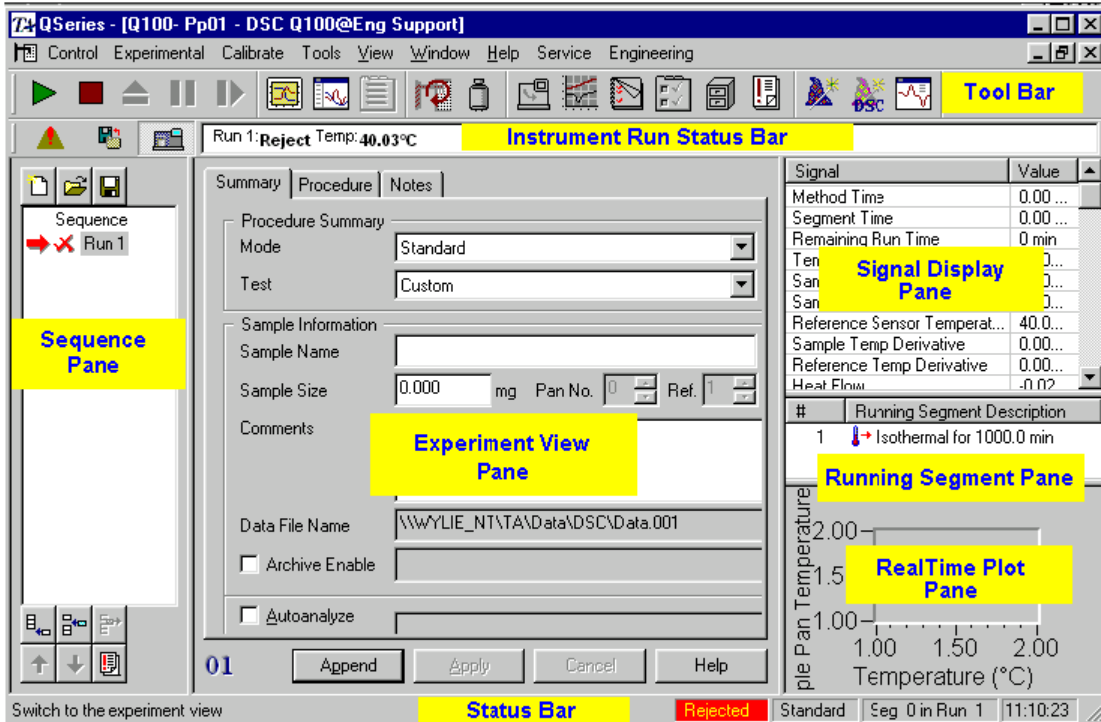
3. If you need to work in oxidative or inert environment, open the Air or Argon cylinder valve correspondingly (**Do not adjust** the pressure regulator)
4. Turn ON the computer.

- Start **TA instrument explorer** software 
- Double click on icon  to open **Control Window**
- If software – instrument communication is lost, reset the communication by right-click on a clear field above menu bar and select **Reset TA message agent**

**Instrument Control Window** enables to set the parameters of the experiment and provides an updated information about its run:

- At the top of the window is the main menu and the tool bar;
- Below the tool bar is the instrument run status bar;
- Sequence Pane displays the sequence or run list information;
- Experiment View Pane enables to set the experiment parameters;
- Signal Display Pane monitors parameters at real time;
- Running Segment Pane displays the method steps. Currently running step is indicated by green arrow;

- RealTime Plot Pane monitors results at real time;
- Status bar at the bottom displays the current method segment and its run number in the sequence.

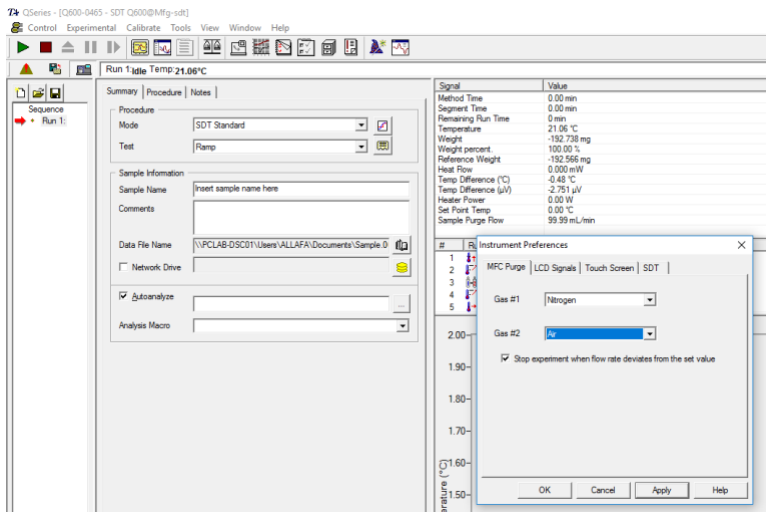


## Running TGA experiment

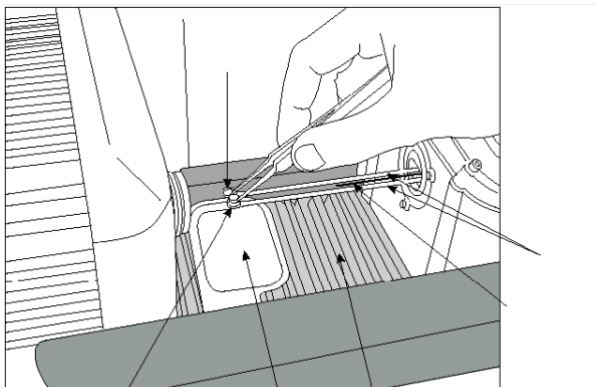
**USE ONLY CLEAN CRUCIBLES** (for the cleaning procedure see APPENDIX).


### In the Control Window

1. If you need to work with Air or Argon select from Menu **Tools > Instrument Preferences > MFC Purge**. Set **Nitrogen** as **Gas #1** and **Air** or **Argon** as **Gas #2**



2. Select from the Menu **Control > Furnace > Open**
3. Gently place empty crucibles into the front (sample) and rear (reference) balance arm caps. The balance arms are very delicate, so do not apply any pressure.

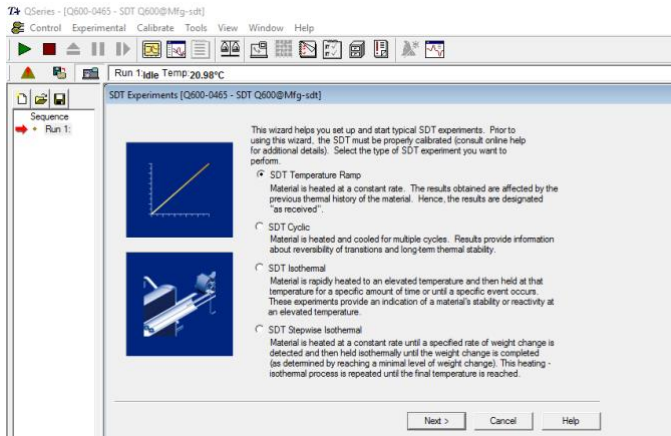


4. Select from the Menu **Control > Furnace > Close**
5. Wait until the weight readings in the **Signal Display Pane** stabilize
6. Click on the Tare icon  and let the weights restabilize at 0.00
7. Crush the sample into powder for more uniform thermal contact. A good sample size is around 10 mg. It is recommended not to exceed 20-25 mg to ensure a uniform sample heating.
8. Select from the Menu **Control > Furnace > Open**. Take aside a crucible from the front balance arm. Pour the sample in the crucible and carefully return it back to the front balance arm cap.
9. Select from the Menu **Control > Furnace > Close**.
10. Connect vacuum suction tube to the furnace exhaust.

11. Wait until the weight readings in the **Signal Display Pane** stabilize

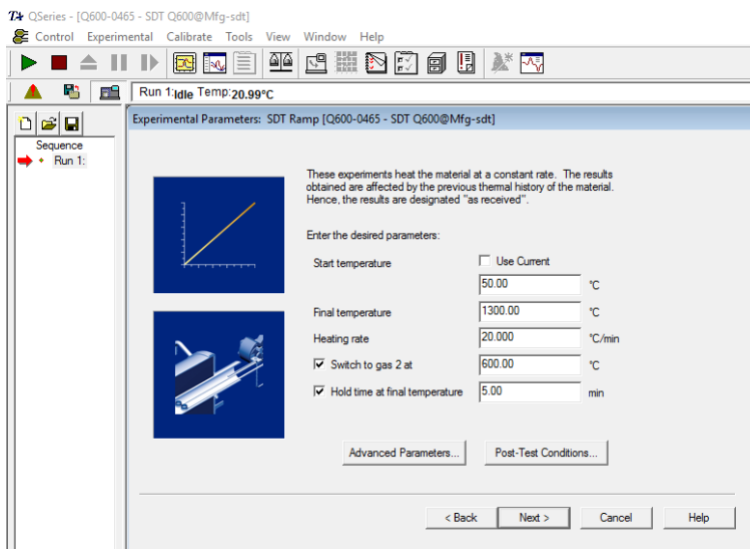
12. On the Tool Bar menu click **Experiment Wizard** icon

- Make sure **SDT Temperature Ramp** is selected and press **Next**

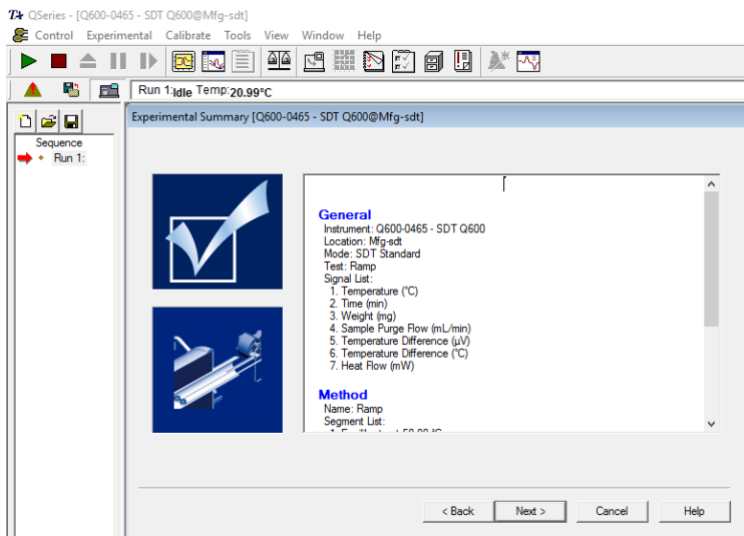


- Set starting and final temperatures and heating rate and press **Next**. The experiment is designed to heat the material at a constant rate. It is recommended to set the heating rate 20°C/min or less to ensure a uniform sample heating. Make sure the crucible material is stable at the Final temperature. Press the **Help** button for more details.

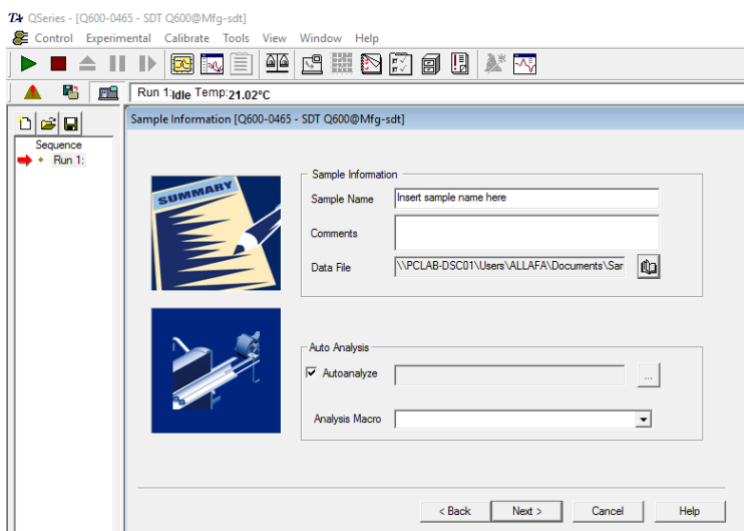
If the entire run is carried out only under Nitrogen, make sure **Switch to gas 2 at** box is clear. If the acquisition starts under Nitrogen atmosphere and then Air or Argon should be opened at a certain point, enable **Switch to gas 2 at** and set the temperature for the gas change. If the run is carried out under Air or Argon from the beginning, leave **Switch to gas 2 at** box clear. You will set the gas as **Purge Gas Setting** in a few steps.



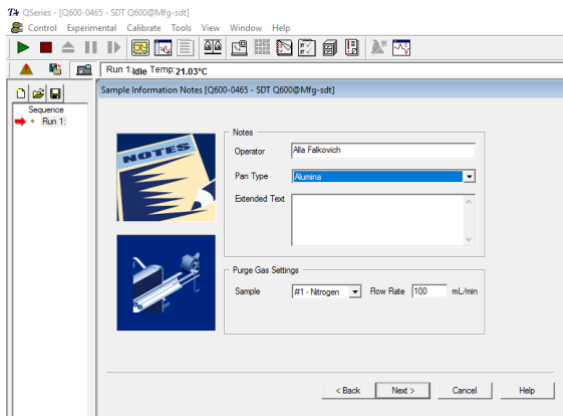
- Check the starting conditions of the experiment are appropriate and press **Next**



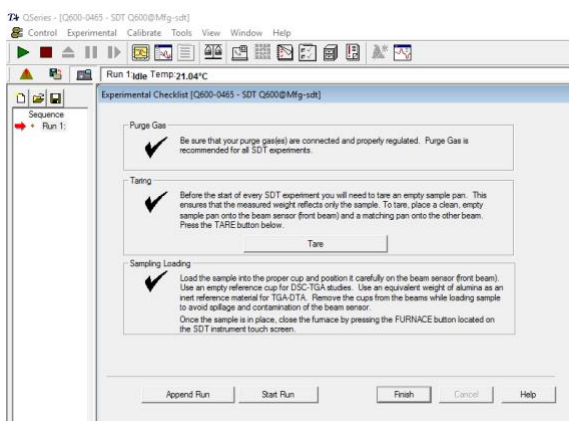
- Fill the sample information and data file pathway and press **Next**



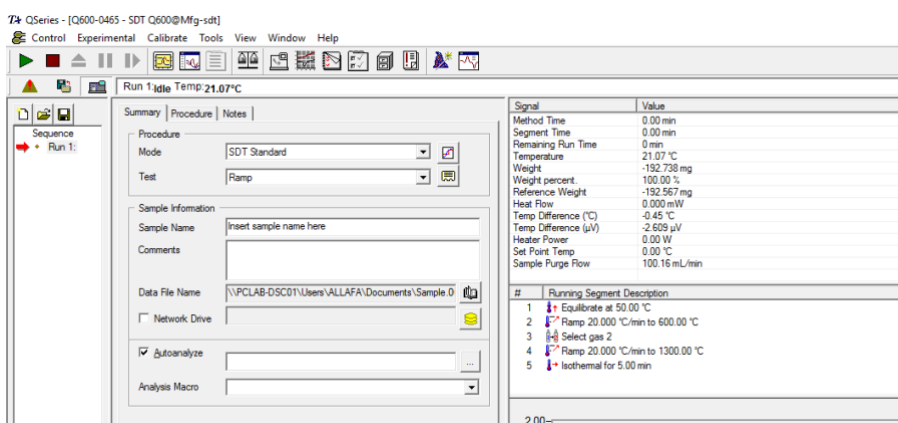
- Fill the operator information. Carefully chose the type of crucible. If the run is carried out under Air or Argon atmosphere from the beginning, select the gas in the **Purge Gas Setting > Sample** (flow rate 100 mL/min). Press **Next**.



■ Click **Finish**



**13.** Starting conditions of the experiment appear in the **Running Segment Pane.**



**14.** On the Tool Bar menu click green **play** arrow  to start the run

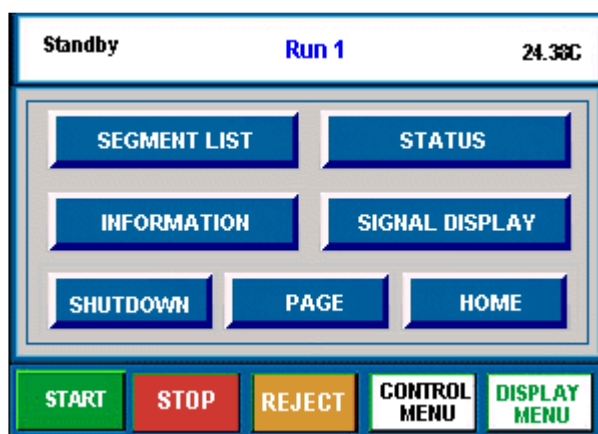
15. Currently running segment is indicated by green arrow. Segments followed the running segment might be modified. Right-click mouse at the Running segment pane and select **Modify method**
16. Modify the method parameters as required. Add or reduce steps. Click **Send changes** and then **Close**. The method will be updated.

## End of instrument operation

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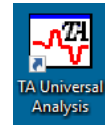
### In the Control Window

1. Disconnect vacuum suction tube from the furnace exhaust.
2. Select from the Menu **Control > Furnace > Open**
3. Remove both crucibles from the balance arm caps.
4. Select from the Menu **Control > Furnace > Close**
5. Close **TA instrument explorer** software.
6. On the instrument screen press **DISPLAY MENU** button.
7. Press **SHUTDOWN** and then **OK**.

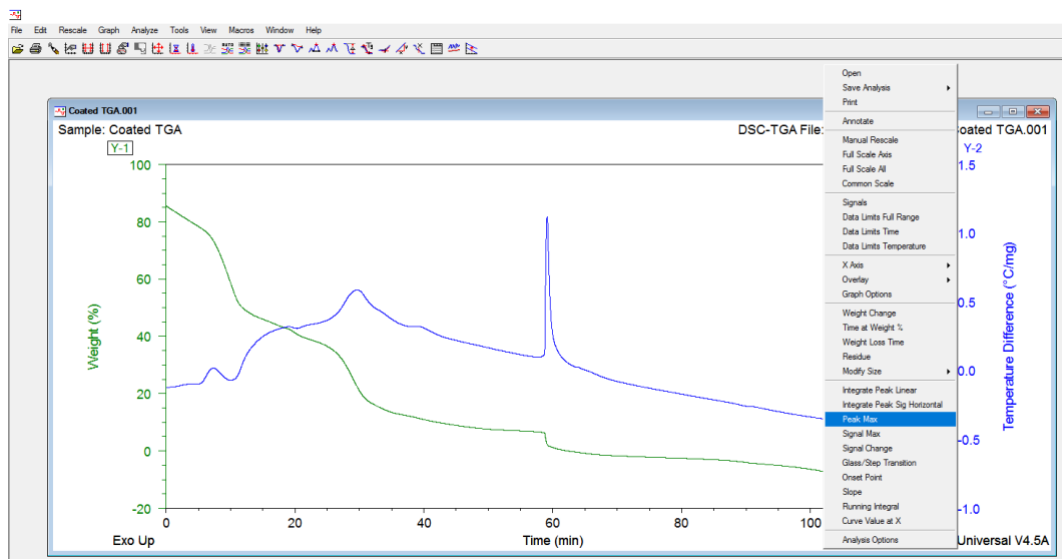


8. Wait until a message providing that **the instrument can be safely shutdown** appears and then switch off the instrument.
9. Close Nitrogen and (if used) Air or Argon valves.

# Data analysis

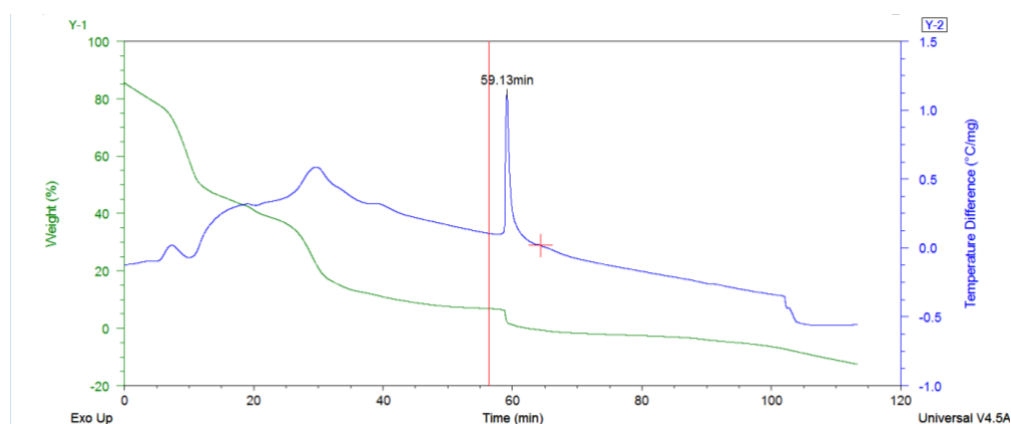


1. Double-click on TA Universal Analysis icon on the Desktop
2. To view and process data during the run select from Menu **File > Open > Local Disk D:\“Your Folder”**. Select the required file > **Open**.
3. You can open up to four (Y1-Y4) signals In the Signal selection window. Select signal’s settings, e.g. Y1 “Normal” and then “Weight, %” and Y2 “Normal” and then “Temperature Difference” from the Signals drop-down lists. Select desired signal for the X axis. Select unit setting and click **OK**. You can reset both signal and unit settings later from the Menu in Data Analysis Window.
4. To change signal setting in Data Analysis Window, right-mouse click on Y1 (upper left corner of the chart) or Y2 (upper right corner of the chart). To switch between the X-axis signals from Menu **Graph > X Axis**.
5. If the scan is not finished yet choose from Menu **Graph > Auto-refresh Data**.
6. To choose the type of analysis that you want to perform on the current data file from Menu **Analyze** or right-mouse click inside the area of the chart. The menu reflects the type of analyses available for the particular type of signal that is currently selected.





- Once the analysis is selected, two red markers appear. Drag and drop the right and left markers to select the desired signal change range and press **ENTER**. The result appears on the graph.



- Read “**UManual**” (open shortcut on the Desktop) for more data processing options.
- To save the file, from the Menu select **File > Save Analysis** or **Save Analysis and Data** and click **OK**.
- Print the data and analysis in \*.pdf format and save it in your folder.  
**Important:** **NEVER** use disk-on-key to save the report. Send the report or raw data files by email.
- Close all software and **SIGN OUT**

## APPENDIX: Cleaning the alumina crucibles

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1. Rinse with de-ionized water the crucibles and gently dry with a kimwipe. DO NOT SCRATCH the alumina crucible with bare tweezers or any metallic sharp object. If you need to clean the bottom of the crucible, wrap the tweezers with a wet kimwipe and clean softly and gently the inner surface.
2. Ultrasonicate for 10-15min the crucible in a 1N HCl solution. Rinse with de-ionized water and dry the crucible with a kimwipe.
3. Ultrasonicate for 10-15min the crucible with Ethanol. Dry the crucible with a kimwipe.
4. Put the crucible to dry under the UV lamp for at least one hour (two hours is best).
5. If contaminants get stuck at the bottom of the crucible DO NOT SCRATCH THE SURFACE. Proceed to clean with Hydrofluoric Acid and then follow the cleaning procedure with HCl as described above.