



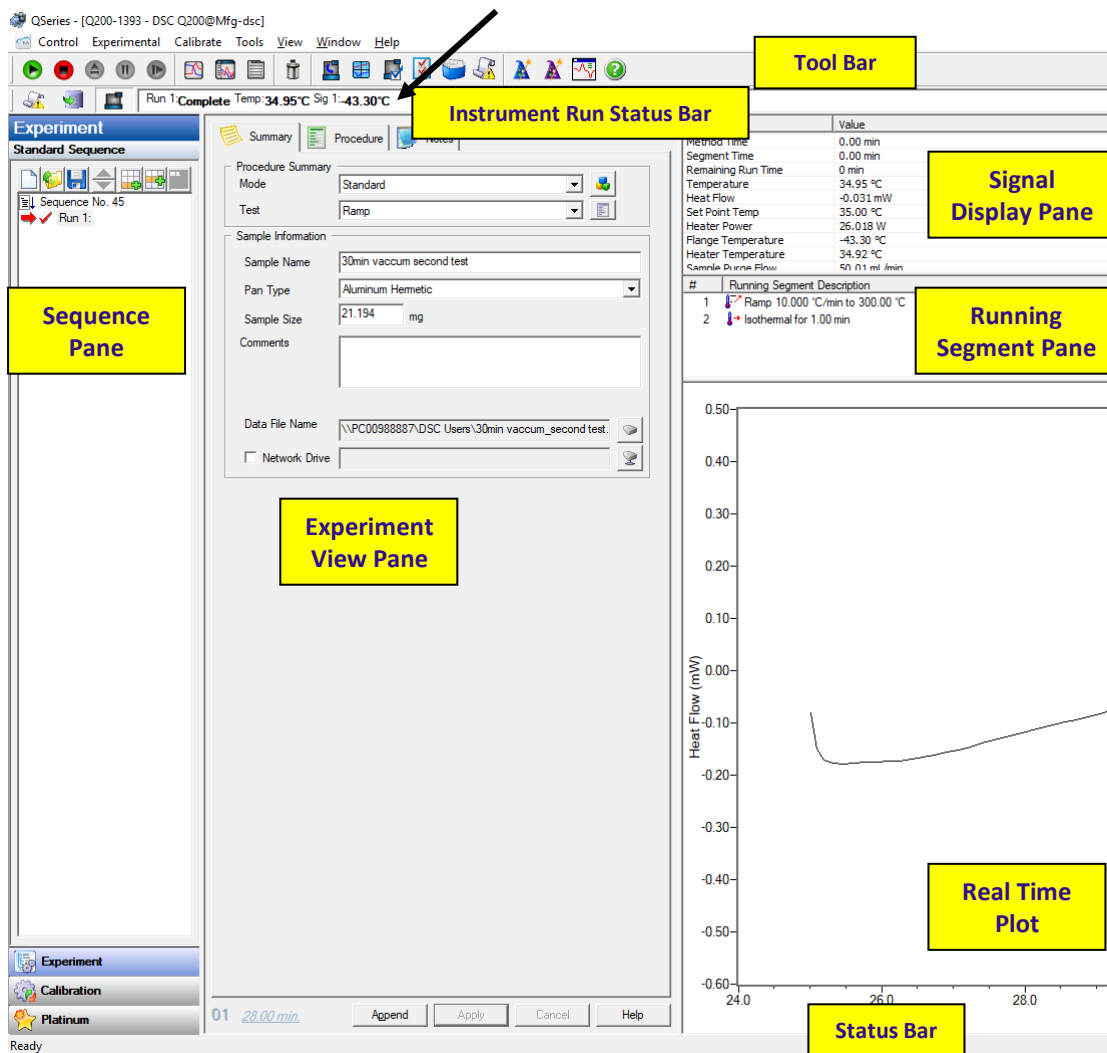
DSC quick Operation Guide

Preparing the sample for DSC measurement

1. Open Nitrogen valve 15 min before the run (**Do not adjust** the pressure regulator)
2. Turn ON the instrument (ON/OFF switch is at the instrument back)



3. Turn ON the refrigerated cooling system (RC-40). Make sure that the switch is on **EVENT**.
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**
5. **In the Control Window**
 - Select from the Menu **Control > Go To Standby Temp** and then **Control > Event on**
 - Wait until the temperature indicated as Sig1 on the instrument run status bar reaches around -40°C (indicated by black arrow on the figure below).



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.

Selecting crimped pan & sample preparation

1. Use **Standard** Pan and Lid of the same color **for solid** samples and **Hermetic for liquid** samples.
2. Always use a reference set of pan and lid of the same type.
3. Chunk or cut samples rather than crush to obtain a thin sample, which has more uniform thermal contact with pan.
4. Distribute the sample into the pan uniformly. Keep the sample as thin as possible to minimize thermal gradient. Cover as much of the pan bottom as possible.
5. Weigh about 2-5 mg (recommended) of the sample into a pan with 0.01 mg accuracy.
6. Use die set of the same color as the pan. Load the upper die into the upper portion of the press. Place the pan into the lower die set and close it with a lid flat side down. Put the lower set into the press. Pull the handle down and then pull the lower set out. Watch <https://www.youtube.com/watch?v=TvVSdfXrbbs> for the procedure.



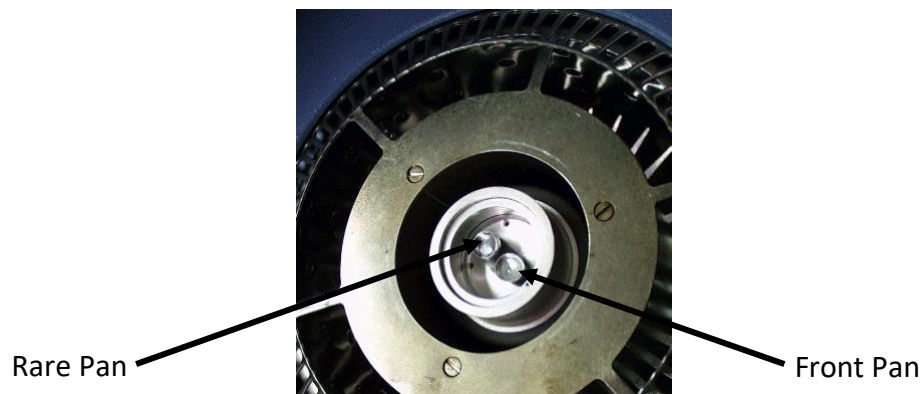
7. When Hermetic set is used penetrate a hole in a lid with a needle.
8. For very small samples use **Tzero** set of pan and lid.

9. When measuring large (>25mg) samples of materials in solution (protein, starch, etc..), use water or buffer solution in the reference pan for flatter baseline and faster stabilization.

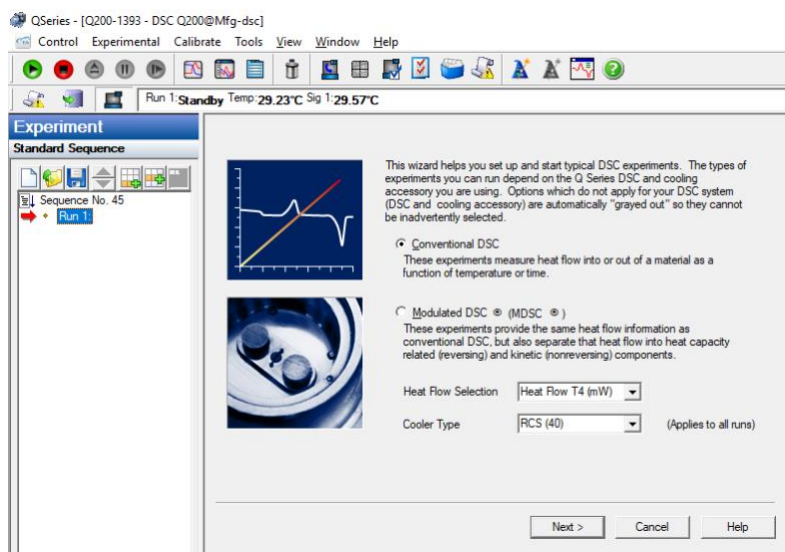
Running DSC experiment

In the Control Window

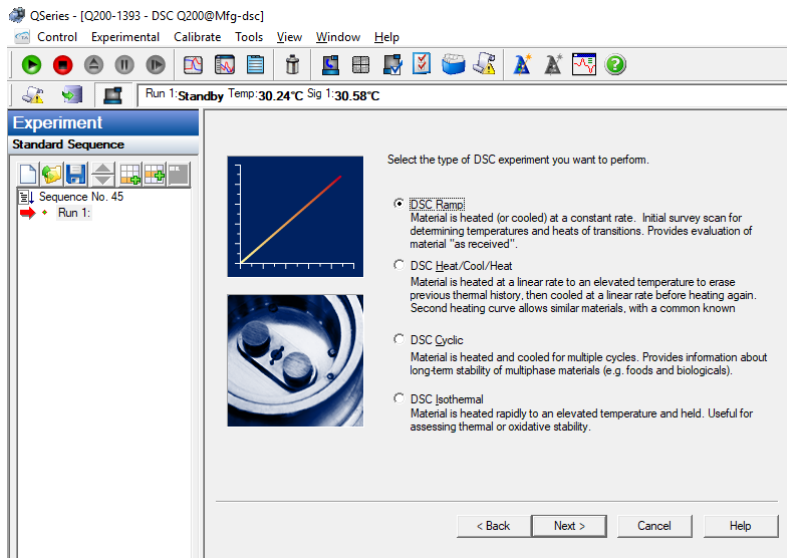
- Select from the Menu **Control > Lid > Open**
- When the lid is moved aside, carefully place the sample pan on the front raised platform and the reference pan on the rear platform. Centering the pans will give more accurate results.



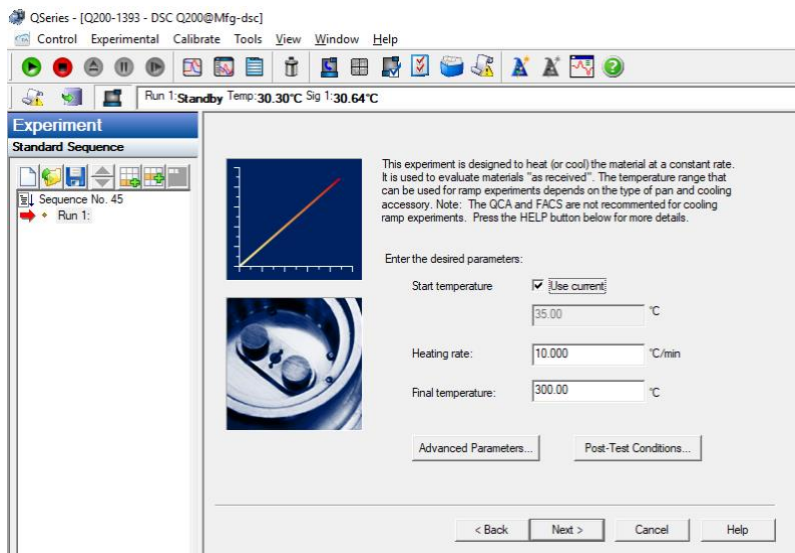
- Select from the Menu **Control > Lid > Close**
- On the Tool Bar menu click **Experiment Wizard** icon
- Make sure **Conventional DSC** is selected and press **Next**



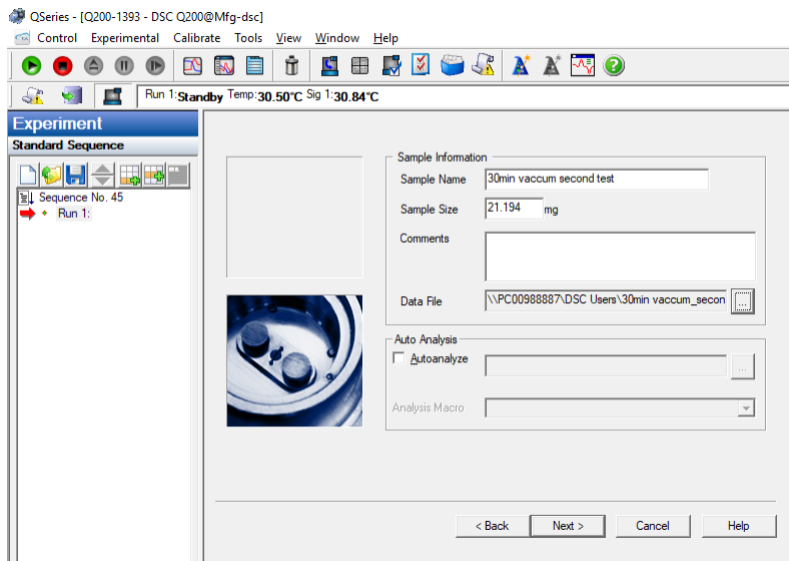
- Select **DSC Ramp** and press **Next**



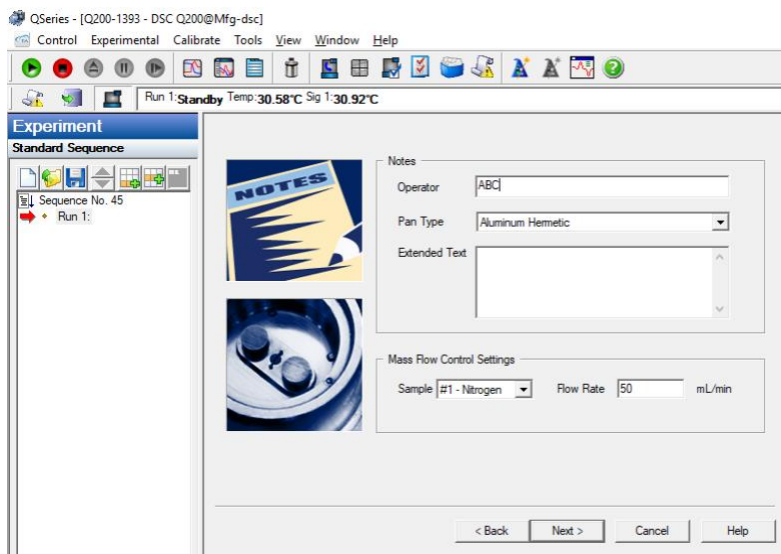
- Set starting and final temperatures and heating rate and press **Next**. The experiment is designed to heat (or cool) the material at a constant rate. The temperature range that can be used for ramp experiments depends on the type of pan. Press the **Help** button for more details.



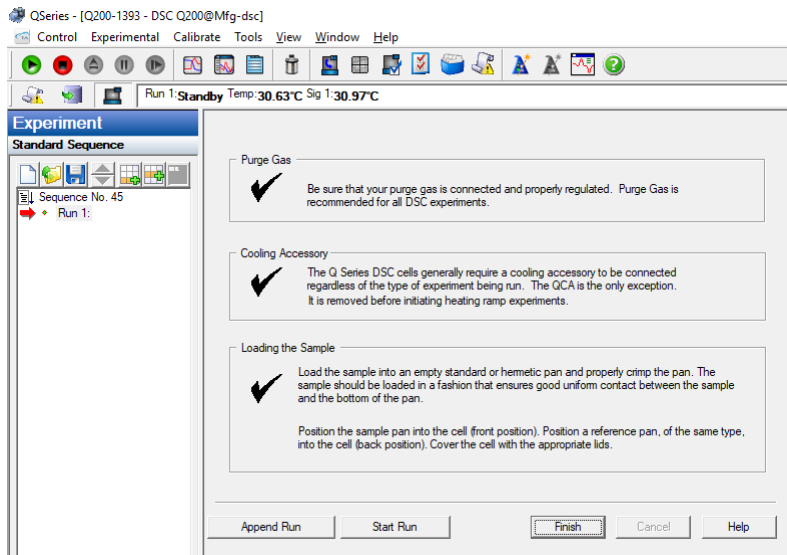
- Check the starting conditions of the experiment are appropriate and press **Next**
- Fill the sample information and data file pathway and press **Next**




- Fill the operator information. Carefully chose the pan type and press **Next**



- Click **Finish**



- Starting conditions of the experiment appear in the **Running Segment Pane**.
- On the Tool Bar menu click green **play** arrow  to **start the run**
- 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**
- Modify the method parameters as required. Add or reduce steps. Click **Send changes** and then **Close**. The method will be updated.

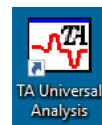
End of instrument operation

In the Control Window

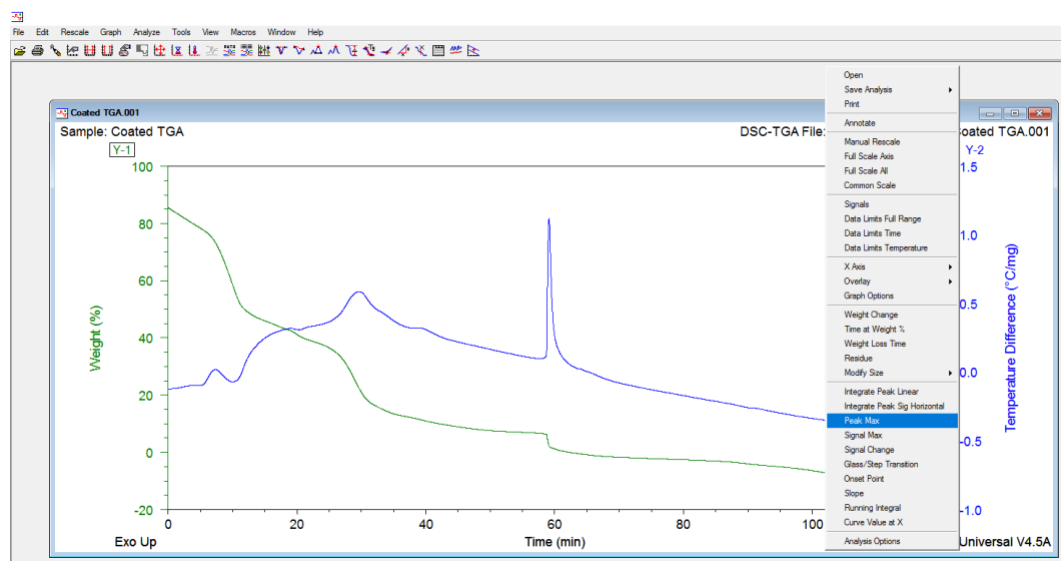
- Select from the Menu **Control > Lid > Open**
- Remove the Sample pan (if the Reference pan is empty it can stay in the instrument).
- Close the lid **Control > Lid > Close**
- Control > Event off**. Wait for Sig1 to reach around room temperature and then switch off the instrument.
- Close Nitrogen valve.



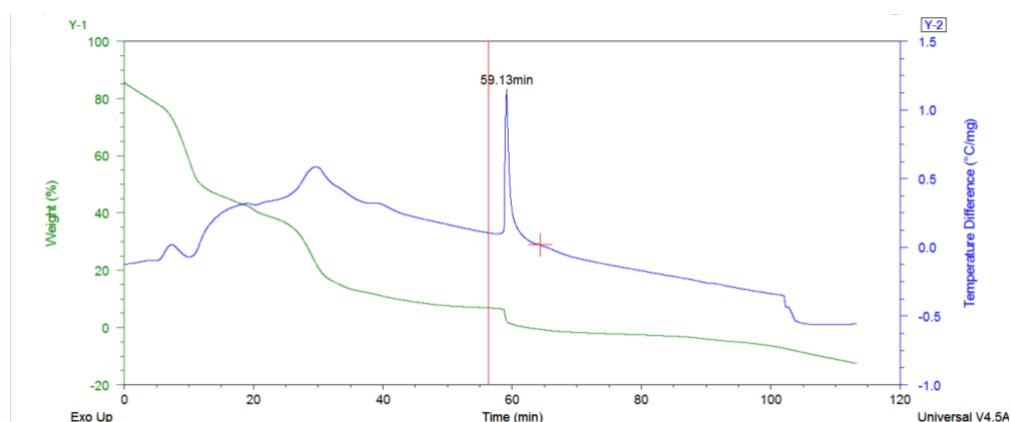
Data analysis



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.



7. 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.



8. Read “**UManual**” (open shortcut on the Desktop) for more data processing options.
9. To save the file, from the Menu select **File > Save Analysis** or **Save Analysis and Data** and click **OK**.
10. 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.
11. Close all software and **SIGN OUT**