	SOP TA Instruments SDT-Q600	**I-02**	
		Revision	03
		By	Sami Vuori
		Issued	22/4/2022

Standard Operating Procedure
TA Instruments SDT-Q600 TGA/DSC

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1. Introduction

The thermal analysis studies the relationship between a sample property and its temperature as the sample is heated or cooled in a controlled manner. The thermogravimetric analysis (TGA) evaluates changes in the sample mass as a function of temperature, while the differential scanning calorimetry (DSC) measures the differences in the heat flows between a sample and the furnace as well as a non-reacting reference material, and the furnace as a function of temperature. The TGA-DSC measurements are often used to obtain information about the physical and chemical phenomena which may be associated to mass changes and /or heat changes. This equipment provides simultaneous TGA-DSC measurements from ambient temperature to 1500 °C.

2. Safety

Normal laboratory precautions: a lab coat, gloves, and goggles.

3. Measurement

3.1. Switch on the computer and the equipment from the switch at the back of the instrument.

3.2. Log in to the computer (username **kemlaite**, password **Maja123paja**) and right click the program *TA Instrument Explorer shortcut → Run as administrator*.
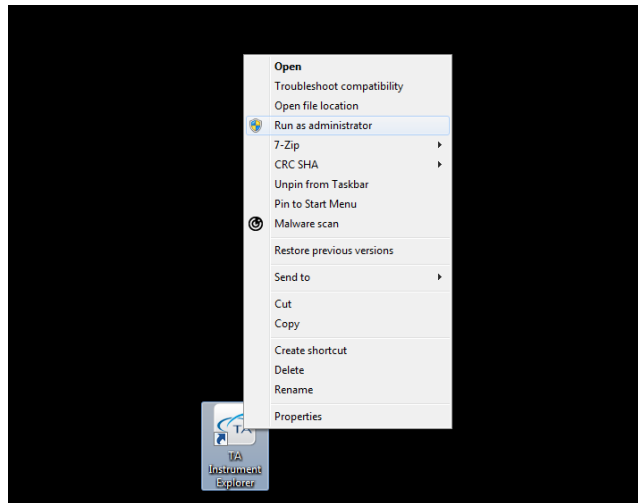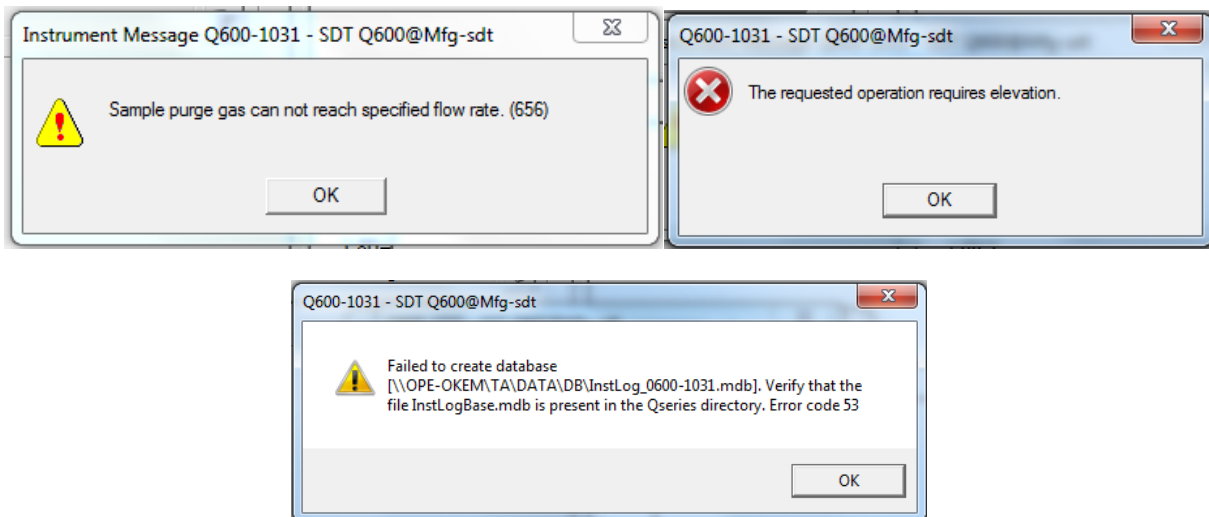
3.3. When the machine has initialized (takes a few minutes), double click the instrument in the window; there is a red circle on the icon until it's ready.

You may see some errors; just click **OK** on them.

3.4. Decide which gas you will use in the analysis and open the gas flow accordingly: use only the valve marked in the photo with red arrows; do not touch the other two marked with the red DO NOT TOUCH labels!

Gas #1 = N_2 (Typpi N_2), **Gas #2** = pressurized air (paineilma).

3.5. In the program window, click **Control → Gas → Gas 2** (or **Gas 1** if you're using nitrogen).

3.6. In the **Summary** tab, change the data:

Mode: SDT standard

Test: Custom (you may choose some other if you want, but this is generally used)
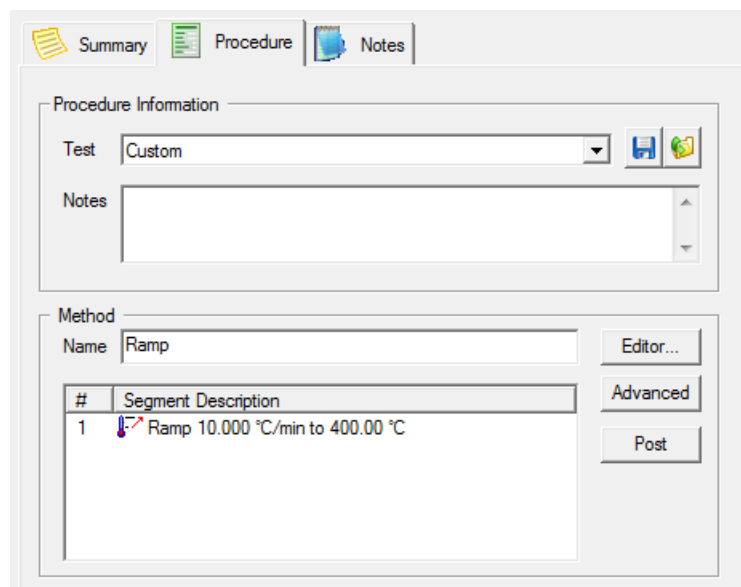
Sample name: [Your sample name]
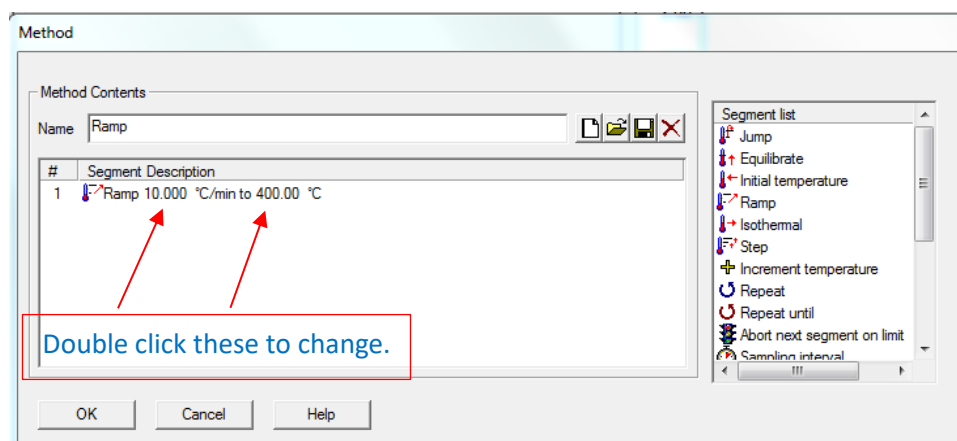
Pan type: Alumina

Comments: Air (or "N2 gas" if using nitrogen)

Data file name: make a file for this measurement by clicking the icon on the right side

3.7. Go to the **Procedure** tab and check the parameters:

If you want to change the heating parameters, click the **Editor** button and change the values by double clicking them.

3.8. Go to the **Notes** screen. Choose the gas to be used in the measurements and check that the flow rate is set to **100 ml/min**.

Check that the gas flow (**Sample purge flow**) is actually 100 ml/min in the upper right side of the window.

3.9. Open the furnace by clicking **Control → Furnace → Open**.

3.10. *CAREFULLY* place two crucibles onto the arms (they break easily and are very expensive!).

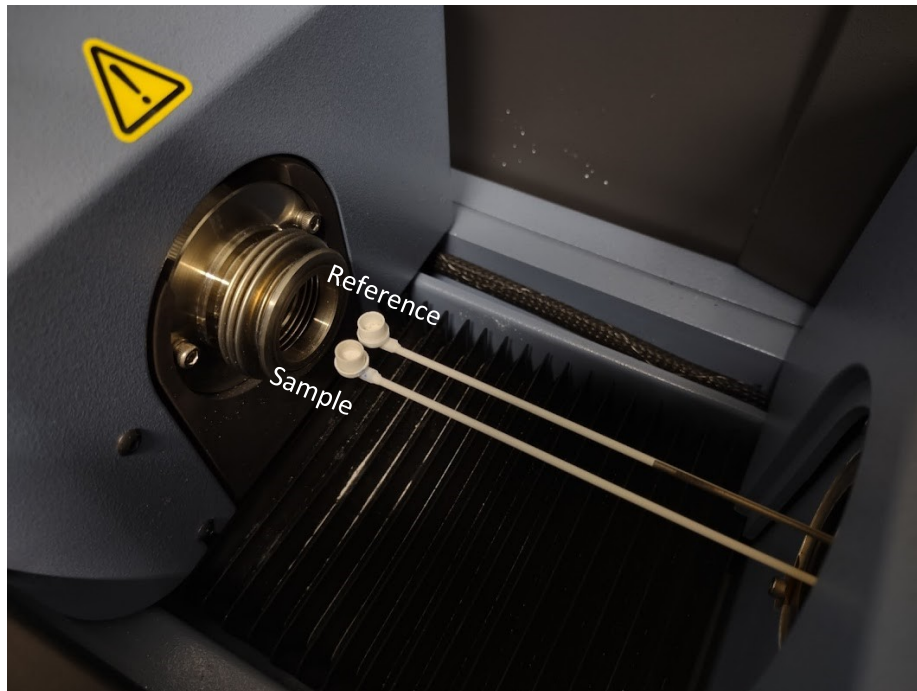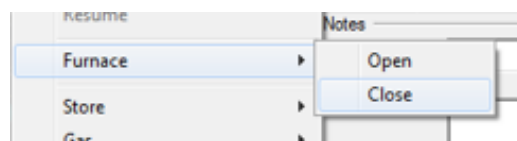**3.11.** Close the furnace by clicking **Control → Furnace → Close** and wait for it to close.

3.12. Tare the balance by clicking the scales icon.
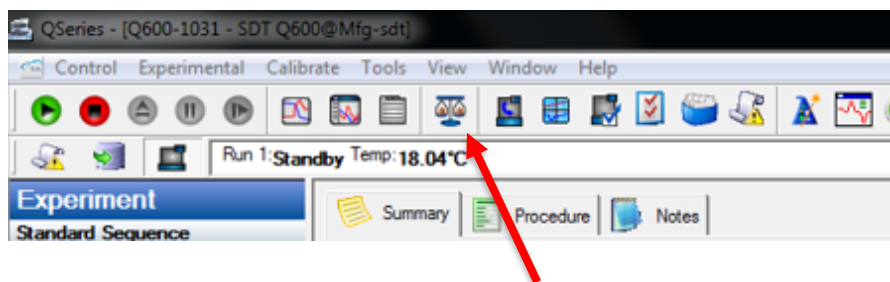
3.13. Now open the furnace (**3.9.**) again and add ~10 mg of your sample to the sample crucible (leave the reference cup empty). In the right part of the window you can check how much sample you have added. Close the furnace (**3.11.**) after you have enough. After this, in case you need the heat flow curve, wait until the heat flow has stabilized (after ~1 h). If you only need the mass loss data, you can start the analysis when the mass has stabilized.

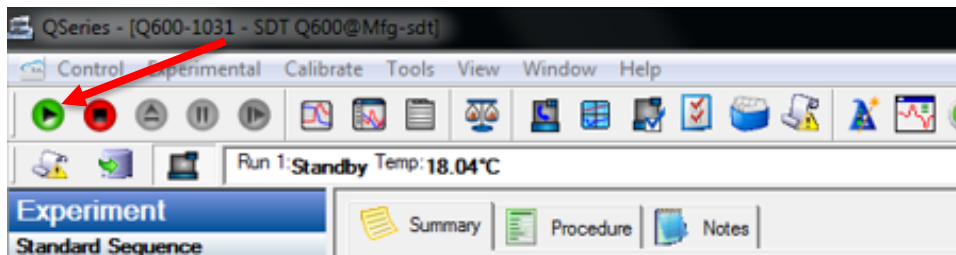**3.14.** Start the measurement by clicking the green start button.
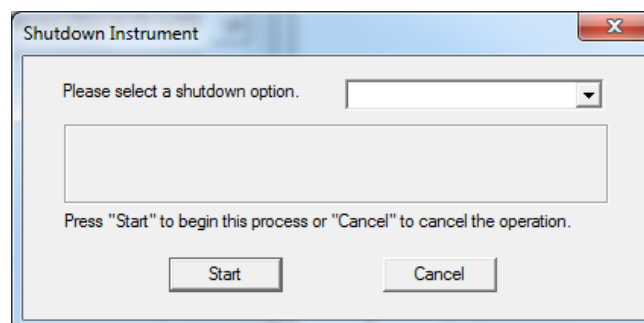
3.15. Write your run details to the instrument's notebook.

3.16. When the analysis has finished (wait until the furnace temperature is below 50 °C), open the furnace (**3.9.**), remove the crucibles and close the furnace (**3.11.**).

3.17. Shut down the instrument by clicking **Control → Shutdown Instrument**.
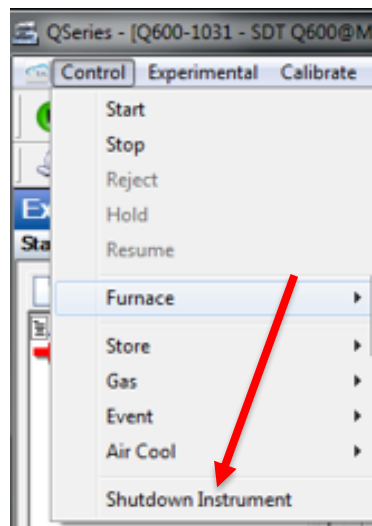
Click **Start** in the next window.

3.18. When the instrument's display shows "*Shutdown complete, it is now safe to reboot your computer*", turn it off from the back (**3.1.**)

3.19. Turn off the gas flow from the valve you opened in **3.4.**

3.20. Clean your crucibles by scraping loose material off, submerging them in aqua regia and finally firing the crucibles at 1500 °C.

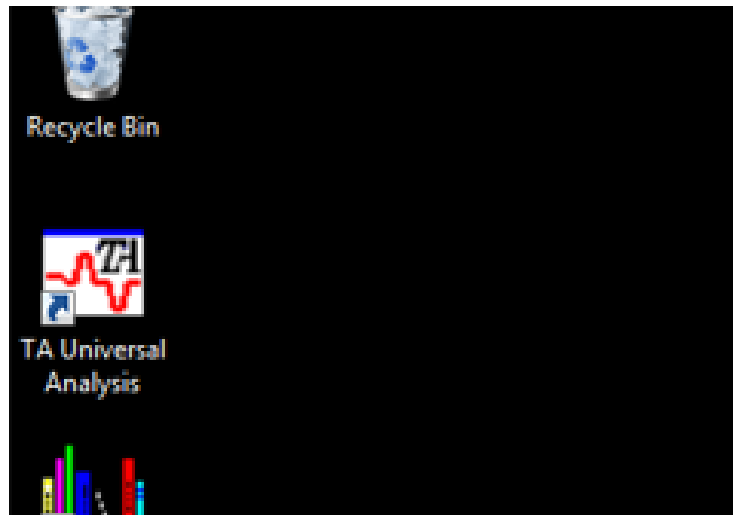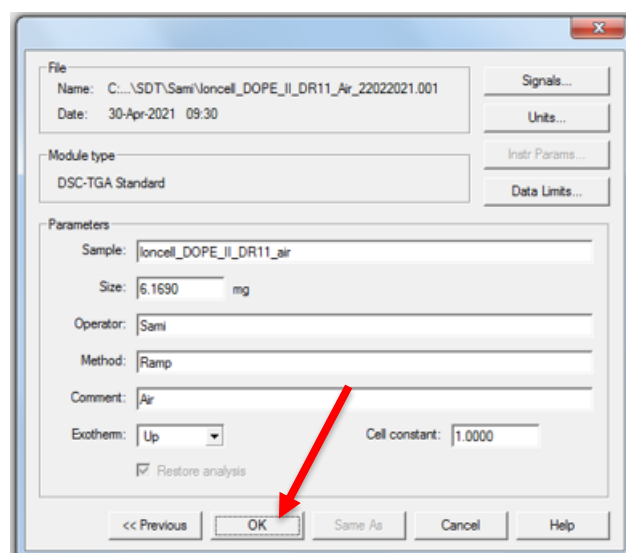|	**SOP** TA Instruments SDT-Q600	**I-02**	
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4. Data export

4.1. Double click the **TA Universal Analysis** program on the desktop.

4.2. Select your file by clicking **File → Open** and double click your file in the **C:\TA\SDT\Data** folder. Click **OK** in the next window.

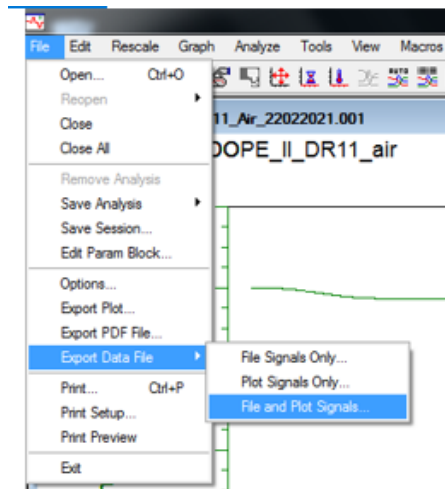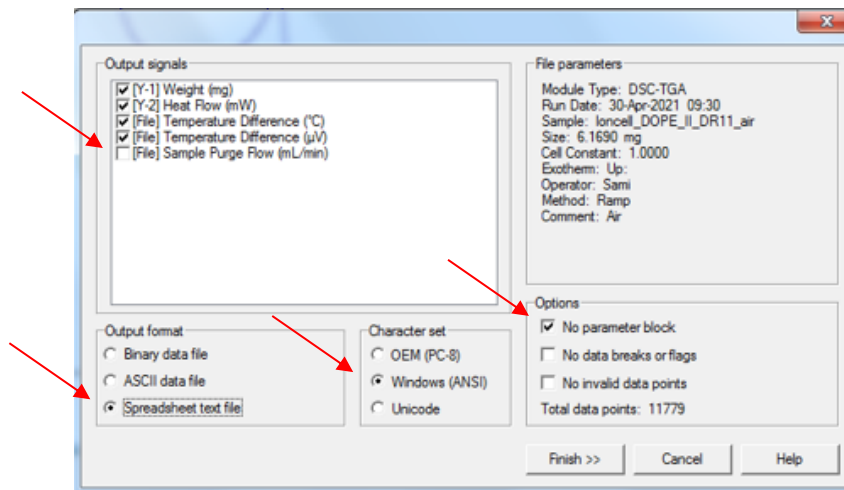|		**I-02**	
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4.3. Export the data by clicking **File → Export Data File → File and Plot Signals**.

4.4. Tick/untick the following items and click **Finish**, then save it as a text file to your folder.

5. Performance data

Balance sensitivity: 0.1 µg

TGA sensitivity: 0.001 °C

Calorimetric accuracy/precision: ±2 %

6. Version log

Revision	By	Date	Notes
01	Hellen Silva Santos	3/14/2014	Initial version
02	Hellen Silva Santos	22/03/2018	
03	Sami Vuori	22/04/2022	Updated the SOP for Aurum.