

# **Standard Operating Procedure** TA Instruments SDT-Q600 TGA/DSC

## **Table of Contents**

1. Introduction	2
3. Measurement	2
4. Data export	9
5. Performance data	
6. Version log	



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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*  $\rightarrow$  *Run as administrator.* 

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	<b>Open</b> Troubleshoot compatibility Open file location		
	Run as administrator		
	CRC SHA   Unpin from Taskbar		



**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.

Instrument Message Q600-1031 - SDT Q600@Mfg-sdt	Q600-1031 - SDT Q600@Mfg-sdt
Sample purge gas can not reach specified flow rate. (656)	The requested operation requires elevation.
ОК	ОК
Q600-1031 - SDT Q600@Mfg-sdt	
Failed to create database [\\OPE-OKEM\TA\DATA\DB\InstLog file InstLogBase.mdb is present in the	_0600-1031.mdb]. Verify that the : Qseries directory. Error code 53
	ОК

**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!

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	SOP	Revision	03	
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Gas #1 =  $N_2$  (Typpi  $N_2$ ), Gas #2 = pressurized air (paineilma).

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

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

Summary	Procedure Notes
Procedure	
Mode	SDT Standard 🗾 🛃
Test	Custom 💌 🗾 💕
- Sample Information	
Sample Name	Sample name
Pan Type	Alumina
Comments	Air
Data File Name	\\Ty1506087ta\Data\SDT\Sami\hackmanite.001
Network Drive	2

Mode: SDT standard

Test: Custom (you may choose some other if you want, but this is generally used)

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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:

≫ Sumi Procedu	re Information	
Test	Custom	- 📙 💋
Notes		A T
Method Name	Ramp	Editor
Method Name	Ramp Segment Description	Editor Advanced
Method Name	Ramp Segment Description F* Ramp 10.000 °C/min to 400.00 °C	Editor Advanced Post

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

Method			
Method Name #1	I Contents Ramp Segment Description Ramp 10.000 °C/min to 400.00 °C	Segment list ↓ Jump ↓ Equilibrate ↓ Thitial temperature ↓ Sothermal ↓ Sothermal ↓ Step ↓ Increment temperature ↓ Repeat ↓ Repeat until	
Dou	uble click these to change.	Abort next segment on limit	Ŧ
	K Cancel Help	,	

**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.

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Summary	Procedure	Notes			
Notes					
Operator	Sami				
Extended Text					
Mass Flow Cont Sample	rol Settings	•	Flow Rate	100	mL/min
Mass Flow Cont Sample Auto Analysze — Autoanalyze	rol Settings	•	Flow Rate	100	mL/min

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

Signal	Value
Temperature	18.05 °C
Weight	-183.538 mg
Weight percent.	100.00 %
Reference Weight	-187.513 mg
Heat Flow	-3.620 mW
Temp Difference (°C)	-0.42 °C
Temp Difference (µV)	-2.392 µV
Heater Power	0.00 W
Set Point Temp	0.00 °C
Sample Purge Flow	99.97 mL/min

**3.9.** Open the furnace by clicking **Control**  $\rightarrow$  **Furnace**  $\rightarrow$  **Open**.

🛋 QSeries - [Q600-1031 - SDT Q600@Mfg-sdt]							
	Control Experimental	Calibrate	Tools	View	Wind	ow	He
	Start			<u>a</u> a			Į,
H	Stop			-	_	_	-
1 4	Reject		18	.05°C			_
Б	Hold		Sumo	nary	Pr	oed	me
Sta	Resume		Notes -				
	Furnace	•	0	pen		1	
	Store	•	c	lose		⊢	_
	Gas	•					
	Event						
	Als Cool						

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

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

Resume	Notes
Furnace	Open
Store	Close
Gas	•

**3.12.** Tare the balance by clicking the scales icon.



**3.13.** Now open the furnace (**3.9.**) again and add  $\sim 10$  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  $\sim 1$  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  $^{\circ}$ C), open the furnace (**3.9**.), remove the crucibles and close the furnace (**3.11**.).

**3.17.** Shut down the instrument by clicking Control  $\rightarrow$  Shutdown Instrument.

🛎 QSeri	ies - [Q600-1031 - SDT Q600@Mf
Cor	ntrol Experimental Calibrate
	Start
÷7	Stop
9	Reject
Б	Hold
Sta	Resume
	Furnace >
	Store +
	Gas 🔸
	Event +
	Air Cool 🔸
	Shutdown Instrument

Click Start in the next window.

Shutdown Instrument	×
Please select a shutdown option.	<b>_</b>
Press "Start" to begin this process or	"Cancel" to cancel the operation.
Start	Cancel

**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.

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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  $\rightarrow$  Open and double click your file in the C:\TA\SDT\Data folder. Click OK in the next window.

	X
File Name: C: \SDT\Sami\loncell DOPE II DB11 Air 22022021.001	Signals
Date: 30-Apr-2021 09:30	Units
Module type	Instr Params
DSC-TGA Standard	Data Limits
rarameters       Sample:       Ioncell_DOPE_II_DR11_air       Size:     6.1690 mg       Operator:     Sami       Method:     Ramp       Comment:     National Samp	
Exothem: Up Cell constant: 1.0	000
<< Previous OK Same As Cance	Help

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**4.3.** Export the data by clicking File  $\rightarrow$  Export Data File  $\rightarrow$  File and Plot Signals.



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

		<b>X</b>
Output signals ▼ [Y-1] Weight (mg) ▼ [Y-2] Heat Row (mW) ▼ [File] Temperature Difference (µV) ▼ [File] Temperature Difference (µV) ■ [File] Sample Purge Rlow (mL/min)		File parameters Module Type: DSC-TGA Run Date: 30-Apr-2021 09:30 Sample: Noncel_DOPE_II_DR11_air Size: 6.1690 mg Cel Constant: 1.0000 Exothem: Up: Operator: Sami Method: Ramp Comment: Air
Output format C Binary data file C ASCII data file Gpreadsheet text file	Character set C OEM (PC-8) Windows (ANSI) C Unicode	Options  I No parameter block  No data breaks or flags  No invalid data points  Total data points: 11779  Finish >> Cancel Help

#### 5. Performance data

Balance sensitivity:  $0.1 \ \mu g$ 

TGA sensitivity: 0.001 °C

Calorimetric accuracy/precision:  $\pm 2$  %

#### 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.