

# USE OF THE SIMULTANEOUS THERMOGRAVIMETRIC ANALYSER-DIFFERENTIAL SCANNING CALORIMETRY SDTQ600.

		Date
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## PURPOSE

This SOP describes how to operate and use the simultaneous thermogravimetric analyser (TGA)-differential scanning calorimetry (DSC) SDTQ600 (TA) located in the CAF facility (for more information on the instrument please see the APPENDIX).

## SCOPE

This SOP describes procedures to be followed by staff and students when using the TGA-DSC SDTQ600 instrument in the CAF facility.

## RISKS.

DO complete the [CAF Blackboard safety test](#), official training and ONE run supervised by the CAF technician before using the equipment. For future runs you will need to book the instrument.

See the attached [Risk assessment](#) of the instrument.

You must have your [own Risk assessment](#) of the chemical products you are going to analyse.

Please if you want to analyse toxic, explosive, flammable or longer term health hazards chemicals talk with the CAF technician before running your samples ([check their MSDS](#)). Take also the MSDS of the likely degradation products into account. We may need to take extra safety measures.



## PROCEDURE.

### PRECAUTIONS

- Check the **sample compatibility** with your sample pan at temperatures, atmospheres and times you plan to use the instrument. See Appendix.
- Maximum **operating temperature** is 1500°C, **More information** about the instrument specifications and some theory/applications is found in the binder next to the instrument,
- Do **not let anything drop into the furnace or in the tray**. If anything does notify the responsible CAF technician and wait until he can assist you. Please **clean any spillage** on the instrument surrounding area.
- Please look for technical assistance if you face any instrument warning/error. **The only error you can click okay** is "this operation requires elevation". This only indicates that you do not have admin rights,
- **Do not attempt to use** if signage indicates that the instrument is out of use or is currently in use,

## INSTRUMENT STARTUP

- No other experiments should be running, since you can **only run one** sample at a time. Check the instrument status at the top bar of the software.
- The **instrument and the computer** should **always be on**, if not please ask for technical assistance.
- Ideally you should **open the N<sub>2</sub> (g)** regulator the night before the analysis or at least a few hours before it. It is located on the wall, behind the instrument, do not confused with the air pressure tab (red tube). You need to put the black gas tap vertical. Check that the flow is already adjusted to 15-20 psi (the black mark), it takes a couple of minutes to reach this value. If it is not adjusted, please use the white screw to regulate it.
- Switch on **the extractor hood** used for TGA. It is located above the UV-VIS-NIR PC, the switch is on the wall close to the TGA N<sub>2</sub> (g) regulator. You will immediately hear the extraction system working.
- **Log on** to the TAuser, password: SDTQ600caf. If somebody has signed out you will need to sig in to the PC:

Username: SCPC9016\TAuser

Password: SDTQ600caf

Do not log on to Rdg-home with your individual logon as the instrument specific software will not function.

- To **open the instrument software**, on the computer double click on the "TA Instrument explorer" and then on the SDTQ600 icon, if they are not already open.

TARING THE SAMPLE PAN. This has to be done before the sample is loaded,

See the APPENDIX for the **kind of pans/cups** you can use with this instrument.

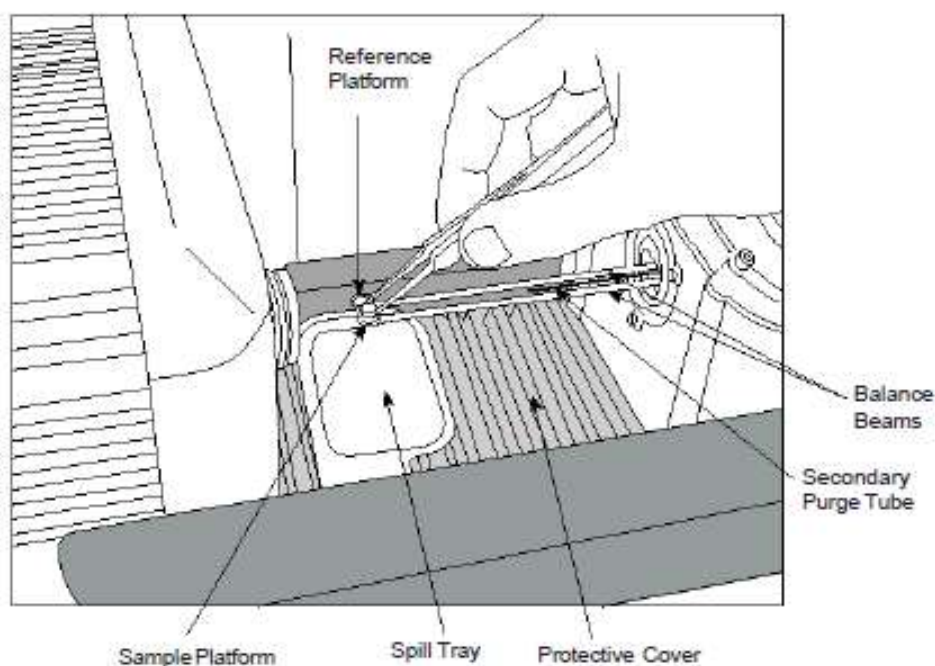


Figure 1. Loading the SDT sample pan.

**CAUTION:** The balance is quite delicate.

The **balance beams are extremely fragile** and expensive. **Failure to comply** with this SOP which result in damage to the instrument may involve restriction of CAF access and payment of the damaged parts.

Always use **tweezers** to handle the pans/cups.

While the instrument is in progress **do not lean on** the bench since the balance is very sensitive.

If there is **any problem during the pan loading process**, please press the **red button** on the instrument software and look for technical assistance. This stop option (not the reject one) saves the data.

Always carry the pans in a **metallic tray** to avoid any spillages.

- Select the **EXPERIMENT tab** on the left hand menu bar of the software.
- To **open the furnace** go to the top of the software and select Control > Furnace > Open. Inside there are two beams (which contain the thermocouple) where the pans area seated (see the Figure 1).
- Underneath the black hand rest there is a **spill tray**, you should push this under the beams to catch anything that gets dropped.
- Place an **empty SDT cup** on the platform for the front **sample** balance position, making sure that it is seated properly (see the Figure 1). To do this rest your hand on the black hand rest, it is useful to use your spare hand to steady the other.

**CAUTION:**

Reproducible positioning of the sample and reference cups on the sample and reference platforms (beam sensors) is critical to obtaining optimum SDT performance. Therefore, **always put the pans on the beams in the same direction**. To this end it is recommended to very gently push both pans to the left edge of the beams (all the way to one side of the platform). You do not need to put a mark on the pans to get the same rotational placement as explained in the manual.

**Before the furnace is closed**, be sure this area is clear.

- Place an **empty SDT cup** on the platform for the back **reference** balance assembly, making sure that it is seated properly (as previously done). Return the spill tray to its position under the armrest.
- To **close the furnace** go to the top of the software and select Control > Furnace > Close.
- Select **calibrate tare** from the top of the software. **Do not lean on the desk** while the instrument is taring. The instrument status (next to the furnace T, in the top bar, below the control tab) should change from standby to tare and then to standby again, it goes very fast.
- **Open the furnace** as previously explained. Remove the sample cup from the front (sample) platform.

## LOADING THE SAMPLE

### CAUTION:

**Spilling sample material** on the platform could cause permanent contamination of the platform and of the beams if you do not clean it before running the sample. Therefore, **remove the cup from the beams when loading** your sample and always **use the metallic plate** to carry the sample pan.

- You need to **load your pan away** from the instrument, on a piece of aluminium foil, on the bench. This will be the loading/dirty area. Remember to keep clean the metallic tray that you use to load your samples.

### What is the best way to prepare a sample?

*This depends on the nature of your sample. Below are six (6) suggestions to consider when preparing a sample.*

- *The sample size should be between 2 and 10 mg.*
  - *If you have minimum amount of sample or a dangerous sample, run at least 1 mg.*
  - *If possible, only cover the bottom of the pan (fine layer) with the sample material.*
  - *The sample pans, can accommodate liquids, powders, films, solids or crystals.*
  - *Once you have decided on the sample form, then for best results, use approximately the same sample weight during each experiment. This will ensure reproducibility.*
  - *Many small pieces of sample (ground samples) are better than one large chunk. It is better to have a large surface area exposed to the sample purge.*
- Use a small spatula to **add about** half a spatula tip of the sample to your pan. Carefully smash the sample on the pan to spread it over the bottom.
  - Gently **tap the pan** to remove the out (if solid). Remember that the sample should only cover the bottom to avoid any contamination of the instrument pan.
  - To avoid any contamination **put a piece of aluminium foil covering** the protective cover which is below the spill tray (see Figure 1) and rotate the spill tray into position under the beams.
  - **Place the sample cup back on** the sample platform, making sure that it is seated properly with the correct orientation (see Figure 1). To do this rest your hand on the black hand rest, it is useful to use your spare hand to steady the other.
  - **Remove** the piece of aluminium foil from the protective cover and return the spill tray to its position under the armrest.
  - To **close the furnace** go to the top of the software and select Control > Furnace > Close.

*NOTE: If the weight is out of range (Range), the furnace will not close. This feature prevents damage to the balance beams.*

## RUNNING A SAMPLE IN A CUSTOM PROGRAMME.

It is recommended to leave the **sample** in the **closed furnace for 10 minutes** before running it. In the meantime you can set up the method through the instrument control software. However, write down the weight of your sample and pay attention to its change. If there is a significant change look for technical assistance, you may need to start the run as soon as you have set up the method. The weight value is at the top bar, together with the N<sub>2</sub> flow and the temperature.

- Select '**Run 1**' and delete any other runs, remember that you can only have one run at a time. As default settings this run will have the programme parameters last used on the system.

- Enter details under '**Summary**' tab

Mode → The standard mode. This mode is used for conducting all other non-calibration experiments. In this mode the signals to be stored for each experiment can be individually selected. Some of the available signals are Weight, Heat Flow, Delta T (°C) and Delta T (microV).

Test → Custom

Sample name → As chosen by user

Pan type → Alumina (ceramic)

Comments → As required by user. This information is displayed next to the file when you open it.

Data File name → C:\TA\DATA\SDTQ600\USERS DATA\YOUR SUPERVISOR'S FOLDER\YOUR OWN FOLDER (each user should create his own folder)

- Enter details under '**Procedure**' tab

Test → Custom

Notes → Not needed

Method → You can open, save or create a new method. You can also modify the current method without saving the changes.

To create a method select Editor and double click on the desired segment of the list to add it to your method.

Common method:

(You can start with a equilibrate at a T few degrees higher than current T or at the current T)

Ramp: \_\_\_ °C/min to final T \_\_\_ °C. **If you want to use other segments**, please first talk with the CAF technician. Some segments are not allowed.

(Heating rate, choose between 0.1 and 100 °C/min below 1000 °C, common ones are 10, 20 °C/min. 1000-1500 °C: the maximum heating rate is 25 °C/min)

(Final T: from room T to 1500 °C, if you want to use it above 1000 °C please first talk with the CAF technician)

If you want you can save the method file using the diskette icon. Then click OK.

Please do not change:

*Advanced parameters* (0.50 s/pt)

*Post-test parameters* (furnace open: it may automatically open below 600 °C, do not select air cool)

- Enter details under '**Notes**' tab
  - Only complete Operator name
  - Don't change any other option (N<sub>2</sub> flow rate must be 100 mL/min)
- Click on '**Apply**' to save details.
- **Select** the only run (1) of your sequence list. The red arrow should appear opposite Run 1, if not move the mouse cursor to the left of "Run 1" and click.
- **Before starting** there should not be a hash on the weight signal, which would indicate that the balance is not stable.
- To **START** the experiment press the Start icon on the left hand side of the top bar of the instrument software. The system will automatically load the sample pan, close the furnace and run the experiment to completion.

**CAUTION:**

The instrument is very sensitive to motion, so **it is advisable not to work near** the instrument after you have started the run.

If there is **any problem during** the pan loading process or during the run, please press the **red stop** icon on the instrument software, left hand side of the top bar) to stop the experiment and look for technical assistance.

**Furnace is hot:** Do not touch the surface area or allow any material that may melt or burn to come in contact with this surface.

- If you already have the results you were expecting, **you can stop** the experiment at any point by selecting the stop red icon (not the reject one). The data will be saved.
- You can **extend the run** beyond the initial temperature while the instrument is running. On the right hand side of the instrument software you can find the running segment description, right click and select modify running method. Then save changes.
- If you want to get **reproducible results** more than one run is recommended.

## SHUTDOWN PROCEDURE

- At the **completion of the run**, the furnace will remain closed (it may open below 600 °C)
- Allow the furnace to **cool down** to at least 60 °C. This may require 30 minutes. The T is displayed within the instrument software at the top bar of the screen. To check the T the furnace has to be closed.
- In the meantime, fill in the SDTQ600 **Excel Logbook** (Desktop).
- To **open the furnace** go to the top of the software and select Control > Furnace > Open.
- Place **a piece of aluminium foil covering** the protective cover and place the spill tray too.
- **Remove the sample cup** from the platform and the reference cup too if you are not going to analyse more samples. Always remember to use the metallic plate to carry the sample pan.
- **Remove** the piece of aluminium foil from the protective cover and return the spill tray to its position under the armrest.
- You **can clean** your pan to reuse it. Please see the appendix.
- To **close the furnace** go to the top of the software and select Control > Furnace > Close. Ask the CAF technician for a couple of spare cups to have them on the beams, which is recommended.
- Leave the instrument and the PC **on**.
- **Only if** the furnace T is around 20 °C and you are the last user of the day, you can **switch off the N<sub>2</sub> (g)** supply. You need to put the black gas tap horizontal.
- **Switch off** the extractor hood, which is closed to the TGA.
- Please send all your **data by email**, do not save it in a pen drive. Please note that data may be purged from the instrument once a year (after back up) and therefore backing up your data onto your own computer regularly is advisable. For data analysis please see Appendix.

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## APPENDIX.

### PURPOSE OF THE INSTRUMENT:

The SDT is used to perform simultaneous measurements of TGA and either DSC (most commonly used) or DTA, depending upon the desired experiment. This nomenclature refers to experiments that save heat flow and weight, or weight and delta T ( $\mu\text{V}$  and/or  $^{\circ}\text{C}$ ), respectively. Throughout this SOP you have seen how to use the instrument for DSC-TGA experiments.

According to specialist engineers the DSCQ2000 is more accurate for its range of T than the SDTQ600.

You can also perform **TGA-DTA** experiments with this instrument. However, there are several issues to take into account for these kind of analysis. Please look for technical assistance if you want to perform these analysis (there is more information in the help menu and in the online documentation). Some of these issues are:

- The kind of pans you can use, see the beginning of this appendix.
- After taring the SDT cups, for TGA-DTA experiments it is recommended that you load your reference material (such as aluminum oxide). The purpose is to minimize the difference in heat capacity between the reference and sample cups; thereby improving the baseline. Reference material should not be used for DSC-TGA experiments. Rather, calibration and subsequent sample experiments should be performed with an empty alumina cup as the reference.

### SAMPLE COMPATIBILITY WITH YOUR SAMPLE PAN.

Samples that react with a pan can cause serious damage to an instrument since they may also react with the furnace beneath. Solder pastes and inorganic salts are typical of the type of samples where care must be taken. If in doubt check it out separately from the instrument, and then choose a pan type which is inert.

Alumina (ceramic) and platinum pans can be used with the SDTQ600. Both are not disposable items, so you will need to clean them after use.

- **Ceramic** (alumina 90 $\mu\text{l}$  cups) are the recommended in the manual for DSC-TGA experiments. These cups are compatible/inert with most high temperature materials.

**There are alumina lids** that can be used to keep sample to the bottom of the cup: The SDT also takes a DSC signal and this is highly dependent on the contact of the sample with the sample pan (resolution suffers if there is poor contact). It makes little difference for the TGA signal unless you have a very light weight material where the purge gas can blow sample away. Again, the lid will help prevent that. It's quite unusual to use lids unless for these special circumstances.

- **Pt pans** can also be used but they are not recommended in the manual for DSC-TGA experiments. Be aware that inorganics and corrosive materials may react with these pans. Metals will most likely react with Pt pans if you get close to their melting temperature. Pt also has a catalytic effect.

Pt pans can be used up to 300-400  $^{\circ}\text{C}$  maximum according to a specialist engineer. For higher T the pans may stick to the Pt sensor of the beams, which would result in the need to replace them. To prevent this you need to place fine-grain alumina powder (stored in a desiccator) between the Pt cup and the sensor before starting the experiment.

- For TGA-DTA experiments: please see the SDTQ600 get start guide (desktop), page 43.

## CLEANING THE PAN.

This procedure must be done outside CAF lab, following your own SOP and risk assessment. The following information are just general guidelines.

You can clean both Pt and ceramic (alumina) pans.

Gently tap the residue in the pan into the waste container (yellow bin or toxic barrel). Take care not to chip or dent the pans.

Put the pan in a small glass vial full of solvent (any solvent that could dissolve your sample residues) and sonicate it. The alumina crucibles may be cleaned by soaking in mineral acid and then rinsing well with deionised water.

Sometimes, you may need to burn the pan in a Bunsen burner to clean it perfectly ( $\text{Al}_2\text{O}_3$  melting p. 2054 °C and Pt 1772 °C). Hold the pan in the flame with ceramic tweezers until red-hot for about 15s. Place the pan on a ceramic tile to cool it down. Then you might need to scrape off remaining ash (DSC fiberglass brush).

*Caution: Do not heat the furnace to high temperatures to clean the pans, clean them outside as described above. Leaving the furnace at high T for extended periods of time may shorten its life.*

## DATA ANALYSIS.

You can use TA UNIVERSAL ANALYSIS SOFTWARE, installed in the desktop of all the Thermal PCs. You can also download the software from TA website: <https://www.tainstruments.com/installation-of-universal-analysis-from-the-advantage-software-package/>. Ask the CAF technician for a training. You can find a manual in the Desktop too.

You can export your data into Excel selecting File > EXPORT DATA FILE > plot signals or file and plot signals > then select: spreadsheet text file, Unicode, finish and save it adding the ".txt" to the file name. Please be careful since not all the data are exported in the desirable units (such as weight normalised units).

You can also export the plot: File > Export plot > Choose the following options: clipboard, metafile, full size, pen width 3 is (the default). Then select export and paste it wherever you want. You can also remove the head of the picture: right click on the picture and select crop.

To save the graph as a PDF you can select file > save as > analysis and data or export as PDF.