

USE OF THE THERMOGRAVIMETRIC ANALYSER TGA Q50.

		Date
Author:	Pedro Rivas Ruiz	25/03/2020
Reviewed by:		
Approved by:		
Authorised by:		
Next review:		

PURPOSE

This SOP describes how to operate and use the thermogravimetric analyser TGA Q50 (TA) located in the CAF facility.

SCOPE

This SOP describes procedures to be followed by staff and students when using the TGA Q50 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 the 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 800°C, Please talk with the CAF technician if you need to go beyond this limit, we have an alternative instrument. **More information** about the instrument specifications and some theory/applications is found in the blue binder next to the instrument,

- Do **not let anything drop into the furnace**. 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 "The requested 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** (this is the one close to the DSC) should **always be on**, if not please ask for technical assistance.
- Ideally you should **open the N₂ (g)** regulator 30 minutes before the analysis. It is located on the wall close to the UV-VIS-NIR, just under the extraction hood. 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** above the UV-VIS-NIR PC, the switch is on the wall close to the N₂ (g) regulator. You will immediately hear the extraction system working.
- **Log on** to the DSC CAF user, password: Tgacaf19. If somebody has signed out you will need to sig in to the PC:

Username: SCPC9017\dsccaf Password: Tgacaf19

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 TGAQ50 icon, if they are not already open.

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

CAUTION: The balance is quite delicate.

Do **not** attempt to **load a pan manually**, always use tweezers to handle the pans.

While the instrument is in progress **avoid bumping** the bench and the instrument.

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

- **Place your empty sample pan** on the centre of instrument Pt pan. There is a groove on the sample platform where the instrument Pt pan has to sit perfectly. To this end the wire of the instrument Pt

pan should align with the white marks of the groove so that this pan can be picked up by the hang-down wire.

- Press the **tare button** on the TGA control keypad. Alternatively, you can select calibrate tare from the instrument software.
- The pan will **automatically** be loaded and the furnace raised. When the tare procedure is complete, the furnace will automatically lower and unload the pan.

LOADING THE SAMPLE

- **Remove the instrument Pt pan** with your pan from the platform and place it on the clean metallic plate (Figure 1). Then took your pan and place it on a piece of aluminium foil, on the bench. This will be the loading/dirty area.

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**. See more information in Appendix.*
- *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.*



Figure 1. How to pick up the instrument pan.

- 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.
- Place the **sample pan on the instrument Pt** pan and place this Pt pan on the instrument platform. To avoid any spillage please always use the metallic plate to carry the instrument Pt pan.
- Remember that the wire of the instrument **Pt pan should align** with the white marks of the groove so that this pan can be picked up by the hang-down wire.
- Press the **sample button** on the instrument Keypad. Alternatively, you can select Control > Sample > Load from the instrument software.
- The instrument will **automatically** load the sample pan onto the furnace.

- **Only if** you have used the instrument software to load the sample pan, now you will need to select Control > Furnace > Up to raise the furnace.

RUNNING A SAMPLE IN A CUSTOM PROGRAMME.

It is recommended to leave the **sample** in the **closed furnace for 5 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 N₂ flow and the temperature. For samples sensitive to oxygen please see the Appendix.

- Select the **EXPERIMENT tab** on the left hand menu bar.
- 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 → It depends on the pan selection.

Test → Custom

Sample name → As chosen by user

Pan type → Platinum, Alumina (ceramic) or Aluminium.

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

Data File name → C:\TA\DATA\TGA\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)

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, common ones 10, 20 °C/min)

(Final T: from room T to 800 °C, for Al pans the maximum T is 590 °C).

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 (furnace closed, do not select air cool)

- Enter details under '**Notes**' tab
 - Only complete Operator name
 - Don't change any other option (N₂ flow rate must be 40 mL/min for the balance and 60 mL/min for the sample)
- 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. The hash indicates that the balance is not stable.
- To **START** the experiment press the Start green button on the instrument keypad or click the green start icon on 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:

While the instrument is in progress **avoid bumping** the bench and the instrument.

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

- If you already have the results you were expecting, **you can stop** the experiment at any point by selecting the stop red icon on the instrument software (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 remain closed to avoid any toxic gas release.
- Allow the furnace to **cool down** to at least 60 °C. This may require 15-30 minutes. The T is displayed within the instrument software at the top bar of the screen.

- In the meantime, fill in the TGA **Excel Logbook** (Desktop).
- To automatically **unload** the pan press the sample button on the instrument keypad. Alternatively, you can select Control > Sample > Unload from the instrument software.
- With one hand **remove** the instrument Pt pan with your sample pan from the platform. Meanwhile, with the other hand, hold a clean metallic plate underneath the pan to avoid any spillage.
- If you have used an aluminium pan, please **dispose** it in the plastic vial close to the instrument or in the toxic barrel. However, if you have used a Pt or a ceramic pan, you **can clean** it to reuse it. Please see the appendix.
- **Replace** the instrument Pt pan on the platform. It should align with the white marks. If you have dirtied it please look for the CAF technician.
- Then **close the furnace** by pressing the Furnace button on the instrument keypad, or select Control > Furnace > Up on the instrument software.
- 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 N₂ (g)** supply. You need to put the black gas tap horizontal.
- **Switch off** the extractor hood.
- 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.

Technical support: Dr Pedro Rivas scs14s2@reading.ac.uk

APPENDIX.

METHOD FOR SAMPLES SENSITIVE TO AIR (OXYGEN).

For samples sensitive to air, it is a good idea to have the sample held in the furnace for 10-15 minutes before starting a run in Nitrogen. This is to allow air to be flushed out of the furnace prior to measurement. Ideally you should leave the purge gas flowing through the Instrument for 5-6 hours before running measurements where samples are highly Oxygen sensitive. This is to allow all the air inside the balance mechanism from being flushed out as well.

Use the following method for Oxygen sensitive measurements

1. Jump to Zero Deg C (This will hold the sample at room temperature (No power supplied to the furnace)).
2. Data storage OFF (you can delete this segment if you want to save this part of the method to know if the weight of your sample has changed during this isothermal period).
3. Isothermal 10 Minutes.
4. Data storage ON (not needed if you have deleted the previous data storage off order).
5. Ramp X deg C/Min to Y deg C (or any other segment you wish to use).

Keep the furnace in the closed position when not in use.

SAMPLE COMPATIBILITY WITH YOUR SAMPLE PAN.

Samples that react with a pan can cause serious damage to an analyser 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 analyser, and then choose a pan type which is inert. Sometimes the effect of catalysis is of interest and copper pans may be used to provide a catalytic effect.

- Pt pans can also be used. However, 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 too. Pt has a catalytic effect

- Ceramic (alumina) are usually the most inert option.

- Aluminium pans are normally made of very high-purity metal to prevent unwanted catalytic effects. Corrosive chemicals may react with these pans (especially bases), water can also react with the aluminium used to make these pans.

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 only clean Pt and ceramic (alumina) pans.

Gently tap the residue in the pan into the waste container (yellow bin or toxic barrel).

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 (Al_2O_3 melting point $2054\text{ }^\circ\text{C}$ and Pt $1772\text{ }^\circ\text{C}$). 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.

FACTORS AFFECTING REPRODUCIBILITY.

Because most events that occur in a TGA are kinetic in nature (meaning they are dependent on absolute temperature and time spent at that temperature), any experimental parameter that can affect the reaction rate will change the shape / transition temperatures of the curve. These things include:

- Pan material type, shape and size.
- Ramp rate.
- Purge gas.
- Sample mass, volume/form and morphology.

For more information see the printed ppt presentation of the instrument binder: Thermal Gravimetric Analysis. TA. University of Alabama at Birmingham.