Protocol
Thermogravimetric Analysis (TGA)

1. Method

Thermogravimetry (TGA) is a technique that measures the change in weight of a sample when it is heated, cooled or held at constant temperature. Its main use is to characterize materials with regard to their composition.

2. General recommendations

- When expecting significant weight loss > 50 [%], 5 [mg] of powder is enough.
- If expecting 5-20 % weight loss, then 10-20 mg of powder is normal.
- The nanopowders must be put in the crucible under the fume hood.
- If the material decomposes giving large quantities of gas, it could lead to an expulsion of the sample from the crucible. In this case, one must fill the crucible not more than 20%.
- If the sample forms a liquid phase, one must be sure that this phase does not react with the crucible (mostly Al_2O_3). Otherwise, do not program above 30 °C below the melting point temperature ($T_{mp} – 30$).
- If the sample is sensitive to moisture (H_2O vapour) or to oxygen, it can modify the initial weight and loss. This must be taken in account for further interpretation.

3. Equipment

- Instrument: Mettler Toledo TGA/SDTA 851e (more info from http://us.mt.com/home);
- Automatic sample robot (34 samples);
- Crucibles in alumina (mostly 150 [µL]).

4. Protocol

Defining a measurement program

- Start the STARe software (username : LTP / password:no password)
- In the left window, go to “Editeur de routine”
- In the Méthode menu, choose “Nouvelle”
- Select “Ajouter dyn” to define a heating/cooling step
    - Introduce the starting temperature in “Température de:”
    - Introduce the end temperature in “Température finale:”
    - Introduce the heating/cooling velocity in “Vitesse de chauffe:”
    - Introduce the gaz used for the analysis in “Gaz du segment” (mostly at 30 [mL/min])
- Select “Ajouter iso” to define a plateau of temperature
    - Introduce the temperature in “Température finale”
    - Introduce the plateau length in “Durée”
- Add as many steps as required for the analysis
- Choose the type of crucibles in “Creuset”: mostly Oxyde aluminium 150 [µL]
- Save with “Enregistrer”. Give a name to the method, then OK.

Starting the program

- Place the gas arrival (air, N_2…) on the apparatus
- On the TGA apparatus, press the "double bottled" button, and regulate gas ingress to the chosen flow (mostly at 30 [mL/min]). Press the "double bottled" button again until the temperature indication appears.
- Place the empty crucibles on the automatic robot
    - Always add a crucible at the end of a series of samples for the blank
    - Replace the cover carefully
- Start the STARe software (username : LTP / password: none)
- In the left window, got to “Expériences sur module”
- With a right click, select “Vider la liste des expériences”, to delete all the former measurements
- Then in the left window, go to “Editeur de routine”
- In the “Méthode” menu, choose “Sélectionner”. Choose the method (defined as described above) in the list. Click on “Ouvrir”
- Name the first sample in “Nom d’échantillon”, and select its position in the automatic robot (101). Click on Reset, then click on “Envoyer expérience”
- If there are several samples for the same method, put the corresponding name and position, and click on “Envoyer expérience” each time
- The last sample must be a blank
- Once all the samples have been added, go to “Expériences en file d’attente” in the left window. Select all the samples, and with a right click, select “Pesée automatique”, then “Creusets”, and OK. The empty crucibles are then weighed (around 2 [min] per sample).
- When the empty crucibles have been all weighed, remove them and fill them with the desired powder. Place your filled crucibles back into the machine ***in their original position***, and close the cover.
- Go to “Expériences en file d’attente” in the left window. Select all the samples ***but not the blank***, and with a right click, select “Pesée automatique”, then “Echantillon”, and OK. The crucibles are weighed with the powder (around 2 [min] per sample).
- When all the samples have been weighed, go to the "Contrôle" menu, then "Démarrer expérience". A new window appears that will display live results. The amount of time required for the measurement will be indicated (but this time does not include the cooling time of the cell, that is approximately the same as the rising temperature time)

5. Presentation of the results, data storage and data treatment

Collect the data

- Go to the root STARe programm and select “Fonctions / fenêtre exploitation”. Open “Fichier/Ouvrir courbe”. Select one experiment and the blank with the CTRL button, and click on “Ouvrir”
- Select ***first*** the curve of the experiment, ***and then*** the curve of the blank with the CTRL button

- Go to the menu “Math”, then select “Soustraire courbes”. The blank is substracted from the experimental curve
- Select the new curve (experiment – blank). Go to the “TA” menu, then select “Rapporter à la taille d’échantillon”. The measurement is now given in [%]
- Select the curve of the experiment and the curve of the blank with CTRL, and with a right click, delete them with “Découper”
- Arrange the scale in the y and the x axes by double clicking on them
- Select this curve (experiment – blank). Go to the “Math” menu, then select “Dérivée 1ère”. The derivative curve is drawn, which allows to identify more easily the phenomena related to the mass loss (the resulting curve is a [1/min] versus [time or Temperature rise])
- Select this curve (experiment – blank). Go to the “TA” menu, then select “Palier horizontal”. Place the first cursor at the beginning of the measurement, and the second at the end of the measurement: the total mass loss is then indicated
- The color of each segment can be changed by double-clicking on it, to give a clearer analysis

Print the results

- Go to “Fichier”, “Aperçu graphique avant impression”, and “Imprimer”

Export the results,

- Go to “Fichier”, “Importer/Exporter”, “Exporter autre format”, and save the document as Powder-Lotn°-TGA-Experimentn°-Operator.txt.
- Go to “Fichier”, “Aperçu graphique avant impression”, and “Imprimer”. Then choose PdfCreator. Save as Powder-Lotn°-TGA-Experimentn°-Operator.pdf.

Data storage

- Copy the TXT file, and the PDF report.
- Go to \\Ltppc40\powderfiles. Copy the folder *Powderfiles*. Paste it in your project folder, and change its name into Powder-Lotn°.
- Paste the TXT and PDF files respectively in the folders Project/Powder-Lotn°/TGA/Data and PDF.

Data treatment

- Go to \\Ltppc40\powderfiles. In the folder Project/Powder-Lotn°, open the Excel sheet “Powdersheet.xls”
- Click on the *TGA* button, and follow the instructions given in the Excel sheet.