THERMOGRAVIMETRIC ANALYSIS OF A MIXTURE
(2015)

Introduction
Thermogravimetry or thermogravimetric analysis (TGA) measures the mass loss of a sample as the temperature of the sample is increased in a controlled manner. One of the first important applications was the determination of correct drying temperature for precipitates in gravimetric analysis. The second application was the identification of the gases given off while the sample is heated and the corresponding weight loss measured. This information reveals the thermo-decomposition process and permits identification of the formula of the starting material.

TGA is used for the identification of compounds present in mixtures of materials. When such mixtures are heated the thermal curve produced consists of all possible weight losses from all components superimposed on each other. If careful interpretation and identification of the individual thermal events is carried out, identification of the components of the mixture is possible.

Identification of all the individual thermal events allows for quantitative analysis of the mixture. This laboratory experiment illustrates this through the determination of the magnesium oxalate dihydrate content in a mixture of magnesium oxalate dihydrate and magnesium oxide.

Magnesium oxalate dihydrate first loses water and then decomposes at ~500 °C. Pure magnesium oxide is stable well above 500 °C. From the weight losses observed in the TGA curves, the known formulas of both compounds and knowledge of the thermal events that have occurred, a determination is made of the % of each compound in the mixture.

Procedure
A solid mixture of magnesium oxalate dihydrate and magnesium oxide is analyzed in duplicate to determine the percent composition of each compound in the mixture. Standard magnesium oxalate dihydrate is also analyzed for comparison to the unknown. Both solids have been dried overnight at 100°C and are stored in a desiccator. Keep the solids dry.

Operating the TGA
The TGA instrument contains a very sensitive microbalance. Avoid bumping the instrument and the table – any interference will affect the results. If an error message appears, leave the message on the screen and report it to the demonstrator.

Three runs are required – standard and duplicate of the unknown.
1) The 4110.prc method is used for this experiment. If the method is not opened, in the Procedure window, select Custom Test. Open the Editor. Click the open file icon. Select
\dataserve3.creait.mun.ca\data\TGA\4110. Double click 4110.prc. The method contains the following parameters:

- **Pan type:** Platinum
- **Heating program:** 20 °C/min to 100 °C, then 12 °C/min to 550 °C
- **Cooling down:** 5 min air cool with open furnace
- **Flow rate to balance:** 40 mL/min (N₂)
- **Flow rate to sample:** 50 mL/min (N₂)

2) In the Summary window, under Sample Information, type in the sample name. Add any comments needed to differentiate the runs (comments will appear on the plots). Click the button to the right of Data File Name to open up the File Name window - add filename. Ensure files are saved to following directory \dataserve3.creait.mun.ca\data\TGA\4110.

3) Using brass tweezers carefully place a clean platinum pan on the tray platform. **Do not touch the pan with fingers.** Ensure the wire on the bottom of the pan fits into the groove on the platform. The pan must be in the correct position before proceeding.

4) On the computer screen, click the tare icon (looks like a scale) in the top toolbar. The instrument will load the pan and tare it against an internal weight. The pan is automatically unloaded when the tare procedure is complete. **Do not attempt to manually load or unload the pan.**

5) Using the brass tweezers, carefully remove the pan from the platform and place the pan in the glass dish provided. Use the small spatula provided to add about half a spatula tip of solid to the pan – should just cover the bottom of the pan. **Gently tap the pan to spread out the solid.** Use the brass tweezers to return the pan with the solid to the tray platform – ensure pan is properly aligned.

6) On the computer screen, under Control select Sample – Load. When the pan is loaded, select Furnace – Up.

7) Click the green arrow in the top toolbar to start the run. The run takes 41 minutes to complete. **IMPORTANT: While a run is in progress, avoid bumping the table and instrument. Do not use the computer during the run.**

8) At the completion of the run, the pan is automatically unloaded and a 5 min air cool down will occur. During the cool down period clean the pan and then analyze the data (procedures below).

9) Repeat from Step 2 for the next sample. Three runs are required – one of the standard and a duplicate of the unknown.

**Cleaning the Platinum pan**

Clean pan immediately after the completion of each run. **Use extreme caution when handling the pans – damage may occur if mishandled.**
1) Using the brass tweezers, remove the pan from the platform and place it in the glass dish. Use the glass dish to carry the pan into C-1027A for the rest of the cleaning procedure.

2) **Gently** dump the residue in the pan into the waste container. Brush out the residue with the paintbrush provided.

3) **Do not use the brass tweezers for this step.** Using the portable torch (see below), hold the pan in the flame with regular tweezers until red-hot for about 15s. **CAUTION: THE PAN IS EXTREMELY HOT. DO NOT TOUCH THE PAN OR LAY IT DOWN ON ANY SURFACE UNTIL COOLED.** Hold the pan away from the flame to cool for about 30s. Place the cooled pan in the glass dish.

   
   To operate the portable torch:
   i) Ensure nozzle is pointed away from flammable materials (including the operator).
   ii) Open the black knob on the top of the nozzle.
   iii) Activate the trigger. Do not leave a lit flame unattended.
   iv) Close the black knob to turn off the flame.

**Data analysis**

1) In the top toolbar, click the Full Size Plot View icon – a new window will appear. Open the file from the completed run (files are store in the data drive under TGA\4110). Record the mass shown in Size (mass of starting material).

2) In the Data File Information window, select Signals. For Y1 set the Signal as weight % and Type as Normal. For Y2 set the Signal as Weight % and Type as Derivative (time) – this will change the Y2 Signal to Deriv. Weight (%/min). Select OK to open the data plot. The data plot will show the weight loss curve plus the derivative plot.

3) Right mouse click anywhere on the plot. From the drop down window, select Weight change. Two red crosshairs will appear on the plot. Place one set of crosshairs at the beginning of the plot and the other at the end of the first weight loss (ensure mark lines up with the base of the derivative curve). Right mouse button click and select Accept Limits. The % and weight lost will appear next to the curve. Record these values.

4) Repeat step 3 for the second weight loss curve, moving the crosshairs from the beginning of the plot to the end of the plot. Leave the second set of crosshairs in place (at the end of the first weight loss). Right mouse button click and again select Accept Limits. The % and weight lost will appear next to the second curve. Record these values.

5) Print the plot (right mouse click and select print – ensure the printer is selected). Return to Experiment view to continue with the next run.
Report
1) Submit the labeled plots for each run showing compound and weight changes for each step. [1.5 marks]

2) Determine the chemical equations represented by each weight loss in the standard. Give the overall equation for the decomposition of magnesium oxalate dihydrate. [1.5 mark]

3) Calculate the percent composition of magnesium oxalate dihydrate and magnesium oxide in each duplicate of the unknown. Calculate the standard deviation, relative deviation and 95% confidence limits of the unknown. Tabulate and discuss the results. [4 marks]

Questions
1) Calculate the expected percent weight loss (at each step and total) of a pure sample of magnesium oxalate dihydrate. (Hint: use the overall decomposition equation.) Compare the expected losses to the experimental losses for the standard and explain any differences. [1.5 mark]

2) Does calcium oxalate monohydrate thermally decompose the same way as magnesium oxalate dihydrate? [You will need to search the literature for this.] Draw a possible thermal curve for both (superimposed) and label all steps. [1.5 mark]

References